Standard Method for Measurement of Stain Resistance of Anodic Coatings on Aluminum

This standard is issued under the fixed designation B 136; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This method is intended to determine whether anodic oxide coatings on aluminum and its alloys, that have undergone a sealing treatment and contact with an acid solution, are stainproof or nonadsorptive with respect to dyes.

1.2 Coatings that have been properly sealed should be proof against adsorption of coloring materials and, hence, “nonstaining” in many types of service.

1.3 This method is applicable to anodic coatings intended for applications where they are exposed to the weather, or for protective purposes in corrosive media, and where resistance to staining is important.

NOTE 1—Performance in this test is predictive only of susceptibility to stain by dyes. It is not intended to be predictive of other factors in service performance such as pitting or general corrosion.

NOTE 2—For Aluminum Association Class I and II architectural anodic coatings that are sealed in solutions containing less than 15 ppm silicates or 3 ppm phosphates, the acid pretreatment may be omitted.

1.4 In the case of coatings colored in deep shades, where estimation of the intensity of any residual dye stain is difficult, interpretation of the test is based on whether or not the original color has been affected by the action of the test.

1.5 This method is not applicable to:

1.5.1 Chromic acid type anodic coatings.

1.5.2 Anodic coatings on aluminum alloys containing more than 2 mass % Cu or 4.5 mass % Si.

1.5.3 Anodic coatings that have been sealed only in dichromate solutions.

1.5.4 Anodic coatings that have undergone a treatment to render them hydrophobic.

1.6 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Summary of Test Method

2.1 The method depends upon the observation that a non-sealed or poorly sealed anodic coating is attacked by acid and easily colored by dye, while an adequately sealed coating is not appreciably attacked and does not retain any dye stain.

2.2 The method comprises contacting the test area of the anodized specimen with nitric acid solution and, after rinsing and drying, applying a special dye solution followed by rinsing and rubbing the test area with pumice powder, drying, and visual examination of the test area for retention of dye stain. Coatings that exhibit no dye stain or change in color are considered to have passed the test.

3. Reagents

3.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,2 where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

3.2 Nitric Acid Solution—Prepare a 40 ± 5 mass % solution of nitric acid (HNO₃) in distilled or deionized water.

NOTE 3—A convenient way is to carefully add one volume of 70 % HNO₃ (sp gr 1.41 at 20°C) to an equal volume of water, while stirring, observing the normal precautions for handling strong acids.

3.3 Special Dye Solution—Dissolve 1 g of aluminum blue 2LW dye3 in 50 mL of distilled or deionized water. The pH of the dye test solution shall be adjusted to 5.0 ± 0.5 and maintained at this value with additions of acetic acid or sodium hydroxide, as required.

NOTE 4—Dye solutions contaminated with soluble phosphates have

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3 A suitable dye for this test is Aluminum Blue 2LW, available from Sandoz Colors and Chemicals, East Hanover, NJ, 07936.
reduced capability for staining nonsealed or poorly sealed anodic coatings. The dye test solution should have less than 20 ppm of phosphorus as soluble \( \text{PO}_4^{3-} \). This corresponds to a phosphorus content of less than 0.05 mass % in the dye powder. Under some conditions of storage and use, dye test solutions may become contaminated or deteriorate with age. To check the reliability of a used or aged dye test solution, it should be applied from time to time to a freshly prepared, nonsealed anodic coating on aluminum to confirm that it will produce a permanent, deeply colored stain in the anodic film.

4. Procedure

4.1 Perform the test on an area that has not been contaminated by handling.

4.2 Apply a drop of \( \text{HNO}_3 \) \((40 \pm 5\) mass %\) on the test area and allow it to remain there for 2 min \( \pm 5\) s. The temperature of the \( \text{HNO}_3 \) and of the test specimens shall be \( 25 \pm 5^\circ\)C.

4.3 Wash the test area thoroughly with running water and blot dry with a clean cloth. The temperature of the water shall be \( 25 \pm 5^\circ\)C.

4.4 Apply a drop of the dye solution to the test area and allow it to remain there for 5 min \( \pm 10\) s. The temperature of the dye solution and of the test specimen shall be \( 25 \pm 5^\circ\)C.

4.5 Wash the test area thoroughly with running water, then rub it with pumice powder (NF or USP Fine Grade)\(^4\) and a clean cloth wet with water, and finally rinse with water and blot dry with a clean cloth. The temperature of the water shall be \( 25 \pm 5^\circ\)C.

Note 5—The pumice scrub removes sealing smut that may absorb the dye. Smut is more difficult to remove after aging than it is from freshly sealed work. Dye that has penetrated the pores of the oxide will not be removed except by removing the oxide. On sound oxides, a pumice scrub will do little more than burnish the surface, even with heavy hand pressure.

5. Interpretation

5.1 The test specimen shall be considered to have passed the test if no color from the dye is visible in the test area. Any visible retention of dye color in the test area is considered a failure.

5.2 In the case of anodic coatings colored in deep shades, where visual detection of any residual dye stain is difficult or impossible, the criterion for passing the test shall be no visible change in the color of the coating from the action of the test.

Note 6—For special applications it may be found desirable to make the staining test using a solution with which the article may come in contact in service. For example, it is sometimes convenient to test anodically coated aluminum cafeteria trays with coffee. Composition of test solution and test conditions should be standardized for each application.

Note 7—Sometimes the time interval between sealing and application of the stain test influences the result. In such cases, the purchaser and seller should agree upon a time limit for application of this test after the sealing operation.

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\(^4\) A suitable material is available as product No. P363 from Fisher Scientific Co., Fairlawn, NJ, 07410.
Designation: B 137 − 95 (Reapproved 2000)

Endorsed by American Electroplaters' Society
Endorsed by National Association of Metal Finishers

Standard Test Method for Measurement of Coating Mass Per Unit Area on Anodically Coated Aluminum

This standard is issued under the fixed designation B 137; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers determination of the mass per unit area of coating on anodically coated aluminum and its alloys.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
D 1193 Specification for Reagent Water

3. Significance and Use

3.1 This test method is a destructive test procedure suitable for quality control within manufacturing operations for coating mass on anodically coated aluminum and aluminum alloys. The mass of the coating is an approximation of its thickness provided the conditions under which the coating was applied, or the density of the coating, are known. An anodic coating’s protective value and amenability to coloring by dying or electrolytic methods are related to mass of coating per unit area.

4. Reagents

4.1 The test solution shall have the following makeup:
Chromic acid anhydride (CrO₃) 20 ± 0.5 g
Orthophosphoric acid of 85 mass %, density 1.69 35 ± 0.5 mL
Distilled or deionized water conforming with Type III Reagent Water of Specification D 1193, to make up to 1000 mL

NOTE 1—This solution is commonly referred to as a nondestructive “stripping solution” for anodic coatings. This solution dissolves the anodic coating with no significant attack of the substrate metal.

5. Test Specimen

5.1 Prepare a test specimen of known surface area, preferably a piece about 75 mm square. If the anodic coating is contaminated or impregnated with oil, grease, lacquer, etc., remove as much as possible of this material before determining the mass of the coating. It is not usually practicable to remove these materials from the pores of the coating, but surface films can frequently be removed by wiping the sample with a cloth wet with a suitable solvent.

5.2 In cases where it is required to measure the mass of coating on only one side of a specimen where both sides have anodic coatings, the following procedure may be used:

5.2.1 Prepare a specimen somewhat larger than required for 5.1.

5.2.2 Apply a suitable stop off around the edges of the specimen so that the area on the side of the specimen that is not to be tested is about 75 mm square. Paraffin wax has been used for this purpose, but any stop off that resists warm sodium hydroxide is suitable.

5.2.3 Swab the area with a warm solution of sodium hydroxide (10 mass %, 100 g/L) until bare metal is exposed, which is indicated by uniform bubbling over the entire exposed surface.

5.2.4 Swab with warm distilled water.

5.2.5 Dry with a clean cloth.

5.2.6 Swab with diluted nitric acid (1 part HNO₃, density 1.41, plus 1 part water).

5.2.7 Rinse with warm deionized or distilled water.

5.2.8 Dry at approximately 100°C until dry.

5.2.9 Cool and shear off the area covered with stop off, so that the coated area is the desired size, preferably at least 75 mm square.

NOTE 2—The stop off is intended to prevent the coating removal process from affecting the test area. Check visually to verify that the solutions have not run over the edges and removed or damaged the coated area to be tested.

6. Procedure

6.1 Measure the area of the coating. Do not include the area of cut edges or other uncoated surfaces.

6.2 Weigh the test specimen before and after stripping the
6.3 Immerse the test specimen for 5 min in the phosphoric-chromic acid solution maintained at a temperature of about 100°C. Remove the specimen, wash in distilled or deionized water, dry, and weigh. Repeat this cycle of operations until the coating is completely removed, which is indicated by the weight of the specimen remaining constant.

7. Report
7.1 Report the mass of coating in milligrams per square decimetre.

8. Precision and Bias
8.1 Precision—The precision for this test method is most affected by the precision of the measurement of the test area. When performed by properly instructed personnel, the method is capable of yielding results with an uncertainty of less than 10 % over a significant range of coating weights.

8.2 Bias—The procedure in this test method for measurement of the mass of coating on anodically coated aluminum has no bias because the value of the mass of coating is defined only in terms of this test method.
1. Scope

1.1 This guide provides information about the deposition of chromium on steel for engineering uses. This is sometimes called “functional” or “hard” chromium and is usually applied directly to the basis metal and is usually thicker than decorative deposits.

1.2 This guide is not intended as a standardized procedure, but as a guide for obtaining smooth, adherent coatings of chromium of a desired thickness while retaining the required physical and mechanical properties of the base metals. Specified chromium electrodeposits on ferrous surfaces are defined in Specification B 650.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 244 Test Method for Measurement of Thickness of Anodic Coatings on Aluminum and of Other Nonconductive Coatings on Nonmagnetic Basis Metals with Eddy-Current Instruments
B 253 Guide for Preparation of Aluminum Alloys for Electroplating
B 254 Practice for Preparation of and Electroplating on Stainless Steel
B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings
B 320 Practice for Preparation of Iron Castings for Electroplating
B 322 Practice for Cleaning Metals Prior to Electroplating
B 481 Practice for Preparation of Titanium and Titanium Alloys for Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
B 499 Test Method for Measurement of Coating Thicknesses by the Magnetic Method Nonmagnetic Coatings on Magnetic Basis Metals
B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
B 507 Practice for Design of Articles to Be Electroplated on Racks
B 558 Practice for Preparation of Nickel Alloys for Electroplating
B 568 Test Method for Measurement of Coating Thickness by X-ray Spectrometry
B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings
B 578 Test Method for Microhardness of Electroplated Coatings
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 630 Practice for Preparation of Chromium for Electroplating with Chromium
B 650 Specification for Electrodeposited Engineering Chromium Coatings on Ferrous Substrates
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 762 Test Method of Variables Sampling of Metallic and Inorganic Coatings
B 849 Specification for Pre-Treatments of Iron or Steel for Reducing Risk of Hydrogen Embrittlement
B 850 Guide for Post-Coating Treatments Steel for Reducing the Risk of Hydrogen Embrittlement
B 851 Specification for Automated Controlled Shot Peening of Metallic Articles Prior to Nickel, Autocatalytic Nickel, or Chromium Plating, or as Final Finish
F 519 Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments

2.2 Military Standard:

1 This guide is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.08 on Engineering Coatings.


2 Annual Book of ASTM Standards, Vol 02.05.

3 Annual Book of ASTM Standards, Vol 15.03.
MIL-S-13165B Shot Peening of Metal Parts

3. Substrates

3.1 Engineering chromium may be plated directly to the surface of a number of commonly used engineering metals such as aluminum, nickel alloys, cast iron, steels, copper, copper alloys, and titanium. The bond strengths of the chromium varies with metallic substrate. Nevertheless, if the procedures cited in the appropriate references are followed, the bond strength is such that grinding and honing can be conducted without delamination of the coating.

3.2 Smoothness—The smoothness of the material surface to be electroplated should be adequate to meet the requirements of the finished product. Chromium electrodeposits do not exhibit leveling, and consequently the surface roughness of the electrodeposits will always be greater than that of the substrate. Any mechanical operations that can result in grinding checks or glazing of the metal are detrimental and should be eliminated. The required surface smoothness may be obtained by suitable chemical, mechanical, or electrochemical procedures. Depending upon the thickness of the electrodeposits and the smoothness required of the electrodeposits, grinding of the electrodeposits may be required.

3.3 Fatigue Considerations—Cracking that can occur in chromium electrodeposits either as a function of the plating bath chemistry or the plating conditions, or both, or as a result of grinding of the electrodeposits can lead to a reduction in the fatigue life of the electroplated part. If this is a design consideration, the use of mechanical methods such as shot peening (see Specification B 851 or MIL-S-13165C, or both) or autofrettage to compressively stress the surface can increase the fatigue strength. This should be done after any stress-relieving heat treatment.

3.4 High-Strength Steel Stress Relief:

3.4.1 All steel parts having an ultimate tensile strength of 1000 MPa (150 000 psi, approximately 32 HRC) or greater, which may contain residual stress caused by various fabrication operations such as machining, grinding, straightening, or cold-forming, usually will require one of the stress relief bakes prescribed in Specification B 849 prior to electroplating. In all cases, the duration of the bake shall commence from the time at which the whole of each part attains the specified temperature. This stress relief is essential if hydrogen embrittlement from subsequent operations is to be avoided.

3.4.2 Parts having surface-hardened areas that would suffer an unacceptable reduction in hardness by baking in accordance with Specification B 849 may be baked at a lower temperature but not less than 130°C for a minimum period of 8 h. Shorter times at higher temperatures may be used, if the resulting loss in surface hardness is acceptable.

3.5 Oxidation—All possible precautions should be taken to prevent oxidation of the metal surface between the final operations of mechanical preparation and electroplating, particularly with steel substrates. Materials such as aluminum and titanium have an inherent oxide film on the surface that can only be removed or minimized just prior to the electroplating process (see 6.1.1 and 6.1.2). When conditions are especially unfavorable, definite steps must be taken to meet this important requirement, including storage in a noncorrosive environment, or the use of a suitable coating to exclude air and moisture.

4. Racks and Anodes

4.1 Steel, cast iron, and stainless steel parts to be electroplated may be racked at any convenient stage in the preparatory process but preferably prior to the final cleaning and etching. Aluminum, titanium, and certain nickel alloys may need to have cleaning and etching operations done before racking due to entrapment of cleaning and etching solutions in the plating rack which can result in adhesion failures due to seepage during chromium electroplating.

4.2 See Practice B 507 for guidance on rack design, but note that while the general principles of good racking as used in other electroplating processes apply, the use of much higher current densities and the desirability of securing coatings of uniform thickness and quality on desired areas require rack construction designs and methods that are much more exacting. The design of racks for chromium electroplating on the various base metals previously mentioned for functional use should provide for the following to the greatest possible extent.

4.2.1 There must be sufficient current-carrying capacity of both cathode and anode circuits to all parts of the rack.

4.2.2 There must be positive electrical contact to the parts to be electroplated, to the anodes, and to the tank contact bus bars.

4.2.3 There must be uniform current distribution on the parts to be electroplated. This often requires anodes of special shapes conforming to the shape of the part or area to be electroplated.

4.2.4 It may be necessary to use thieves, robbers, or guards, which are auxiliary metallic conductors placed near points of abnormally high current density to attract the current away from such points; and shields, which are parts made of nonconductive materials and placed to disperse the current in areas where it tends to concentrate unduly.

4.2.5 It is important to protect areas that are to remain free of any chromium electrolyte by the use of masks made of rigid, nonconductive materials placed against the substrate, or stop-offs, which are especially compounded nonconductive tapes, waxes, lacquers, or plastics for the protection of such substrates. Lead and aluminum tapes will provide a sharp line of demarcation between coated and uncoated areas with a minimum of buildup.

4.2.6 Plugs (conducting and nonconducting) may be used in holes not requiring electroplating to produce a sharp edge without grooves around the periphery of the holes.

4.2.7 It is very important to remember that improperly applied stop-off materials or poorly designed racks can entrap acids that can cause corrosion of the basis material or contamination of the solutions used in subsequent operations, or both.

4.2.8 Construction materials must be used that are sufficiently insoluble and noncontaminating to provide the desired rack life.

4.2.9 Components must be placed in such positions that gas from the parts, rack, thieves, masks, and anodes escapes freely and does not become entrapped so as to prevent electroplating.

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4 Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.
on areas that should be electroplated.

4.3 Anodes—Lead anodes containing 4 to 6 % antimony, 4 to 7 % tin, or 1 % silver, or a combination thereof, are satisfactory. Chemical lead is also satisfactory where hardness and rigidity are not important. However, it tends to form great quantities of scale that may fall off on the work and cause pitting or roughness. Lead wire used for small anodes should contain 0.25 % antimony to obtain the best relationship between rigidity and ductility in close tolerance areas. Lead-sheathed copper, silver, or even steel rods or wire may be used for internal electroplating of small holes, but the latter will contaminate the bath with iron. If the anode contains little or no lead, the reoxidation of trivalent chromium to the hexavalent state will not take place or will be seriously impaired, which will lead to trivalent buildup in the plating solution and poor results.

4.3.1 Some proprietary baths may require special anodes, which should be recommended by the supplier.

5. Cleaning

5.1 Parts to be electroplated may be cleaned in accordance with Practices B 183, B 242, B 254, B 281, B 320, B 322, B 481, B 558, or B 630, or Guide B 253.

5.2 Mechanical methods of cleaning steel prior to electroplating, including abrasive blasting or light grinding, are also suitable. If parts have been shot-peened to develop a compressively stressed surface, it is important to avoid removing that surface by excessive grinding.

6. Deoxidizing and Etching

6.1 Prior to chromium electroplating, most metals need special preparation in order to achieve maximum adhesion of the chromium to the substrate. Depending on the type and nature of the metal and prior surface preparation steps, various deoxidation and etching methods may be used to activate the substrate prior to chromium electroplating.

6.1.1 Aluminum—Chromium may be electroplated directly onto most cast and wrought aluminum materials used for engineering purposes. Guide B 253 offers many useful methods for preparing aluminum prior to chromium electroplating. The removal of the ever-present, tenacious oxide film on the surface of aluminum is what makes electroplating difficult. When using test methods in which a zinc immersion film is applied to the aluminum surface for protection against oxide formation, the article to be plated must enter the chromating solution under live current.

6.1.2 Titanium—Like aluminum, titanium has an ever-present tenacious oxide film that must be removed prior to plating. Practice B 481 offers many ways to prepare titanium prior to chromium electroplating.

6.1.3 Nickel Alloys—Several different activation methods are available in Practice B 558 for the preparation of different nickel alloys. The main difficulty with these materials when chromium plating is polarization of the nickel alloy surface prior to plating which results in deactivation of the material and skip plating.

6.1.4 Copper and Copper Alloys—Practice B 281 offers many suitable methods for preparing copper and copper alloys prior to chromium electroplating. In general, only deoxidizing of the copper or copper alloy surface is necessary for chromium electroplating.

6.1.5 Stainless Steel—Practice B 254 offers many suitable activating procedures for the preparation of stainless steel prior to chromium electroplating. Some stainless steels benefit from a Woods nickel strike prior to chromium electroplating. Polarized surfaces in high-nickel stainless steels can cause skip plating if not properly activated.

6.1.6 Cast Iron—Practice B 320 offers many suitable procedures for activating cast iron prior to chromium electroplating. In general, anodic etching in the chromium plating solution is not recommended. Due to the high carbon content in iron castings, anodic etching leaves a carbon smut on the surface of the metal which results in poor adhesion of the chromium.

6.2 Chromium plating on steel is among the most common combination for engineering purposes. Unique activation procedures for steel exist with chromium plating that merit a separate discussion for successful plating as follows.

6.2.1 Etching of the steel before electroplating is ordinarily desirable to obtain satisfactory adhesion of the chromium to the steel. To reduce the increase in roughness resulting from etching, the etching times should be kept as short as is consistent with good adhesion, particularly in the case of highly finished surfaces.

6.2.2 Anodic Etching in Chromic Acid Solution—The part to be electroplated may be anodically etched in a solution of approximately the same concentration of chromic acid as the plating solution (for example, 250 g/L (33 oz/gal) at approximately the temperature used in plating. There should not be any sulfuric acid present. Enter the tank with the current off and make the part anodic for 10 s to 2 min at a current density of 11 to 32 A/dm² (100 to 400 A/ft²). Tank voltage is normally 4 to 5 V. There does not have to be rinsing before transfer to the plating tank, but parts should be thoroughly drained to prevent spillage of the etching solution.

6.2.3 Anodic Etching in the Plating Solution—Using the same times and current density described in 6.2.2, parts can be etched in the etching solution itself. A reversing switch should be provided to make the part anodic. This process is much simpler than that in 6.2.2 and requires one less tank, but has the disadvantage of contaminating the bath with iron, copper, and so forth.

6.2.4 Anodic Etching in Sulfuric Acid Solution—A sulfuric acid (H₂SO₄) solution of 50 to 70 volume % 66 Be H₂SO₄ may be used for etching. The temperature should be kept below 30°C and preferably below 25°C. The time of treatment is 10 s to 2 min, and the current density 11 to 54 A/dm² (100 to 500 A/ft²) at 4 to 6 V. Lead cathodes should be used and the tank constructed of a material, such as lead or vinyl, that is resistant to sulfuric acid. Two difficulties that may be encountered that make this process less attractive than those described in 6.2.2 or 6.2.3 are:

6.2.4.1 If the rinsing following etching is incomplete, the drag-in of sulfuric acid changes the chromic acid to sulfate ratio in the chromium plating bath with deleterious results, and
6.2.4.2 In handling parts that are difficult to manipulate, there is a danger of rusting of the surfaces before the part can be introduced into the chromium electroplating bath.

6.2.5 Slight Etching by Acid-Immersion—A slight etching may be obtained by a short dip at room temperature in either 10 to 50 volume % hydrochloric acid (HCl 37 weight %) or 5 to 15 volume % sulfuric acid (H₂SO₄ 98 weight %). This is normally used on highly finished steel requiring only a thin chromium deposit as its use may result in less adhesion than other procedures and in hydrogen embrittlement of the steel. Drag-over of either solution into the chromium electroplating bath because of poor rinsing will cause contamination problems.

### 7. Chromium Electroplating

7.1 Unless the parts are etched by reverse in the plating bath (6.2.3), they are introduced into the chromium electroplating bath after all preparatory operations. Any auxiliary anodes integrated with the rack are connected to the anode bus bar. Steel or ferrous parts to be plated are allowed to reach the bath temperature and electroplating is then commenced. If the parts were etched in the plating solution, plating is initiated when the parts are made cathodic at the end of the etching period using the reversing switch. Most nonferrous metals enter the chromium plating solution under live current and are not placed in the chromium-plating solution for warming prior to electroplating.

7.2 Electroplating Baths—In addition to the following two baths, there are various proprietary baths offered that may be satisfactory and should be operated in accordance with the vendor’s instructions. Most proprietary chromium plating baths are co-catalyzed plating solutions in which an additional catalyst is used in conjunction with the traditional sulfate anion catalyst. These co-catalysts may use organic based or inorganic based compounds to achieve higher plating efficiencies and are often employed where higher rates of plating and better throwing and covering power are needed. The most recent baths do not use fluoride co-catalysts and do not etch unprotected low current density areas. These baths produce micro-cracked deposits which may be an advantage in some deposits. There are additives, such as selenium, in the patent-free art which will also produce micro-cracked deposits.

7.2.1 This is the most common bath and will deposit chromium at the approximate rate of 25 µm (0.001 in.) in 80 min at 31 A/dm² (2.0 A/in²).

<table>
<thead>
<tr>
<th>Bath</th>
<th>Description</th>
<th>Current Density</th>
<th>Temperature</th>
<th>Sulfate (SO₄²⁻)</th>
<th>Silicofluoride (SiF₆³⁻)</th>
<th>Cr (CrO₃)</th>
<th>H₂SO₄</th>
<th>H₂SiF₆</th>
<th>CrO₂⁺</th>
<th>CrO₃⁻</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bath 1</td>
<td>Co-catalyzed bath</td>
<td>31 to 62 A/dm²</td>
<td>55°C</td>
<td>1.5 g/L</td>
<td>0.75 g/L</td>
<td>250 g/L</td>
<td>2.5 to 3.1 g/L</td>
<td>1.6 mL/L</td>
<td>1.6 mL/L</td>
<td>0.75 g/L</td>
</tr>
</tbody>
</table>

Note 4—Warning: The silicofluoride (sometimes shown as fluorosilicate) anion may most conveniently be added as hydrofluorosilicic acid (fluorosilicic acid), which is commonly sold at a concentration of 31 weight % H₄SiF₄, in which case the addition of 1.6 mL/L will give the concentration of 2.0 g/L in the bath. This acid also requires great care in handling. Consult safety references or personnel before using.

7.2.2 The following co-catalyzed bath gives greatly improved efficiencies in comparison with the standard bath in 7.2.1 under identical conditions. The addition of fluoride or silicofluoride auxiliary catalysts increase the tendency of the bath to etch steel in unprotected low-current density areas, and more masking may be required than is necessary with the standard bath. Analytical control of the silicofluoride is more difficult than the other components, but ion selective methods are satisfactory. This bath will deposit chromium at an appropriate rate of 37.5 µm (0.0015 in.) in 60 min at 31 A/dm² (2 A/in²).

<table>
<thead>
<tr>
<th>Bath</th>
<th>Description</th>
<th>Current Density</th>
<th>Temperature</th>
<th>Sulfate (SO₄²⁻)</th>
<th>Silicofluoride (SiF₆³⁻)</th>
<th>Cr (CrO₃)</th>
<th>H₂SO₄</th>
<th>H₂SiF₆</th>
<th>CrO₂⁺</th>
<th>CrO₃⁻</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bath 2</td>
<td>Co-catalyzed bath</td>
<td>20 to 90 A/dm²</td>
<td>15 – 25°C</td>
<td>0.75 g/L</td>
<td>0.75 g/L</td>
<td>325 g/L</td>
<td>175 g/L</td>
<td>0.75 g/L</td>
<td>15 – 25°C</td>
<td></td>
</tr>
</tbody>
</table>

Note 5—Warning: Literature references suggest preparing this bath by adding sodium hydroxide to a 4 Mol chromic acid solution. This is a very dangerous exothermic reaction. The preceding solution should, of course, be handled with all the caution required of standard chromium plating baths.

7.2.3 The following bath produces very soft (usually less than 650 VHN₂₅) deposits that are crackfree. The deposits are dull gray in color and can be buffed, if desired. The efficiency is very high and the chromium electrodeposits in a different crystal structure than is obtained in other baths. There are many modifications reported in the literature and some manufacturers offer proprietary baths.

<table>
<thead>
<tr>
<th>Bath</th>
<th>Description</th>
<th>Current Density</th>
<th>Temperature</th>
<th>Sulfate (SO₄²⁻)</th>
<th>Cr (CrO₃)</th>
<th>H₂SO₄</th>
<th>CrO₂⁺</th>
<th>CrO₃⁻</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bath 3</td>
<td>Co-catalyzed bath</td>
<td>20 – 90 A/dm²</td>
<td>15 – 25°C</td>
<td>0.75 g/L</td>
<td>325 g/L</td>
<td>175 g/L</td>
<td>0.75 g/L</td>
<td>0.75 g/L</td>
</tr>
</tbody>
</table>

Note 5—Warning: Literature references suggest preparing this bath by adding sodium hydroxide to a 4 Mol chromic acid solution. This is a very dangerous exothermic reaction. The preceding solution should, of course, be handled with all the caution required of standard chromium plating baths.

7.2.4 Black chromium deposits are produced from the following bath. There are also proprietary solutions available. These deposits are frequently used on solar collectors and for applications on steels and other alloys where a more wear-resistant coating than black oxide types is desired. In operating these baths, it is essential that no sulfate be introduced into the bath. All baths of this type include barium salts or other precipitants for sulfate. As the deposit is nonconductive, the maximum thickness that can be expected is 3 to 5 µm which requires 4 to 8 min. Mild steel anodes are usually employed.
8. Treatments of Chromium Coatings

8.1 Hydrogen Embrittlement—Hydrogen evolved during chromium plating is apt to embrittle steel, and the potential for embrittlement increases with the higher strength (harder) steels. Baking appropriate for the tensile strength of the electroplated part must be performed to reduce the risk of hydrogen embrittlement. Guide B 850 lists bakes appropriate for the tensile strength of the electroplated part and should be consulted for post-electroplating baking procedures and classes. In all cases, the duration of the bake shall commence from the time at which the whole part attains the specified temperature. The bake should be performed as soon as possible after the parts are removed from the plating bath, rinsed, and dried in order to reduce the risk of hydrogen embrittlement. Consult Specification B 850 for maximum length of time permitted between plating and baking operations.

NOTE 6—It is suggested that the selection of an appropriate bake be discussed with the purchaser to ensure that the bake selected does not cause distortion in the part or adversely affect its mechanical properties.

NOTE 7—The effectiveness of hydrogen embrittlement relief baking of chromium-plated high-strength steels can be tested in accordance with Test Method F 519.

8.2 Mechanical Finishing—Chromium electrodeposits are commonly finished to the required final dimension by grinding, honing, or lapping. If grinding is very aggressive, removing a large amount of metal per grinding pass and generating high localized temperatures, the chromium is apt to develop a network of macrocracks visible to the naked eye. This condition will greatly reduce the fatigue life of the part and should be avoided. Compressively stressing the substrate surface prior to plating by shot peening (see Specification B 851 or MIL-S-13165C, or both) or other means will help prevent any diminution of the fatigue life. Chromium deposited from the higher concentration sulfate catalyzed baths are less prone to macrocracking during grinding than those deposited under similar conditions from a cocatalyzed bath (see 7.2.2) or the lower concentration sulfate bath (see 7.2.1). Proprietary baths should be evaluated for the tendency towards macrocracking if fatigue life is an important design consideration. For parts loaded in compression or not subject to cyclical applications of stress during operation, or both, this may not be a consideration.

9. Repair of Chromium Electrodeposits on Steel Substrates

9.1 A worn chromium electrodeposit may be restored to the original dimension by re-electroplating.

9.2 If the part is completely covered in chromium in the areas originally electroplated, it may be prepared for electroplating in accordance with Practice B 630.

9.3 If steel shows through or if the anodic treatment exposes steel, the chromium coating must be completely removed prior to re-electroplating. Stripping the chromium may be done by anodic treatment at 5 to 8 A/dm² (75 A/ft²) in a solution containing 40 to 60 g/L of sodium hydroxide or in a solution containing 40 to 60 g/L sodium carbonate. Either solution should be kept below 25°C during operation using cooling, if necessary. There are also proprietary solutions available which should be operated according to the supplier’s instructions.

10. Test Methods

10.1 Guide B 697, with Test Methods B 602 and B 762, will be helpful in choosing statistically appropriate sample sizes for the following test methods.

10.2 Thickness—The thickness of the chromium deposit is usually not determined directly, the dimension of the finished part being measured instead. When direct measurement of the thickness of the coating is desired and the part can be sacrificed, it should be done in accordance with Test Method B 487. If a nondestructive method is required, magnetic induction methods in accordance with Test Method B 499 are suitable for chromium over magnetic substrates. Test methods in accordance with Test Methods B 499 can measure coating thicknesses from 2.5 µm to 12 mm (0.1 mil to 0.5 in.). Test Method B 244 may be used accurately for chromium up to 500 µm (0.020 in.) over aluminum or copper alloys but not for titanium. For deposits up to 50 µm (2 mils), Test Method B 504 may be used and does not destroy the part, but does remove the chromium electrodeposits on the area tested, which may necessitate replating. X-Ray fluorescence may be used to measure very thin chromium deposits of 1 to 20 µm (3 µin. to 0.8 mils) in accordance with Test Method B 568. Other test methods may be used by agreement between the purchaser and the seller.

10.3 Hardness—Hardness will vary with bath composition and the conditions used for electrodeposition. Hardness should be measured in accordance with Test Method B 578 on a panel plated concurrently with the part unless the part can be sacrificed.

10.4 Adhesion—Adhesion should be measured using Practice B 571 on a panel plated concurrently with the part and on the same material as the part.

11. Keywords

11.1 chromium electroplating
1. Scope

1.1 This practice is intended as an aid to electroplaters in setting up a suitable cleaning cycle preparatory to electroplating of low-carbon steel (Note 1) containing less than 0.35 mass % of carbon and to indicate some of the precautions that must be taken to maintain this cycle in good operating condition.

Note 1—The preparation of high-carbon steel for electroplating is covered in Practice B 242.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 322 Guide for Cleaning Metals Prior to Electroplating

3. Nature of Cleaning

3.1 The preparation of low-carbon steel for electroplating involves three basic steps in the following order:

3.1.1 Removal of oil, grease, and caked-on dirt,
3.1.2 Removal of scale and oxide films by “pickling,” and
3.1.3 Removal of any smut left on the surface after step 3.1.2 and activation of the steel.

3.2 Removal from the steel of fabricating lubricants and finishing compounds may have to be undertaken by “precleaning” before the articles reach the electroplating room. The remainder of the operations should immediately precede the electroplating. In some instances separate removal of smut may not be necessary as in the case of parts which are barrel-electroplated and tumbled.

4. Cleaning Solutions and Apparatus

4.1 All solutions should be subject to chemical control, including determinations of the free acid and iron contents of the pickling solutions and acid dips, and such tests of the cleaning solutions as recommended by the manufacturer.

4.2 Based on tests and experience, all solutions should be discarded before they have lost their effectiveness.

4.3 To conserve cleaning and pickling solutions and to ensure continuous operation when heavy production is involved, doubling of facilities in the same line of operation may be desirable. This arrangement will result in a high degree of contamination of the first of two solutions of the same kind while the second ones will be sufficiently clean to continue to use. It will also reduce the degree of contamination of subsequent solutions, for example, by oil and grease.

4.4 As an alternative to the procedure described in 4.3, the cleaner and pickle tanks may be provided with a large dam overflow and a pump having its intake placed about half-way down the overflow dam between the accumulated grease and oil on top and the settled-out solid dirt at the bottom, and having its outlet placed near the bottom of the tank at the end opposite to the dam overflow.

4.5 Separate tank electrodes, removable from the tank for inspection and cleaning, should be used. Contact hooks for the electrodes should be of the inverted V-type for round tank bars and should be of sufficient size to carry the required current.

4.6 Rinse tanks should be arranged with a dam overflow, and any water inlet other than a spray should be placed so as to ensure thorough circulation of the water and a large working surface free from grease accumulation. An adequate flow of water is essential.
4.7 Heating coils should be placed on the work-piece side of the tank in order to assure a working surface free from grease accumulation.

5. Procedure for Racked Parts

5.1 The cycle described in 5.2 to 5.6 should be used for the preparation of racked parts subsequently electroplated in still tanks, agitated tanks, semi- and full-automatic equipment, except in those cases described in Section 6.

5.2 Precleaning—In general, it is necessary to remove fabricating lubricants, buffing compounds, and other soils by precleaning. This precleaning may be accomplished with alkaline soak cleaners, cleaners designed to remove buffing compounds (including the use of ultrasonic cleaners), alkaline spray cleaners, and the use of chlorinated solvents such as trichloroethylene and perchloroethylene in vapor-type degreasing equipment, or by use of cold chlorinated solvents if vapor equipment is not available. Precleaning normally should be accomplished as soon as possible after fabrication because many stamping lubricants and buffing compounds become much more difficult to remove if allowed to age on the steel surface and can chemically attack the substrate causing etching.

5.3 Electrocleaning—The part to be cleaned should be anodically (reverse) electrocleaned in a solution of a suitably compounded, free-rinsing, high-conductivity steel electrocleaner, at a concentration of about 45 to 90 g/L. The current density should preferably be between 5 and 6 A/dm² and the tank voltage about 6 V. The temperature should be between 60 and 90°C. The time of cleaning following an effective precleaning operation is usually between 1 and 4 min. If proprietary cleaners are used, the recommendations of the manufacturer should be followed.

5.4 Water Rinse—Immerse the part to be cleaned in clean, overflowing water for a minimum of 15 s. If possible, air agitation of the water rinse should be provided. If the water is very cold, below 10°C, slight warming is beneficial. A fresh water spray on the part as it enters and leaves the rinse tank helps ensure complete rinsing.

5.5 Acid Pickling—Pickle the part in a solution of 150 to 500 mL of concentrated, 31 mass %, hydrochloric acid (density 1.16 g/mL) diluted to 1 L, 100 mL of concentrated, 93 mass %, sulfuric acid (density 1.83 g/mL) diluted to 1 L, or a solution of proprietary acid salts at room temperature, for a sufficient length of time to remove all oxides, rust, or scale. (Warning—The acid should be added to the approximate amount of water required and then after thorough mixing, diluted to exact volume.)

5.6 Alkaline Descaling—In place of acid pickling (5.5) it is sometimes found that alkaline descaling may be more practical. Such solutions are operated at about 40°C and average current density of 2 to 5 A/dm². A typical bath composition may be as follows:

- Sodium hydroxide 180 g/L
- Sodium cyanide 120 g/L
- Chelating agent (EDTA-NTA gluconates) 80 g/L

When using this method it must be followed by a water rinse (5.4), an acid dip (5.5), and water rinse (5.4).

5.7 Water Rinse—Rinse the part again, as described in 5.4 but in a separate tank.

6. Variations in Procedure for Racked Parts

6.1 Consideration should be given to separate precleaning between manufacturing operations; for example, between gear-cutting and deburring operations, and between drawing or stamping and buffing operations.

6.2 Vapor phase degreasing with chlorinated solvents is an excellent way to remove mineral oil form recesses such as overlapping joints that cannot be cleaned by any other method.

6.3 Plants with limited facilities sometimes omit precleaning especially with work that is not too heavily soiled. This is permissible; however, electrocleaning time may have to be increased and it is almost always necessary to change electrocleaners more frequently. Likewise, close control is necessary to ensure proper electrocleaner concentration at all times.

6.4 While pickling in strong hydrochloric acid or acic salts in accordance with 5.5 is suitable for most oxide conditions, including heavy annealing scale, welding marks, or wheel burns, it may sometimes be preferable to use hot sulfuric acid (about 100 mL of concentrated, 93 mass %, sulfuric acid (density 1.83 g/mL) diluted to 1 L) containing a suitable inhibitor to protect the steel from over pickling (see Warning in 5.5). The acid temperature may vary between 50 and 70°C. When inhibitors are used, a second alkaline cleaning operation, preferably electrocleaning, should follow pickling to remove any adsorbed inhibitor.

6.5 Heavy scale may be removed more rapidly by making the part to be pickled either anodic or cathodic in a 50 to 100 mL of concentrated, 93 mass %, sulfuric acid (density 1.83 g/mL) diluted to 1 L or proprietary acid salt solution at a temperature of 50 to 65°C and an average current density of about 4 A/dm² (see Warning in 5.5). Anodic pickling avoids hydrogen embrittlement. Cathodic pickling provides a brighter surface provided the acid is not contaminated with heavy metals such as copper, tin or lead.

6.6 In addition to acid pickling, scale may be removed by shot-blasting, tumbling, or sandblasting. These methods avoid hydrogen embrittlement but may work-harden the surface.

6.7 When there is a lapse of time between the final rinsing operation (5.6) of the preparatory cleaning process and electroplating, arrangements should be made for temporary storage of parts. Those which are to be electroplated in a cyanide solution may be stored in a solution of 15 to 30 g/L of sodium cyanide and an equal amount of sodium hydroxide at room temperature. Those which are to be electroplated in an alkaline solution such as alkaline tin, should be stored in a solution containing 8 g/L of sodium hydroxide at room temperature. Storage time for parts to be nickel electroplated should be minimized but, if necessary, they may be held in clean cold water for a very short time (3 or 4 min, depending on water quality).

7. Procedure for Parts in Bulk

7.1 The cycle described in 7.2 to 7.5 should be used for cleaning part to be electroplated in barrels, hand-operated or automatic, except in those cases described in Section 8.
7.2 Alkaline Cleaning—Clean the parts by tumbling with or without current, in a solution of 30 to 60 g/L of a suitable alkaline cleaner at 60 to 90°C. Anodic electrocleaning at about 2 to 4 A/dm² may supplement tumbling for more effective cleaning.

7.3 Water Rinse—Rinse the parts in clean, overflowing water. Air agitation of the rinse water is beneficial. If the water is very cold, rinsing is improved by some warming.

7.4 Acid Pickling—Pickle in a solution of 150 to 500 mL of concentrated, 31 mass %, hydrochloric acid (density 1.16 g/mL) diluted to 1 L at room temperature. Other suitable pickling solutions are proprietary acid salts and 50 to 150 mL of concentrated, 93 mass %, sulfuric acid (density 1.83 g/mL) diluted to 1 L (see Warning in 5.5).

7.5 Water Rinse—Rinse the parts again as described in 7.3, using a separate tank.

8. Variations in Procedure for Bulk Parts

8.1 Two water rinses after both cleaning and acid pickling are beneficial in many barrel electroplating operations. This serves to ensure more complete rinsing and thus prevent carry-over of cleaners or acids into subsequent tanks.

8.2 Heavily scaled parts such as heat-treated nuts and bolts may require pickling in strong inhibited sulfuric acid (see 6.4). Tumble-pickling is preferred to immersion-pickling in bulk because the tumbling action increases the rate of scale and oxide removal.

8.3 Storage—If parts must be stored after cleaning and pickling prior to electroplating, the procedure described in 6.7 should be followed.

Note 2—Additional information on procedures for cleaning of low-carbon steel prior to electroplating may be found in Practice B 322.
Standard Specification for Electrodeposited Coatings of Lead and Lead-Tin Alloys on Steel and Ferrous Alloys

1. Scope

1.1 This specification covers the requirements for electrodeposited coatings of lead and lead-tin alloys on steel and ferrous alloys. The coatings of lead-tin alloys are those that range in tin content up to but not exceeding 15 mass %. The coatings ranging between 3 and 15 mass % in tin content are known also as “terne” metallic electrodeposits.

1.2 This specification does not apply to sheet, strip, or wire in the unfabricated form.

1.3 The following precautionary caveat pertains only to the test method portion, Section 11, of this specification: This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 117 Practice for Operating Salt Spray (Fog) Apparatus
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 320 Practice for Preparation of Iron Castings for Electroplating
B 322 Practice for Cleaning Metals Prior to Electroplating
B 339 Specification for Pig Tin
B 374 Terminology Relating to Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
B 507 Practice for Design of Articles to Be Electroplated on Racks
B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings

3. Terminology

3.1 Definitions—Definitions of the terms used in this specification are in accordance with Terminology B 374.

4. Classification

4.1 The coating designation shall comprise the following:

4.1.1 The chemical symbol for the basis metal.

4.1.2 The chemical symbol for the undercoating of copper or nickel, if used.

4.1.3 The chemical symbol Pb representing lead or the symbol Pb Sn for the lead-tin alloy. When tin is present, the tin content of the coating will appear before the symbol Sn. For example, Pb 5 Sn refers to a coating having the minimum composition 5 mass % tin, remainder lead.

4.1.4 A number indicating the minimum thickness of the coating in micrometres (µm). This minimum thickness shall be 6, 12, 25, or 40 µm, and shall apply to all significant surfaces specified in 8.2.2 and 8.3.1.

4.2 Examples:

4.2.1 Fe-Pb-5-Sn-40 represents a lead-tin alloy coating having 5 mass % tin content, remainder lead, on a ferrous basis metal. The thickness is 40 µm minimum.
4.2.2 Fe-Cu-Pb-6 represents a lead coating on a ferrous basis metal with a copper strike. The thickness is 6 µm minimum.

5. Sampling

5.1 Lot—An inspection lot is defined as a collection of finished articles that are of the same kind, that have been produced to the same specification, that have been coated by a single supplier at one time or at approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

5.2 Selection—A random sample of the size required by Test Method B 602 shall be selected from the inspection lot (see 5.1). The articles in the lot shall be inspected for conformance to the requirements of this specification and the lot shall be classified as conforming or nonconforming to each requirement according to the criteria of the sampling plans in Test Method B 602.

NOTE 1—Test Method B 602 contains three sampling plans that are to be used with nondestructive test methods and a fourth to be used with destructive test methods. The three methods for nondestructive tests differ in the quality level they require of the product. Test Method B 602 requires use of the plan with the intermediate quality level unless the purchaser specifies otherwise. The purchaser should compare the plans with his needs and state which plan is to be used. If the plans in Test Method B 602 do not serve the needs, additional ones are given in Guide B 697.

NOTE 2—When both destructive and nondestructive tests exist for the measurement of a characteristic, the purchaser needs to state which is to be used so that the proper sampling plan is selected. Also, a test may destroy the coating in a noncritical area; or, although it destroys the coating, the tested article might be reclaimed by stripping and recoating. The purchaser needs to state whether the test is to be considered destructive or nondestructive.

5.3 Separate Specimens—If separate specimens are to be used to represent the finished articles in a test, the specimens shall be of the nature, size, number, and be processed as required in 6.1, 6.2, 6.3 and 6.4.

6. Specimen Preparation

6.1 Electroplated Parts or Separate Specimens—When the electroplated parts are of such form, shape, size, and value as to prohibit use thereof, or are not readily adaptable to a test specified herein, or when destructive tests of small lot sizes are required, the test shall be made by the use of separate specimens plated concurrently with the articles represented. The separate specimens shall be of a basis metal equivalent to that of the articles represented. “Equivalent” basis metal includes chemical composition, grade, condition, and finish of surface prior to electroplating. For example, a cold-rolled steel surface should not be used to represent a hot-rolled steel surface. Due to the impracticality of forging or casting separate test specimens, hot-rolled steel specimens may be used to represent forged and cast-steel articles. The separate specimens may also be cut from scrap castings when ferrous alloy castings are being electroplated. These separate specimens shall be introduced into a lot at regular intervals before the cleaning operations, preliminary to electroplating of specimens, including the spacing, plating media, bath agitation, and temperature, in respect to other objects being electroplated, shall correspond as nearly as possible to those affecting the significant surfaces of the articles represented. Unless a need can be demonstrated, separately prepared specimens shall not be used in place of production items for nondestructive and visual examinations.

6.2 Thickness and Adhesion Specimens—If separate specimens for thickness and adhesion tests are required, they shall be strips approximately 25 mm wide, 100 mm long, and 1 mm thick.

6.3 Corrosion Resistance Specimens—If separate specimens for corrosion resistance tests are required, they shall be panels not less than 150 mm long, 100 mm wide, and approximately 1 mm thick.

6.4 Hydrogen Embrittlement Specimens—If specimens are required, the configuration shall be that specified by the purchaser.

7. Significance and Use

7.1 Electrodeposits of lead and lead-tin alloys on steel and ferrous alloys are produced where it is desired to obtain atmospheric corrosion resistance. Deposits of lead and lead-tin alloys on steel have shown to have excellent corrosion protective qualities in atmospheric exposure, especially when undercoated by a thin deposit of copper (or nickel). Applications of lead and lead-tin alloy deposits include the following: protection from dilute sulfuric acid; lining of brine refrigeration tanks; chemical apparatus; and parts for storage batteries; and for coating bearing surfaces. In this last application, lead is electroplated alone, or as an alloy and coated with another metal, such as indium. The indium may be diffused into the lead or lead alloy by heat treatment. See Appendix X1.

8. Ordering Information

8.1 When ordering articles to be electroplated in accordance with this specification, the purchaser shall state the coating designation (see Section 4), the minimum thickness on significant surfaces, in addition to the ASTM designation number and year of issue.

8.2 If necessary, the purchaser shall include on his part, drawings, or purchase order the following:

8.2.1 Electroplating application to high-strength steel, if specified (see 9.2.2).

8.2.2 Location of significant surfaces, to be shown on part drawing, or by the provision of a suitably marked sample.

8.2.3 Hydrogen embrittlement test, if required (see 6.4).

8.2.4 Sample size for inspection, if other than specified (see Section 5).

8.2.5 Supplementary requirements, if applicable (see Supplementary Requirements).

8.2.6 Separate test specimens, if permitted (see 6.1).

8.2.7 Certification, if required (see Section 13).

8.3 The manufacturer of the basis metal parts should provide the supplier of the coating facility, with the following data:

8.3.1 Hardness or tensile strength of steel parts (see 9.2.2 and 9.2.3).

8.3.2 Heat treatment for stress relief, whether or not it has been performed or is required.
9. Coating Requirements

9.1 Composition—The coating compositions shall be as specified in Table 1.

9.2 Process—Lead and lead-tin alloy coatings shall be produced by electrodeposition in aqueous solution of salts. For the preparation of ferrous metal surfaces necessary to assure good deposit, adhesion, and quality, see Practices B 183, B 242, B 320, and B 322.

9.2.1 A copper or nickel strike, 2.5 µm thick, may be employed and is desirable (see X1.1.2).

9.2.2 Steel parts with ultimate tensile strengths greater than 1050 Mpa (approximately 32 HRC) and that have been machined, ground, cold-formed, or cold-straightened shall be stress relieved before processing by heat treating for 5 h at 190 ± 15°C. Steel parts having an ultimate tensile strength greater than 2350 MPa (approximately 50 HRC) shall not be coated with lead or lead-tin alloys by electrodeposition.

9.2.3 Steel parts with ultimate tensile strength greater than 1125 MPa (approximately 35 HRC) and greater, shall be heat treated within 4 h after plating to remove hydrogen embrittlement. The heat treatment shall be at least for 3 h at 190 ± 15°C.

9.2.4 Defects and variations in appearance in the finish that arise from surface conditions of the substrate (for example, scratches, pores, roll marks, inclusions, etc.) and that persist in the finish despite the observance of good metal-finishing practices shall not cause for rejection.

9.3 Thickness—The minimum thickness on significant surfaces shall be 6, 12, 25, or 40 µm, as designated by the purchaser (see 8.1).

9.4 Significant Surfaces—Significant surfaces are defined as those normally visible (directly or by reflection) or are essential to the serviceability or function of the article; or can be the source of corrosion products or tarnish films that interfere with the function or desirable appearance of the article. When necessary, the significant surfaces shall be indicated on the drawings of the parts, or by the provision of suitably marked samples.

**Note 3**—Applied finishes generally perform better in service when the substrate over which they are applied is smooth and free of torn metal, inclusions, pores, and other defects. It is recommended that the specifications covering the unfinished products provide limits for these defects. A metal finisher can often remove defects through special treatments such as grinding, polishing, abrasive blasting, chemical etching, and electropolishing. However, these are not normal in the treatment steps preceding the application of the finish. When they are desired they shall be the subject of agreement between the buyer and the seller.

9.5 Corrosion Resistance—Lead and lead-tin coatings shall show neither corrosion products of lead (or lead-tin) nor basis metal corrosion products at the end of the test period, (see Table 2), when tested by continuous exposure to salt spray in accordance with 11.4. The appearance of corrosion products visible to the unaided eye at normal reading distance shall be cause for rejection except that corrosion products at the edges of specimens shall not constitute failure.

**Note 4**—Corrosion is defined as the presence of more than 66 rust spots per square meter, or more than two rust spots in an area less than 3 square decimeters, or rust spots larger than 1.6 mm in diameter.

9.6 Workmanship, Finish, and Appearance

10. Workmanship, Finish, and Appearance

10.1 The surface of the electroplated article shall be uniform in appearance and free of visible coating defects, such as blisters, pits, roughness, nodules, burning, cracks, or unplated areas, and other defects that will affect the function of the coating. However, superficial staining that results from rinsing or slight discoloration from any drying or baking operation to relieve hydrogen embrittlement, shall not be cause for rejection. On articles where a visible contact mark is unavoidable, its position shall be that chosen by the purchaser. The electroplated articles shall be clean and free of damage.

11. Test Methods

11.1 Composition—The lead-tin alloy coating deposit may be weighed and then analyzed for one of the metals. Lead content may be determined and the tin calculated by difference, for 1 g sample:

\[ \text{Tin} \% = \left( \frac{1 - L}{A} \times 100 \right) \]

\[ \text{Mass} \% \text{ Tin} = \left( \frac{W - L}{W} \times 100 \right) \]

where:

- \( L \) = weight of lead in sample, and
- \( A \) = weight of lead-tin alloy in sample.
- \( W = 1 \) g

**Note 4**—When significant surfaces are involved on which the specified thickness of finish cannot be readily controlled, it will be necessary to apply greater thickness on the more accessible surfaces, to use special

**TABLE 1 Lead-Tin Alloy Coating Composition**

<table>
<thead>
<tr>
<th>Element</th>
<th>Mass %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tin (Sn)</td>
<td>Up to 15 max</td>
</tr>
<tr>
<td>Lead (Pb)</td>
<td>Remainder</td>
</tr>
<tr>
<td>Other metals and nonmetals</td>
<td>1.0 max</td>
</tr>
</tbody>
</table>

**Lead Coating Composition**

| Lead                  | 99.0 |
| Other metals and nonmetals | 1.0 max |

**TABLE 2 Lead and Lead-Tin Alloy Coatings on Ferrous Alloys**

<table>
<thead>
<tr>
<th>Thickness, min µm</th>
<th>Salt Spray Resistance, h</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>200</td>
</tr>
<tr>
<td>25</td>
<td>96</td>
</tr>
<tr>
<td>12</td>
<td>48</td>
</tr>
<tr>
<td>8</td>
<td>24</td>
</tr>
</tbody>
</table>
11.1.1 A sample of the deposit can be obtained by plating on a stainless steel panel from which the coating can be peeled or by employing any recognized stripping method. The alloy composition of the deposit can be determined by methods such as gravimetric or volumetric analysis, density measurements, atomic absorption, spectrophotometry, X-ray fluorescence, and beta backscatter. In addition, the alloy composition produced by a plating solution may be obtained by comparing the weight of a coating deposited by a given number of ampere-hours to the weight of a lead coating produced in a lead coulometer in series with the plating bath.

11.2 Thickness—The thickness of the coating may be determined by Test Methods B 487, B 499, B 504, B 567, and B 568. The method used shall be applicable for the coating material and thickness specified.

NOTE 6—The thickness range of some of these methods may not include the specified thickness or the method may require the knowledge of the coating composition or density.

11.2.1 Other methods may be used if it can be determined that the uncertainty of the measurement is less than 10%.

11.3 Adhesion—Determine adhesion by any suitable procedure in accordance with Practice B 571.

11.4 Corrosion Resistance—When specified in 8.2, determine the corrosion resistance in accordance with Test Method B 117. Subject the selected samples to the salt spray test; the length of time to be applicable for the coating thickness shall be in accordance with the requirements of Table 2.

11.5 Hydrogen Embrittlement Relief—When specified in the contract or purchase order, prepare and test the satisfactory behavior of parts to indicate freedom from hydrogen embrittlement.

11.6 Visual Examination—Examine material for compliance with the requirements of Section 10 after electroplating.

12. Rejection and Rehearing

12.1 Articles that fail to conform to the requirements of this Specification may be rejected. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of a test, the producer or supplier may make a claim for a hearing.

13. Certification

13.1 The purchaser may require in the purchase order or contract that the producer or supplier give to the purchaser certification that the finish was produced and tested in accordance with this Specification and found to meet the requirements. The purchaser may similarly require that a report of the test results be furnished.

14. Keywords

14.1 electrodeposited coatings, lead and lead-tin alloys; lead/lead-tin alloy on steel/ferrous alloys

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirement shall apply only when specified by the purchaser as part of the purchaser’s order or contract and for all agencies of the United States government.

S1. Responsibility for Inspection

S1.1 The producer or supplier shall be responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein unless disapproved by the purchaser. The purchaser retains the right to perform any of the inspections and tests set forth in this specification where such inspections and tests are deemed necessary to ensure that supplies and services conform to the prescribed requirements.

APPENDIXES

(Nonmandatory Information)

X1. GUIDE TO SELECTION OF COATING THICKNESS

X1.1 Corrosion Protection:

X1.1.1 The results of atmospheric exposure tests on unalloyed lead coatings are given in Table X1.1. Tests completed under the auspices of ASTM3 show that a 19-µm coating of lead, electroplated directly on steel, has a life expectancy greater than 9 years under industrial conditions. Under marine conditions, for example, at the lot at Kure Beach, life expectancy of a 19-µm coating with a copper strike is 4 to 6 years. Under rural conditions, life expectancy of a 19-µm coating (with copper strike) is 7.5 to 9.5 years. Tests conducted at ASTM sites for the International Lead Zinc Research Organization, Inc., on lead coatings produced from a lead fluoborate electrolyte with hydroquinone as an addition agent, showed comparable results and also confirmed the beneficial effect of a copper strike.

X1.1.2 The effect of a copper strike on atmospheric corrosion protection is shown by the data in Table X1.1. In industrial, marine, and rural atmospheres, there is a significant improvement in corrosion resistance if a copper strike of minimum thickness 0.25 µm is used.

X1.1.3 Exposure to sulfuric acid and other aggressive industrial chemicals may require coating thicknesses of 40 µm and greater. Where there are exceptionally aggressive conditions or where mechanical damage is possible, then lead coating thicknesses between 125 and 250 µm may be required.

X1.1.4 Resistance to corrosion from less severe atmospheric conditions than those considered in X1.1.1 can be achieved with lead coatings 12 µm thick. Coatings less than 12 µm thick will normally be used in conjunction with paint coatings, etc., where exposure to an aggressive atmosphere is anticipated. The use, for corrosion protection, of coatings less than 12 µm thick is limited to indoor applications or to special situations owing to the possibility of damage during handling and possible increased porosity.

X1.1.5 The atmospheric corrosion resistance of lead-tin alloy coatings is not adversely affected by additions of tin up to 15 mass %.

X1.2 Where a nickel strike is employed, overheating during any joining operation may cause hot shortness and cracking of the nickel coating, from contamination by the lead and in the lead or lead-tin alloy plate.

X1.3 Lead-tin alloy (93 % Pb – 7 % Sn) is used for coating bearing surfaces.

X2. SUGGESTIONS FOR MEETING THE SPECIFICATIONS AS TO THICKNESS AND QUALITY OF THE COATINGS

X2.1 Ensure that the basis metal is properly cleaned.

X2.2 Use anodes of high purity such as 99.99 % of lead for lead plating. Grade A tin (see Specification B 339) and high-purity lead for the alloy anodes.

X2.3 Alloy anodes should not be left in the bath unless current is flowing. If this precaution is not observed, tin will be deposited from the bath onto the anode, and subsequently, it will flake off to cause roughness in deposition.

X2.4 To avoid buildup of impurities, work pieces should not remain in the bath unless current is flowing.

X2.5 Filtration should be continuous.

X2.6 Agitation should be employed. Lead baths should be agitated continuously to prevent concentration and temperature gradients in the solution.

X2.7 A carbon treatment during bath preparation is recommended to remove organic impurities.

X2.8 Demineralized (or deionized) water should be used for the last rinse prior to plating to prevent chloride and sulfate contamination.

X2.9 When hydroquinone is used as an addition agent,

---

### TABLE X1.1 Atmospheric Exposure Tests on Lead-Plated Steel

<table>
<thead>
<tr>
<th>Coating</th>
<th>Coating Thickness</th>
<th>Industrial Life</th>
<th>Marine Life</th>
<th>Rural Life</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>µm</td>
<td>mil</td>
<td>&lt;2</td>
<td>&lt;2</td>
<td>&lt;2</td>
</tr>
<tr>
<td>1.3 µm (0.05 mil)</td>
<td>6.5</td>
<td>0.25</td>
<td>6–&gt;9</td>
<td>&lt;0.8–1.5</td>
<td>&lt;0.8–1.0</td>
</tr>
<tr>
<td>Cu strike + lead</td>
<td>6.5</td>
<td>0.25</td>
<td>7–&gt;9</td>
<td>1.5–3.0</td>
<td>1.5–3.0</td>
</tr>
<tr>
<td></td>
<td>13</td>
<td>0.50</td>
<td>9–&gt;9</td>
<td>3.0–4.0</td>
<td>2.0–4.0</td>
</tr>
<tr>
<td>0.25 µm (0.10 mil)</td>
<td>13</td>
<td>0.50</td>
<td>9–&gt;9</td>
<td>3.5–4.0</td>
<td>5.0–7.0</td>
</tr>
<tr>
<td>Cu strike + lead</td>
<td>19</td>
<td>0.75</td>
<td>&gt;9</td>
<td>3.5–4.0</td>
<td>5.0–7.6</td>
</tr>
<tr>
<td></td>
<td>19</td>
<td>0.75</td>
<td>&gt;9</td>
<td>4.0–6.0</td>
<td>7.5–9.5</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>1.00</td>
<td>&gt;9</td>
<td>4.0–7.0</td>
<td>6.0–8.4</td>
</tr>
<tr>
<td>Cu strike + lead</td>
<td>25</td>
<td>1.00</td>
<td>&gt;9</td>
<td>5.0–10.0</td>
<td>&gt;10</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>1.50</td>
<td>&gt;9</td>
<td>0.8–4.0</td>
<td>1.0–8.0</td>
</tr>
<tr>
<td>Hot-dipped Pb</td>
<td>25</td>
<td>1.00</td>
<td>&gt;5</td>
<td>&gt;5</td>
<td>&gt;5</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>2.00</td>
<td>&gt;5</td>
<td>&gt;5</td>
<td>&gt;5</td>
</tr>
</tbody>
</table>

A Additions of tin up to 15 % do not adversely affect the corrosion resistance of the coatings.

B Ref 1—Program of the International Lead Zinc Research Organization, Inc.—5 years exposure. The ILZRO investigation was based on the use of fluoborate electrolytes with the addition agent hydroquinone.

Ref 2—ASTM program, 1944 Exposure Tests—Plated lead coatings.

Ref 3—ILZRO test in progress.
about 48 h are required at room temperature to allow an equilibrium between quinone and hydroquinone to be established. If this is not done, there may not be adequate grain refinement, and treeing may be excessive with thick deposits.

X2.10 Maintain regular control of all solutions and inspect the equipment at regular intervals, paying special attention to electrical contacts and accuracy of instruments.

X2.11 Maintain an inspection department, using the test methods prescribed in this specification in order to trace immediately the source of irregularities. On jobs running continuously over any length of time, the quality of the coatings on each part should be checked at least twice every shift after initial difficulties have been overcome.
Standard Practice for Testing Chromate Coatings on Zinc and Cadmium Surfaces

This standard is issued under the fixed designation B 201; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by the Department of Defense.

1. Scope

1.1 This practice covers a procedure for evaluating the protective value of chemical and electrochemical conversion coatings produced by chromate treatments of zinc and cadmium surfaces.

1.2 The protective value of a chromate coating is usually determined by salt-spray test and by determining whether or not the coating possesses adequate abrasion resistance.

1.3 Other methods, such as exposure to a humidity environment, can be used, but are generally of too long a duration to be of practical value. “Steam Tests” using pressure cookers have also been used for testing chromate films on hot-dip galvanized surfaces.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 117 Practice for Operating Salt Spray (Fog) Apparatus

3. Terminology

3.1 Descriptions of Terms:

3.1.1 time to failure—time to failure will depend on the type of coating tested. A list of some expected protective values obtainable in a given salt spray test is shown in Appendix X2.

3.1.1.1 Discussion—In most instances, failure is defined as the first appearance on significant surfaces of white corrosion products visible to the unaided eye at normal reading distance, except that the presence of white corrosion products at sharp edges (for example, on threaded fasteners) and at junctions between dissimilar metals should not be considered failure. In some instances, it may be desirable to regard the first appearance of red rust as failure.

3.1.2 significant surfaces—in general, significant surfaces are those surfaces that are visible and subject to corrosion or wear, or both, except that surfaces that are normally difficult to coat by electroplating or mechanical deposition may be exempt. The designation of significant surfaces may be indicated on the drawing.

4. Significance and Use

4.1 This practice is applicable to chromate coatings of the colorless (both one and two-dip), iridescent yellow or bronze, olive drab, black, colorless anodic, yellow or black anodic types, and of the dyed variety, when applied to surfaces of electrodeposited zinc, mechanically deposited zinc, hot-dipped zinc, rolled zinc, electrodeposited cadmium, mechanically deposited cadmium, and zinc die castings.

Note 1—Colorless coatings are also referred to as clear-bright or blue-bright coatings.

4.2 Because of variables inherent in the salt-spray test, which may differ from one test cabinet to another, interpretation of test results for compliance with expected performance should be specified by the purchaser.

4.3 Properties such as thickness, color, luster, and ability to provide good paint adhesion are not covered in this practice, nor are the chemical composition and the method of application of these finishes.

5. Conditioning

5.1 Aging—Before subjecting a chromate coating to test, it must be aged at room temperature in a clean environment for at least 24 h after the chromating treatment.

5.2 Preparation of Specimen—The test surface must be free of fingerprints and other extraneous stains and must not be cleaned except by gentle wiping with a clean, dry, soft cloth to remove loose particles. Oily or greasy surfaces should not be used for testing, and degreasing with organic solvents is not recommended.
6. Procedure

6.1 Salt Spray Test—Expose the clean specimen to a 5% solution salt spray and conduct the test in accordance with the latest revision of Practice B 117. Unless otherwise specified, only those surfaces that are positioned in the test chamber in accordance with Practice B 117 are considered pertinent for evaluating failure.

6.2 Abrasion Resistance Test—To determine whether the coating is adherent, nonpowdery, and abrasion resistant, rub the chromated surface with a gritless, soft gum eraser (art-gum) for 2 to 3 s by hand (about 10 strokes) using normal pressure (about 70 kPa (10 psi)) and a stroke approximately 50 mm long. The chromate coating should not be removed or worn through to the underlying metal as a result of this treatment.

6.3 Test for Colorless (Clear) Coatings—This test applies only to coatings that are free of secondary supplementary coatings, such as oil, water or solvent-based polymers, or wax.

6.3.1 Determine the presence of a colorless (clear) coating by placing a drop of lead acetate testing solution on the surface. Allow the drop to remain on the surface for 5 s. Remove the testing solution by blotting gently, taking care not to disturb any deposit that may have formed. A dark deposit or black stain is indicative of the absence of a coating.

6.3.2 Prepare the test solution by dissolving 50 g of lead acetate trihydrate (Pb(C₂H₃O₂)₂·3H₂O) in 1 L of distilled or deionized water. The pH of the solution should be between 5.5 and 6.8. Any white precipitate formed during the initial preparation of the solution may be dissolved by small additions of acetic acid; provided that the pH is not reduced to a value below 5.5. Upon formation of a white precipitate thereafter, the solution should be discarded.

6.3.3 For comparative purposes, treat an untreated surface similarly. On an untreated surface, a black spot forms almost immediately.

APPENDIXES

(Nonmandatory Information)

X1. NATURE OF COATINGS

X1.1 The primary purpose of chromate finishes is to retard the formation of white corrosion products upon exposure to stagnant water, moist atmosphere, or stagnant environments containing organic vapors, such as may emanate from certain plastics, paints, and other organic materials. Chromate finishes will not prevent the growth of metallic filaments, commonly known as “whiskers.”

X1.2 Coatings covered by this practice generally contain oxides of the basis metal and tri- and hexavalent chromium in varying proportions, except that colorless coatings contain little or no hexavalent chromium. They may be produced by either chemical or electrochemical processes from solutions containing hexavalent chromium compounds with one or more of certain anions which act as activators, film formers or both. There is evidence that over an extended period, chromate coatings undergo some chemical changes even under ordinary conditions. These changes increase with increase in temperature. At temperatures above approximately 65°C, these changes take place fairly rapidly, converting the soluble hexavalent chromium ion into an insoluble compound and thereby reducing its protective value under salt spray and humid conditions. Colorless or light iridescent coatings appear to be less sensitive to elevated temperatures than are heavy chromate coatings.

X1.3 The quality of the chromate film depends to a large extent on the chemical purity and the physical condition of the basis surface to which it is applied. In order to produce an acceptable coating, it is essential that the surface be properly cleaned and free of heavy metallic impurities such as lead, copper, and contamination (specific for zinc), such as brightener occlusions, and oxides, which interfere with the chromating reaction.

X1.4 The thickness of the coating to be chromated should be not less than 5.0 µm and the thickness requirement on the coating and chromated finish should apply after the chromate treatment. The color and luster produced by a given treatment will depend to some extent on the surface condition of the metal to which it is applied and may vary from part to part, or even on one single part.

X2. PROTECTION BY CHROMATE COATINGS

X2.1 Table X2.1 illustrates the minimum degree of protection that can be expected from the various types of chromate coatings on electrodeposited zinc when subjected to a 5% salt spray test.

X2.2 These values are shown for guidance purposes only and are not to be construed as endpoint requirements. All types of zinc and cadmium coatings can be chromated, and there may or may not be differences in the protection afforded by the chromate depending on the type of coating and the method of processing, so the actual protection required should be established to the satisfaction of the manufacturer and the purchaser.
**TABLE X2.1 Expected Protection**

<table>
<thead>
<tr>
<th>Type of Coating</th>
<th>Expected Minimum Hours to White Corrosion of Zinc</th>
</tr>
</thead>
<tbody>
<tr>
<td>One-dip colorless (clear bright)</td>
<td>12</td>
</tr>
<tr>
<td>Two-dip colorless (clear bright)</td>
<td>24</td>
</tr>
<tr>
<td>Black dip</td>
<td>48</td>
</tr>
<tr>
<td>Anodic-colorless</td>
<td>48</td>
</tr>
<tr>
<td>Anodic-black</td>
<td>96</td>
</tr>
<tr>
<td>Iridescent yellow or bronze</td>
<td>96</td>
</tr>
<tr>
<td>Anodic-yellow</td>
<td>150</td>
</tr>
<tr>
<td>Olive-drab</td>
<td>150</td>
</tr>
</tbody>
</table>
1. Scope

1.1 This test method covers the determination of the flow rate of metal powders and is suitable only for those powders that will flow unaided through the specified apparatus.

1.2 The values stated in SI units are to be regarded as the standard (except for the flowmeter funnel, which is fabricated in inch-pound units). The values given in parentheses are for information only.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 215 Practices for Sampling Finished Lots of Metal Powders
B 243 Terminology of Powder Metallurgy

3. Terminology

3.1 Definitions —Definitions of powder metallurgy terms can be found in Terminology B 243.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 flow rate, n—the time required for a powder sample of standard mass to flow through an orifice in a standard instrument according to a specified procedure.

4. Summary of Test Method

4.1 A weighed mass (50.0 g) of metal powder is timed as it flows through the calibrated orifice of a funnel.

5. Significance and Use

5.1 The rate and uniformity of die cavity filling are related to flow properties, which thus influence production rates and uniformity of compacted parts.

5.2 The ability of a powder to flow is a function of interparticle friction. As interparticle friction increases, flow is slowed. Fine powders may not flow.

5.3 Humidity and moisture content influence flow rate. Wet or moist powders may not flow.

5.4 This test method may be part of the purchase agreement between powder manufacturers and powder metallurgy (P/M) part producers, or it can be an internal quality control test by either the producer or the end user.

6. Apparatus

6.1 Powder Flowmeter Funnel—A flowmeter funnel (Fig. 1) having a calibrated orifice of 0.10 in. (2.54 mm) in diameter.

Note 1—The dimensions shown for the flowmeter funnel, including the orifice, are not to be considered controlling factors. Calibration with emery, as specified in Section 9, determines the working flow rate of the funnel.

6.2 Stand—A stand (Fig. 2) to support the powder flowmeter funnel.

6.3 Base—A level, vibration free base to support the powder flowmeter stand.

6.4 Timing Device—A stopwatch or other suitable device capable of measuring to the nearest 0.1 s.

6.5 Chinese Emery—An emery powder used to calibrate the flowmeter funnel.

6.6 Balance—A balance suitable for weighing at least 50.0 g to the nearest 0.1 g.

7. Sampling

7.1 A quantity of powder sufficient to run the desired number of flow tests shall be obtained in accordance with Practices B 215.

8. Preparation of Apparatus

8.1 Clean the funnel with clean dry toweling paper.

8.2 Clean the funnel orifice with a clean dry pipe cleaner.
9. Calibration of Apparatus

9.1 The manufacturer supplies the powder flowmeter funnel calibrated as follows:

9.1.1 Heat an open glass jar of Chinese emery in a drying oven at a temperature of 102° to 107°C (215° to 225°F) for 1 h.

9.1.2 Cool the emery to room temperature in a dessicator.

9.1.3 Follow the procedure outlined in steps 10.1.1-10.1.8.

9.1.4 Repeat steps 10.1.2-10.1.8 using the identical 50.0 g mass of emery for all the tests until 5 flow times, the extremes of which shall not differ by more than 0.4 s, have been recorded.

NOTE 2—The flow rate of Chinese emery calibration powder is sensitive to moisture. Starting from the dried condition, it will absorb moisture from the ambient air as five (or more) flow tests are performed. Flow times can vary dependent upon the humidity of the test area.

9.1.5 The average of these five flow times is stamped on the bottom of the funnel.

9.2 The flow rate of Chinese emery powder was established by an interlaboratory study conducted by Subcommittee B09.02 in 1995.4 It represents the flow rate through the master flowmeter funnel that had been used in a previous interlaboratory study with the former Turkish emery calibration powder, which is no longer available.

9.3 It is recommended that the flow rate be checked periodically, at least every six months, using the procedure outlined in steps 9.1.1-9.1.5. If the flow rate has changed from that stamped on the instrument, the new correction factor will be 40.0 divided by this new flow rate. Before adopting the new correction factor, however, it is recommended that the cause of the change be investigated. If the flow rate has increased (faster flow), it is probable that repeated use has burnished the orifice and the new correction factor may be used. A decrease in flow rate (slower flow) may indicate a plating of soft powder upon the orifice. This should be removed carefully with the aid of a pipe cleaner and the calibration test rerun, the new correction factor being calculated if required. It is recommended that the use of a funnel be discontinued after the flow rate of the emery has increased such that the time of flow is less than 37 s.

10. Procedure

10.1 Method 1—Stationary Powder Start to Flow Measurement:

10.1.1 Weigh out a 50.0 g mass of powder, as sampled, into a clean weighing dish.

10.1.2 Block the discharge orifice at the bottom of the funnel with a dry finger.

10.1.3 Carefully pour the 50.0 g sample of powder into the center of the flowmeter funnel without any tapping, vibration or movement of the funnel.

10.1.4 Place the emptied weighing dish on the flowmeter stand directly under the funnel orifice.

10.1.5 Simultaneously start the stopwatch and remove your finger from the discharge orifice.

10.1.6 If the powder fails to start flowing, one light tap on the funnel rim is permitted. Further tapping of the funnel,
however, or poking or stirring of the powder in the funnel with a wire or any other implement is not permitted.

10.1.7 Stop the stopwatch the instant the last of the powder exits the orifice.

10.1.8 Record the elapsed time to the nearest 0.1 s.

10.1.9 More than one flow may be run if desired. Use a fresh 50.0 g quantity of powder for each flow test. Average the flow times.

10.2 Method 2—Moving Powder Start to Flow Measurement:

10.2.1 Place an empty receptacle directly under the discharge orifice.

10.2.2 Weigh out a 50.0 g mass of powder, as sampled, into a clean weighing dish.

10.2.3 Pour the 50.0 g powder specimen into the center of the funnel and start the stopwatch the instant the powder exits the orifice.

10.2.4 Stop the stopwatch the instant the last of the powder exits the orifice.

10.2.5 Record the elapsed time to the nearest 0.1 s.

10.2.6 More than one flow may be run if desired. Use a fresh 50.0 g quantity of powder for each flow test. Average the flow times.

11. Calculation

11.1 Calculate the correction factor by dividing 40.0 by either the flow rate stamped on the bottom of the funnel, or the new calibration flow rate established in 9.3.

11.2 Multiply the elapsed time (see 10.1.9 or 10.2.6) by the desired correction factor (see 11.1).

12. Report

12.1 Report the corrected flow rate in seconds to the nearest second.

13. Precision and Bias

13.1 Precision—Precision has been determined from an interlaboratory study performed by seven laboratories of Subcommittee B09.02.4

13.1.1 Repeatability intervals, $r$, are listed in Table 1. In 95 % of flow rate determinations, on the basis of test error alone, duplicate tests in the same laboratory by the same operator on one homogeneous lot of powder will differ by no more than the stated amount in seconds.

13.1.2 Reproducibility intervals, $R$, are listed in Table 1. For 95 % of comparative trials done in two different laboratories, and on the basis of test error alone, single tests on the same homogeneous lot of powder will differ by no more than the stated amount in seconds.

13.2 Bias—No statement can be made about bias, because there is no standard reference material for flow rate measurement.

14. Keywords

14.1 flow rate; metal powder flow; powder flow

### TABLE 1 Precision of Flow Rate Measurements of Metal Powders

<table>
<thead>
<tr>
<th>Powder</th>
<th>Apparent Density (g/cm³)</th>
<th>Flow Rate x Avg. (s/50 g)</th>
<th>Repeatability ($r$) Avg. 3 Flows (s)</th>
<th>Reproducibility ($R$) Avg. 3 Flows (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spherical bronze</td>
<td>5.04</td>
<td>12</td>
<td>0.1</td>
<td>0.8</td>
</tr>
<tr>
<td>Iron #1</td>
<td>2.46</td>
<td>31</td>
<td>0.7</td>
<td>2.5</td>
</tr>
<tr>
<td>Iron #2</td>
<td>3.03</td>
<td>26</td>
<td>1.0</td>
<td>2.1</td>
</tr>
<tr>
<td>Iron (lubricated)</td>
<td>3.18</td>
<td>26</td>
<td>1.3</td>
<td>2.2</td>
</tr>
<tr>
<td>Bronze premix (lubricated)</td>
<td>3.31</td>
<td>31</td>
<td>1.7</td>
<td>2.6</td>
</tr>
<tr>
<td>Brass (lubricated)</td>
<td>3.61</td>
<td>42</td>
<td>4.1</td>
<td>8.7</td>
</tr>
</tbody>
</table>

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Standard Test Method for
Sieve Analysis of Metal Powders

This standard is issued under the fixed designation B 214; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of the dry sieve analysis of metal powders.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 212 Test Method for Apparent Density of Free-Flowing Metal Powders Using the Hall Flowmeter Funnel
B 215 Practices for Sampling Finished Lots of Metal Powders
B 243 Terminology of Powder Metallurgy
E 11 Specification for Wire-Cloth Sieves for Testing Purposes
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 MPIF Standard:
MPIF 05 Determination of Sieve Analysis of Metal Powders

3. Terminology

3.1 Definitions—Useful definitions of terms for metal powders and powder metallurgy are found in Terminology B 243.

4. Significance and Use

4.1 The particle size distribution of a metal powder affects its behavior in P/M processing and other applications of these materials. The test method may be part of the purchase agreement between powder manufacturer and user, or it may be an internal quality control test for either. This test method is appropriate for materials with size distributions typified by metal powders used in powder metallurgy.

5. Apparatus

5.1 Sieves—A set of standard sieves selected from Table 1 of Specification E 11, or the equivalent Tyler standard sieves. The sieves shall be 8 in. (203.2 mm) in diameter and either 1 or 2 in. (25 or 50 mm) in depth and fitted with bronze, brass, stainless steel, or other suitable wire cloth. The U.S. sieves given in Table 1 of this method shall conform to Specification E 11. If Tyler standard sieves are substituted, they shall conform to the permissible variations given in Table 1 of Specification E 11.

NOTE 1—The new U.S. Series standard sieves, adopted in 1970, are the preferred sieves to use. The old U.S. Series standard sieves and equivalent sieves manufactured by other companies, such as Tyler, may also be used if the new U.S. Series is not available. Care should be taken to make sure that sieve opening sizes are correct when performing standardization work.

5.2 Sieve Shaker—A mechanically operated, single eccentric sieve shaker which imparts to the set of sieves a rotary motion and tapping action of uniform speed, shall be used. The number of rotations per minute shall be between 270 and 300. The number of taps per minute shall be between 140 and 160. The sieve shaker shall be fitted with a plug to receive the impact of the tapping device. The entire apparatus shall be rigidly mounted by bolting to a solid foundation, preferably of concrete. A time switch should be provided to ensure accuracy of duration of the test.

NOTE 2—Use of a sound proof enclosure is recommended.

5.3 Balance—A balance having a capacity of at least 110 g and a sensitivity of 0.01 g.

6. Test Specimen

6.1 The size of the test specimen shall be 90 to 100 g for any metal powder having an apparent density greater than 1.50 g/cm³ when determined in accordance with Test Method B 212. A 40 to 60-g specimen shall be used when the apparent density of the powder is less than 1.50 g/cm³. The test specimen should be obtained in accordance with Practices B 215.

7. Procedure

7.1 Assemble the group of sieves selected in consecutive
9. Precision and Bias 

9.1 Precision:

[6] The precision contained in this standard was determined by the Metal Powder Producers Association Standards Committee of the Metal Powder Industries Federation for MPIF Standard 05. The precision is used herein with the permission of the Metal Powder Industries Federation, 105 College Road East, Princeton, NJ 08540-6692, USA.

9.1.1 An interlaboratory study of the sieve analysis of metal powders was run in 1993 and 1994 using the procedures contained in MPIF Standard 05 (1992). Each of twelve laboratories made three tests on four powder samples using each of two sets of sieves. One set of sieves was a standard set that was circulated to each laboratory in turn. A second set of sieves was chosen by each laboratory from its in-house sieve stock. Practice E 691 was followed for the design and analysis of the data. The details are given in MPPA Research Report MPPA R-05-95.5

9.1.2 There were five U.S. Series standard sieves in each set of sieves. For in-house sieves (or by analogy matched sieves obtained from an accredited manufacturer, mesh plus a cover and a pan. The sum of the masses of all the fractions shall not be less than 99 % of the mass of the test specimen. Add the masses of all the fractions shall not be less than 99 % of the mass of the test specimen. Add the masses of all the fractions shall be calculated from the data. The details are given in MPPA Research Report MPPA R-05-95.5

9.1.3 The precision information given in 9.1.4-9.1.7 covers the percent retained between any pair of sieves, the percent retained on the coarsest sieve, the percent passing the fitness sieve, and the cumulative percentages calculated from all sieves of greater openings above any sieve in the set.

9.1.4 The 95 % repeatability limit, r, (within a laboratory) is represented by the equation:

\[ r = 0.4 + 0.03 \times [SF] \] (1)

where [SF] is the % retained on the sieve of interest.

9.1.5 The 95 % reproducibility limit, R, (between laboratories) is smaller for the circulated sieves than for the in-house sieves. For in-house sieves R can be calculated from the following equations:

\[ R = 1.2 + 0.15 \times [SF] \text{ for } [SF] \text{ from } 0 \text{ to } 22 \] (2)

\[ R = 4.5 \text{ for } [SF] \text{ from } 22.1 \text{ to } 50 \] (3)

where [SF] is the % retained on the sieve of interest.

For circulated sieves (or by analogy matched sieves obtained
by two laboratories) $R$ can be calculated from the following
equations:

$$R = 0.3 + 0.064 \times [SF] \text{ for } [SF] \text{ from } 0 \text{ to } 30 \quad (4)$$

$$R = 2.2 \times [SF] \text{ from } 30.1 \text{ to } 50 \quad (5)$$

Where $[SF]$ is the % retained on the sieve of interest.

9.1.6 Duplicate results from the same laboratory should be
considered acceptable at the 95 % confidence level unless they
differ by more than $r$, the repeatability interval.

9.1.7 Duplicate results from the different laboratories should
be considered acceptable at the 95 % confidence level unless
they differ by more than $R$, the reproducibility interval.

9.2 Bias—No information can be presented on bias in test
method B 214 for sieve analysis since there are no universally
accepted standard sieves.

10. Keywords

10.1 mesh designation number; particle size; screened frac-
tion; sieves; Tyler; U.S. standard series

APPENDIXES

(Normandy Information)

X1. CERTIFIED SIEVES—MASTER SET

X1.1 Sieves conforming to Specification E 11 can be
obtained from the sieve manufacturers, and arrangements can
be made through them to have the sieves certified by the
National Institute of Standards and Technology (formerly the
National Bureau of Standards). If used continually, the sieves
will, after a period of time, become less accurate and might no
longer be acceptable as certified sieves. The common practice
which would be considered acceptable according to this
standard, would be to use the certified sieves as a master set for
checking other working sets of sieves. By comparing sieve
tests on the same sample, run in both the master set and the
working set, a factor can be established for correcting results
on the working sieves.

X2. MATCHED SIEVES

X2.1 The use of a matched set of sieves, established
through use of a standard powder, is recommended when closer
correlation of tests between supplier and consumer is desired.

X3. SIEVE SERIES GUIDELINES

X3.1 Suggested combinations of sieves are given in Table
X3.1 for several nominal mesh size metal powders.

| TABLE X3.1 Suggested Sieve Series for Metal Powders |
|---------------------------------|--------|--------|--------|--------|--------|--------|--------|
| Nominal Powder Mesh Size        | 20     | 40     | 60     | 100    | 140    | 200    | 325    |
| New U.S.                        |        |        |        |        |        |        |        |
| 20                              | ✓      | ✓      |        |        |        |        |        |
| 40                              | ✓      | ✓      | ✓      |        |        |        |        |
| 60                              | ✓      | ✓      | ✓      | ✓      |        |        |        |
| 80                              |        |        |        |        |        |        |        |
| 100                             |        | ✓      | ✓      | ✓      | ✓      | ✓      | ✓      |
| Series                          |        |        |        |        |        |        |        |
| 140                             |        |        |        |        |        |        |        |
| 200                             |        |        |        |        |        |        |        |
| 230                             |        |        |        |        |        |        |        |
| 325                             |        |        |        |        |        |        |        |
| Pan                             |        |        |        |        |        |        |        |

7 Contact the National Institute of Standards and Technology, Gaithersburg, MD 20899.
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Standard Practices for Sampling Finished Lots of Metal Powders

This standard is issued under the fixed designation B 215; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These practices cover two procedures for selecting representative samples of metal powders for subsequent testing:

1.1.1 **Practice A**—For powders in the process of being packaged from blenders or storage tanks.

1.1.2 **Practice B**—For powders already packaged in containers.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 243 Terminology of Powder Metallurgy

3. Terminology

3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243. Additional descriptive information is available in the Related Materials section of Vol 02.05 of the Annual Book of ASTM Standards.

3.2 Description of Terms:

3.2.1 **composite sample**—the blended entire gross sample or a representative part thereof.

3.2.2 **gross sample**—a quantity of powder consisting of all the increments taken from a single lot.

3.2.3 **increment**—a quantity of powder obtained by a sampling at one time from a single lot.

3.2.4 **lot**—a definite quantity of powder processed or produced under uniform conditions.

3.2.5 **test portion (test specimen)**—a defined quantity of powder actually drawn from the test sample and on which the test is actually performed.

3.2.6 **test sample**—a quantity of powder taken from the composite sample for determining a single property or for preparing the test pieces. It normally should be taken by splitting the composite sample.

4. Significance and Use

4.1 Since many tests are performed using very small amounts of powder, it is most important that the test portion be obtained in a standardized manner. The practices described here take into account the possibility of segregation of the metal powder during the filling of containers and after the containers are full.

PRACTICE A

5. Apparatus

5.1 **Rectangular Receptacle.** capable of being moved completely across the stream of flowing powder at a constant speed and having a length and width greater than the stream of powder. It must be large enough that there is no overflow when collecting the sample.

5.2 **Small Blender.**

5.3 **Sample Splitter (+).**

6. Procedure

6.1 Pass the rectangular receptacle completely through the stream of flowing powder at a constant speed. If the entire contents of one lot of powder is being packed in a single container, take increments when the container is ¼, ½, and ¾ filled. If several containers are to be filled by one lot of powder, take the first increment when the first container is ½ filled, the second increment in the middle of the run, and the third increment near the end of the run. Additional increments may be agreed upon by the parties concerned. The total of all increments shall be at least 5000 g.

6.2 Blend the gross sample composed of at least 5000 g in a small blender for 10 to 15 revolutions.
6.3 Pass the blended gross sample or composite sample through a sample splitter to form a number of test samples depending on the design of the splitter. Then use these test samples to supply the test portions for subsequent testing.

7. Frequency of Increments
7.1 Take at least three increments for each lot of powder. The actual number depends on the size of the lot and the powder involved and shall be agreed upon by the parties involved.

PRACTICE B

8. Apparatus
8.1 Keystone Sampler (Fig. 1).
8.2 Sample Splitter (Fig. 2).

9. Procedure
9.1 Insert the Keystone sampler in the closed position into the contained powder through a point 70% of the distance between the center of the cross section and the periphery down to the bottom of the container. Then twist the sampler to open the slots allowing powder to flow into the sampler. When filled, the slots are closed. Remove the sampler, and empty the contents into a suitable container.
9.2 When a suitable number of increments have been taken, blend them for 10 to 15 revolutions to obtain a composite sample.
9.3 Pass the composite sample through a sample splitter to obtain the test samples.
9.4 Obtain the test portion from the test sample by using a micro sample splitter or by quartering.

10. Frequency of Increments
10.1 See Table 1.

11. Keywords
11.1 blender; composite sample; Keystone sampler; sample splitter; sampling segregation

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3 The Keystone Sampler is available from Gamet Mfg. Co., 6237 Penn Ave., Minneapolis, MN 55423.

<table>
<thead>
<tr>
<th>No. of Containers in the Lot</th>
<th>No. of Containers to be Sampled</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 to 5</td>
<td>all</td>
</tr>
<tr>
<td>6 to 15</td>
<td>5</td>
</tr>
<tr>
<td>16 to 35</td>
<td>7</td>
</tr>
<tr>
<td>36 to 60</td>
<td>8</td>
</tr>
<tr>
<td>61 to 99</td>
<td>9</td>
</tr>
<tr>
<td>100 to 149</td>
<td>10</td>
</tr>
<tr>
<td>150 to 199</td>
<td>11</td>
</tr>
<tr>
<td>200 to 299</td>
<td>12</td>
</tr>
<tr>
<td>300 to 399</td>
<td>13</td>
</tr>
<tr>
<td>More than 400</td>
<td>13 + 1 per 100 additional containers</td>
</tr>
</tbody>
</table>
1. Scope

1.1 This guide is intended as an aid in establishing and maintaining a preparatory cycle for electroplating on high-carbon steel (Note 1) producing a minimum of hydrogen embrittlement and maximum adhesion of the electrodeposited metal. For the purpose of this guide, steels containing 0.35% of carbon or more, and case-hardened low-carbon steel, are defined as high-carbon steels. There is no generally recognized definite carbon content dividing high from low-carbon steels for electroplating purposes.

Note 1—Electroplating of plain high-carbon steel introduced problems not found in similar operations on low-carbon steel. During the cleaning and electroplating cycle, high-carbon steel differs from low-carbon steel in regard to its greater tendency to become embrittled and the greater difficulty in obtaining maximum adhesion of the electrodeposit. The preparation of low-carbon steel for electroplating is covered in Practice B 183.

1.2 This guide does not apply to the electroplating of alloy steel. For methods of chromium electroplating directly on steel see Guide B 177.

1.3 This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For a specific hazards statement, see 3.1.

2. Referenced Documents

2.1 ASTM Standards:

B 177 Practice for Chromium Electroplating on Steel for Engineering Use
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 849 Specification for Pre-Treatments of Iron or Steel for Reducing the Risk of Hydrogen Embrittlement
B 850 Specification for Post-Coating Treatments of Iron or Steel for Reducing the Risk of Hydrogen Embrittlement

3. Reagents

3.1 Purity of Reagents—All acids and chemicals used in this practice are technical grade. Acid solutions are based upon the following assay materials:
- Hydrochloric acid (HCl) 31 mass %, density 1.16 g/mL
- Nitric acid (HNO₃) 67 mass %, density 1.40 g/mL
- Sulfuric acid (H₂SO₄) 93 mass %, density 1.83 g/mL

Caution—Dilute sulfuric acid by slowly adding it to the approximate amount of water required with rapid mixing. After cooling, bring the mixture to exact volume.

3.2 Purity of Water—Use ordinary industrial or potable water for preparing solutions and rinsing.

4. Nature of Steel

4.1 Hardness—High hardness is a major cause of cracking of the steel during or after electroplating. The recommended maximum hardness range for classes of products depends on their geometry and service requirements (Note 2). Parts hardened by heat treatment should be inspected before electroplating for the presence of cracks by a suitable method, such as magnetic or fluorescent powder inspection.

Note 2—Some examples of parts and Rockwell hardness ranges are as follows:

<table>
<thead>
<tr>
<th>Rockwell Hardness Range</th>
<th>Springs</th>
<th>Spring washers</th>
<th>Small instrument parts</th>
<th>Parts to be chromium electroplated for engineering use</th>
</tr>
</thead>
<tbody>
<tr>
<td>C45 to C48</td>
<td>C45 to C53</td>
<td>C52 to C55</td>
<td>C57 to C62</td>
<td></td>
</tr>
</tbody>
</table>

4.2 Hydrogen Embrittlement—Difficulties resulting from hydrogen embrittlement increase with increasing hardness, whether produced by heat treatment or cold work. Difficulties, during or after electroplating of hardened high-carbon steel parts, may in some cases be minimized without material change in hardness by baking before final pretreatment. For a listing of such hydrogen embrittlement relief bake cycles, consult Specification B 850.

4.3 Surface Oxidation—In order that subsequent treatments be facilitated, every reasonable precaution should be taken throughout the processing to limit oxidation or scale formation. In particular cases pre-electroplating with copper to a minimum thickness of 13 µm may assist in maintaining a preferred surface through the heat treatment. A nonoxidizing atmosphere
should be maintained in the furnace. This copper shall be
removed prior to the regular electroplating cycle. Care should
be used in oil-quenching parts heat treated in a salt bath, to
prevent the charring effect that can be caused by salt-bath
drag-out. Proper lead-bath quenching results in only slight
oxidation.

4.4 Steel Quality—The quality of the steel should be char-
acteristic of the requirement of the product and the electroplat-
ing operation. The steel should be free of injurious surface
defects, and of at least average cleanliness.

5. Preparation of Steel, General

5.1 Preparatory Treatments—A wide variety of surface
conditions are encountered in high-carbon steel articles to be
electroplated. The surface may require the removal of one or
more of the following contaminants: grease, oil or drawing
compounds, burned-in oil scale, light to heavy treatment scale,
permeable oxide films, emery and fine steel particles resulting
from the grinding operation. The removal of such contaminants
is accomplished by one or more of the following pretreatment
procedures where applicable:

5.1.1 Substantial removal of oil, grease, and caked-on dirt
by precleaning before the part enters the electroplating cycle
(applicable in all cases).

5.1.2 Mechanical treatment of the surface by tumbling, sand
or grit blasting, vapor blasting, or grinding (optional).

5.1.3 Final and complete anodic cleaning in an electrolytic
alkali cleaner.

5.1.4 Acid treatment in HCl to remove the last trace of oxide
and scale. This should be avoided for spring temper and
case-hardened parts. This treatment also removes residual
traces of lead that may be present following proper lead-bath
quenching.

5.1.5 Smut removal by cyanide dipping or by anodic treat-
ment in cyanide or alkali.

5.1.6 Final preparation for electroplating may be accom-
plished by an anodic etching treatment in H₂SO₄ (used when-
ever possible in the interest of high yield and adhesion).

5.1.7 Conditioning of the surface to be electroplated may be
accomplished, where necessary for the electroplating process,
by a short dip or rinse in a solution equivalent to the
electroplating solution without its metallic content.

5.2 Rinsing—Inadequate rinsing after each solution treat-
ment step is the recognized cause of a large portion of
electroplating difficulties. Not enough rinsing is characteristic
of most pretreatment cycles.

5.3 Pretreatment Time—All processing steps involving hy-
drogen generation must be designed to operate for a minimum
length of time, to avoid hydrogen embrittlement of the high-
carbon steel.

5.4 Control—All pretreatment steps should be carried out
with solutions that are maintained in good working condition
by control of composition and contaminants, and used under
conditions of time, temperature and current density specified to
meet the requirement of the work being processed.

5.5 Pretreatment Cycle Design—Depending upon the re-
quirements for the particular high-carbon steel parts to be
electroplated, a minimum cycle should be selected from the
general steps listed in 5.1. Different classes of materials require
selected process steps combined into pretreatment cycles of
greater or less complexity according to the condition and
properties of the material. The minimum number of steps
necessary to accomplish the electroplating satisfactorily is
recommended.

6. Preliminary Pretreatment Procedures

6.1 Application—Degreasing and mechanical surface treat-
ment are necessary only where the high-carbon steel parts are
contaminated to such an extent that otherwise the burden
imposed on the pretreatment cycle would impair its efficiency,
increase its complexity, and tend to prevent the attainment of
the required quality of the deposit. The overall cost of the
electroplating process is usually reduced by using the prelimi-
nary treatments where applicable. Oil, grease, dirt, drawing
compounds, burnt-in oil, heavy scale, and emery and steel
particles are typical of the gross contaminants encountered.

6.2 Precleanning—Solvent-degreasing with clean solvent,
Spray-Washing, or emulsion-cleaning, followed by electrolytic
or soak-alkali cleaners are recommended. The former types are
prefered to reduce the burden on the alkali treatments.
Soak-alkali cleaning is usual for parts that are to be barrel
electroplated. Electrolytic cleaning should always be anodic
where the control of embrittlement is a problem.

6.3 Stress Relief Treatment—It is recommended that hard-
ened high-carbon steel parts receive a stress-relief bake before
the parts are mechanically pretreated or enter the final pretreat-
ment cycle, or both. For a listing of typical stress-relief bakes,
consult Specification B 849.

6.4 Mechanical Treatment—The purpose of mechanical
treatment is to reduce subsequent acid pickling to a minimum.
Where mechanical treatment has been accomplished with
precision, it is sometimes possible to eliminate acid pickling
entirely, thus improving the control of hydrogen embrittlement.
When required, mechanical treatment of small parts is best
effectted by tumbling. All scaled and nearly all oil-quenched
materials require mechanical cleaning such as by tumbling
with or without abrasive, or by sand, grit, or vapor blasting.
These operations should be carried out so as to avoid severe
roughening of the surface with accompanying notch effect.
One resorts to grinding in certain cases where the surface
smoothness or dimensions of the parts are of critical impor-
tance, for example, in chromium electroplating for engineering
use.

7. Final Pretreatment Procedures

7.1 Application—Final cleaning, oxide removal, and anodic
acid treatment are fundamental steps required for preparing
high-carbon steel for electroplating. These pretreatment steps
are designed to assist in the control of hydrogen embrittlement
and in securing the maximum adhesion of the electroplated
coating.

7.2 Electrolytic Anodic Cleaning:

7.2.1 All work, except work to be barrel electroplated,
should preferably be cleaned in an electrolytic anodic alkali
cleaner. Anodic cleaning is recommended to avoid hydrogen
embrittlement that is likely to result from cathodic cleaning. An
exception is barrel work which, because of the work size, is
preferably cleaned by soaking or tumbling in an alkaline
cleaning solution without the use of current.

7.2.2 The purpose of this cleaning step is to remove completely the last traces of contaminants. In all cases it should be preceded by heavy-duty precleaning as covered in 6.2.

7.2.3 The electrolytic anodic cleaner should be used at a temperature of 90°C or higher, and at a current density of 5 A/dm² or higher, in order that the required degree of cleanliness be obtained in a time period not exceeding 2 min.

7.2.4 On removal from the cleaner, the work should be thoroughly rinsed, first with water warmed to 50°C, and then in a cold-water spray at room temperature, prior to the acid dip.

7.3 Rinsing:

7.3.1 The most thorough fresh-water rinsing operation possible is mandatory after each processing step if the best results in electroplating high-carbon steel are to be obtained. The purpose of rinsing is to eliminate drag-over by complete removal of the preceding solution from the surface of the work. Many existing commercial operations are characterized by inadequate rinsing.

7.3.2 Warm to hot rinses should be used following alkaline solutions or where the subsequent processing solution is hot. The rinse temperature should not be so high as to induce drying of the steel surface between processing steps. Room temperature rinses are suitable for use following acid solutions where the solution in the next processing step is cold. In no case should very cold water be used for rinsing.

7.3.3 The recommended rinsing practice includes the use of an immersion rinse, always followed by a spray rinse of fresh water at the required temperature. Not using a spray rinsing is an invitation to trouble in the electroplating of high-carbon steel.

7.4 Hydrochloric Acid Treatment—The purpose of the HCl treatment is to remove completely the last trace of oxide from the surface of the high-carbon steel. The intensity of the HCl treatment should be held to the minimum required by the nature and amount of oxide present. The use of H₂SO₄ instead of HCl is not recommended for descaling high-carbon steel because of its smut-forming tendency, in spite of the somewhat lowered tendency to rusting of H₂SO₄-treated surfaces. The addition of wetting agents to the HCl solution is not recommended. Care and caution must be exercised in the use of inhibitors where they are required, because they sometimes interfere with adhesion. Inhibitors are of benefit only in special cases where surface finish and dimensions are of prime importance.

7.5 Treatment for Smut Removal—When the HCl treatment of the high-carbon steel results in the presence of smut, the smut must be removed before the surface is electroplated. Light oxides formed on exposure to air after acid treatment must likewise be removed. This can be done by an anodic cyanide or alkaline treatment. Air-formed oxide, if not too heavy, can be removed by a cyanide dip after the rinse following the acid treatment. A concentration of 22 g/L of NaCN is sufficient for the cyanide dip. Where a severe smut condition exists, it can be eliminated by a ½ to 1-min anodic treatment at 1.5 to 2 A/dm² in a solution of a NaCN of the noncritical concentration of 45 g/L used at room temperature. An alternative treatment for a somewhat lighter smut condition is electrolytic anodic treatment in the noncyanide alkaline cleaning solution (6.3) above 70°C, for 15 to 30 s at 2.5 to 5 A/dm². The current density is not critical.

7.6 Anodic Acid Etching:

7.6.1 The use of an anodic acid etch and subsequent rinse as final steps in the preparation for electroplating of high-carbon steel is of importance in securing adhesion. Without such an anodic treatment, poor adhesion may occur. The anodic acid treatment is capable of removing a small amount of smut formed by the preceding HCl treatment; more substantial amounts of smut should be removed according to the procedures described in 6.5.

7.6.2 A 150 to 600 mL/L H₂SO₄ solution used at a temperature of not more than 30°C, and preferably below 25°C, is effective for anodic etching of high-carbon steel. See Caution in 3.1. The addition of 125 g/L of Na₂SO₄ (based on the anhydrous salt) is of benefit for many steel grades. Anodic treatment in this solution for a time usually not exceeding 1 min at a current density of 16 A/dm² (range of 10 to 43 A/dm²) is sufficient. A high acid content, high current density, and low temperature (with reference to the ranges specified) will minimize the attack on the basis metal and produce a smoother surface. This H₂SO₄ solution is very stable and not affected appreciably by iron build up. Besides securing adhesion of the subsequent electrodeposit, it improves the uniformity of the deposit and reduces hydrogen embrittlement.

7.6.3 A dip for 5 to 10 s in a 55 mL/L HNO₃ solution, followed by rinsing and anodic cyanide treatment for smut removal, has in certain cases been found effective. Although nonelectrolytic, the HNO₃ treatment requires an additional electrolytic cyanide treatment before electroplating.

7.7 Electropolishing:

7.7.1 Electropolishing is used to remove highly stressed metal and metal debris from the surface of cold-worked steel, thereby improving the bond strength and corrosion resistance of electroplated coatings. It accomplishes this function without the tendency to form smut, which may result from anodic etching. Because it does not etch the steel, it is preferred by some electroplaters to anodic etching for preparing steel surfaces for decorative electroplating. Proprietary mixtures of phosphoric and sulfuric acids³ for electropolishing are of special interest in view of their ability to remove smut from cast iron surfaces. The addition of chromic acid to sulfuric-phosphoric acid mixtures⁴ further provides a surface passivation that is beneficial in preventing rusting during transfer or during temporary storage of steel prior to electroplating.

7.7.2 An activating treatment after electropolishing can be beneficial to subsequent electroplating. An example of a suitable activating treatment is anodic cleaning followed by acid dipping. Electropolishing can be used in addition to or instead of anodic etching.

7.7.3 Electropolishing of fine-grained steel provides a uniform, smooth finish. Electropolishing of a coarse-grained structure (No. 6 or coarser) results in less smoothness. In either case, however, a word of caution must be interjected, namely

that any seams, voids, stringers, and other surface defects can be damaging to the appearance of the electroplated coating. Yet, the removal of sharp edges or scratches and the removal of nonmetallic inclusions in seams and stringers can reduce the harmful effect of these defects on the corrosion resistance of the electroplated steel.

8. Electroplating Procedures

8.1 Standard electroplating procedures can be used on high-carbon steel when the proper preparatory steps described in Sections 6 and 7 have been selected and followed. The conditions of use of the electroplating bath should assure a minimum evolution of hydrogen at the cathode surface (highest cathode current efficiency). The use of the minimum length of time in each step of preparation and electroplating is recommended. The material should be handled with the minimum number of steps consistent with proper treatment.

8.2 The electrodeposition of tin and cadmium on high-carbon steel is easier to accomplish than that of zinc. Electrodeposition of nickel and chromium is not difficult, if the recommended practice is followed. Low internal stress of the deposit is desirable.

8.3 Springs and similar materials should not be electroplated while subject to externally applied stress.

9. Heat Treatment After Electroplating

9.1 Application—One purpose of the special preparatory treatments of high-carbon steel (Sections 6 and 7) is to minimize hydrogen embrittlement. In most commercial production it is necessary to bake the electroplated work for final embrittlement relief. With springs and similar materials, precautions should be observed to avoid flexing the articles before they are baked.

9.2 Procedure—The hydrogen may be largely removed and the physical properties of the steel substantially restored by heating, for example, for 1 to 5 h at temperature in an oven maintained at a temperature of 150 to 260°C, the temperature and length of treatment depending on the severity of embrittlement, the cross section of the article, the requirements of the steel, and the kind and thickness of the electrodeposited coatings. The baking should be done as soon as possible after electroplating, and before any supplementary chemical treatment of the electroplated surfaces. The best time and temperature in some cases must be established experimentally. A temperature not exceeding 205°C is recommended for zinc- or cadmium-electroplated articles. A lower temperature may be required if the coating is to be given a subsequent chemical treatment.

10. Test Methods

10.1 Adhesion—No universally satisfactory nondestructive test for adhesion is known. Poor adhesion may be revealed during grinding operations.

10.2 Embrittlement—The test for the effectiveness of the procedures used to control embrittlement lies in the subsequent service use of the material. Applicable control tests for embrittlement can be selected by analogy to the particular service in which the high-carbon steel article being processed will be used.
1. Scope

1.1 This terminology standard includes definitions that are helpful in the interpretation and application of powder metallurgy terms.

2. Referenced Documents

2.1 ASTM Standards:

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1 This terminology is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.01 on Nomenclature and Technical Data.

3. Terminology

3.1 Powder—Terms associated with production, characterization, use, and testing of metal powders.

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3.1.2 General:

1001 agglomerate, n—several particles adhering together.
1002 particulate matter, n—see powder.
1003 P/M, n—the acronym representing powder metallurgy. Used as P/M Part, P/M Product, P/M Process, and so forth.
1004 powder metallurgy, n—the production and utilization of metal powders.
1005 powder, n—particles that are usually less than 1000 µm (1 mm) in size.
1006 metal powder, n—particles of elemental metals or alloys, normally less than 1000 µm (1 mm) in size.

3.1.3 Processes to Produce Powder:

1101 atomization, n—the dispersion of a molten metal into particles by a rapidly moving gas or liquid stream or by mechanical means.
1102 granulation, n—the production of coarse metal particles by pouring the molten metal through a screen into water (shooting) or by violent agitation of the molten metal while solidifying.

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2 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard’s Document Summary page on the ASTM website.
1103 classification, n—separation of a powder into fractions according to particle size.
1104 air classification, n—the separation of powder into particle size fractions by means of an air stream of controlled velocity.
1105 gas classification, n—the separation of powder into particle size fractions by means of a gas stream of controlled velocity.
1106 chemical deposition, n—the precipitation of one metal from a solution of its salts by the addition of another metal or reagent to the solution.
1107 chemically precipitated metal powder, n—powder produced by the reduction of a metal from a solution of its salts either by the addition of another metal higher in the electromotive series or by other reducing agent.
1108 reduced metal powder, n—metal powder produced, without melting, by the chemical reduction of metal oxides or other compounds.
1109 disintegration, n—the reduction of massive material to powder.
1110 milling, n—the mechanical treatment of metal powder, or metal powder mixtures, as in a ball mill, to alter the size or shape of the individual particles or to coat one component of the mixture with another.
1111 pulverization, n—the reduction in particle size of metal powder by mechanical means, a specific type of disintegration.

3.1.4 Types of Powder:
1201 atomized metal powder, n—metal powder produced by the dispersion of a molten metal by a rapidly moving gas, or liquid stream, or by mechanical dispersion.
1202 electrolytic powder, n—powder produced by electrolytic deposition or by the pulverization of an electrodeposit.
1203 dendritic powder, n—particles, usually of electrolytic origin, having the typical pine tree structure.
1204 carbonyl powder, n—a metal powder prepared by the thermal decomposition of a metal carbonyl.
1205 master-alloy powder, n—a powder with high alloy concentration, designed to be diluted when mixed with a base powder to produce the desired composition.
1206 pre-alloyed powder, n—powder composed of two or more elements that are alloyed in the powder manufacturing process in which the particles are of the same nominal composition throughout. Synonymous with completely alloyed powder.
1207 completely alloyed powder, n—see pre-alloyed powder.
1208 partially alloyed powder, n—a powder in which the alloy addition or additions are metallurgically bonded to an elemental or pre-alloyed powder.
1209 diffusion-alloyed powder, n—a partially alloyed powder produced by means of a diffusion anneal.
1210 mechanically alloyed powder, n—a composite powder produced by mechanically incorporating other constituents which are generally insoluble within the deformable particles of the matrix metal.
1211 matrix metal, n—the continuous phase of a polyphase alloy or mechanical mixture; the physically continuous metallic constituent in which separate particles of another constituent are embedded.
1212 composite powder, n—a powder in which each particle consists of two or more distinct constituents.
1213 spongy, n—a porous condition in metal powder particles usually observed in reduced oxides.
1214 sponge iron, n—a coherent, porous mass of substantially pure iron produced by solid-state reduction of iron oxide (for example, iron ore or mill scale).
1215 sponge iron powder, n—ground and sized sponge iron, which may have been purified or annealed or both.
1216 mixed powder, n—a powder made by mixing two or more powders as uniformly as possible. The constituent powders will differ in chemical composition or in particle size or shape, or a combination thereof.
1217 premix, n—a uniform mixture of ingredients to a prescribed analysis, prepared by the powder producer, for direct use in compacting powder metallurgy products.
1218 comminuted powder, n—a powder produced by mechanical attrition of solid metal or powder.
1219 nanopowder, n—a powder consisting of particles typically less than 100 nanometers in size.

3.1.5 Shapes of Powder Particles:
1301 acicular powder, n—needle-shaped particles.
1302 needles, n—elongated rod-like particles.
1303 granular powder, n—particles having approximately equidimensional nonspherical shapes.
1304 nodular powder, n—irregular particles having knotted, rounded, or similar shapes.
1305 irregular powder, n—particles lacking symmetry.
1306 spherical powder, n—globular-shaped particles.
1307 flake powder, n—flat or scale-like particles whose thickness is small compared with the other dimensions.
1308 plates, n—flat particles of metal powder having considerable thickness.

3.1.6 Additives to Powder:
1401 binder, n—a cementing medium; either a material added to the powder to increase the green strength of the compact, and which is expelled during sintering; or a material (usually of relatively lower melting point) added to a powder mixture for the
specific purpose of cementing together powder particles which alone would not sinter into a strong body.

1402 feedstock, n—in metal injection molding (MIM), a moldable mixture of metal powder and binder.

1403 powder lubricant, n—an agent mixed with or incorporated in a powder to facilitate the pressing and ejecting of the compact.

1404 dispersion-strengthened material, n—a material consisting of a metal and finely dispersed, substantially insoluble, metallic or nonmetallic phase.

1405 pore-forming material, n—a substance included in a powder mixture that volatilizes during sintering and thereby produces a desired kind and degree of porosity in the finished compact.

3.1.7 Treatment of Powder:

1501 blending, n—the thorough intermingling of powders of the same nominal composition (not to be confused with mixing).

1502 equalizing, n—see blending.

1503 mixing, n—the thorough intermingling of powders of two or more materials.

1504 cross-product contamination, n—the unintentional mixing of powders with distinct differences in either physical characteristics or chemical composition or both.

1505 lubricating, n—mixing with, or incorporating in, a powder, some agent to facilitate pressing and ejecting the compact from the die body; applying a lubricant to the die walls and punch surfaces.

3.1.8 Properties of Powder:

1601 apparent density, n—the mass of a unit volume of powder, usually expressed as grams per cubic centimetre, determined by a specified method.

1602 bulk density, n—the mass per unit volume of a powder under nonstandard conditions, for example, in a shipping container (not to be confused with apparent density).

1603 tap density, n—the apparent density of the powder in a container that has been tapped under specified conditions.

1604 flow rate, n—the time required for a powder sample of standard weight to flow through an orifice in a standard instrument according to a specified procedure.

1605 specific surface, n—the surface area of one gram of powder, usually expressed in square centimetres.

1606 compactibility, n—a conceptual term, encompassing the powder characteristics of compressibility, green strength, edge retention, and lamination tendency, that relates to the ability of a powder to be consolidated into a usable green compact.

1607 compressibility, n—the capacity of a metal powder to be densified under a uniaxially applied pressure in a closed die.

DISCUSSION—Compressibility is measured in accordance with Test Method B 331 and may be expressed numerically as the pressure to reach a specified density, or alternatively the density at a given pressure.³

1608 compression ratio, n—the ratio of the volume of the loose powder to the volume of the compact made from it. Synonymous with fill ratio.

1609 fill ratio, n—see compression ratio.

1610 oversize powder, n—particles coarser than the maximum permitted by a given particle size specification.

1611 plus sieve, n—the portion of a powder sample retained on a standard sieve of specified number. (See minus sieve.)

1612 minus sieve, n—the portion of a powder sample which passes through a standard sieve of specified number. (See plus sieve.)

1613 fines, n—the portion of a powder composed of particles which are smaller than a specified size, currently less than 44 µm. See also superfines.

1614 superfines, n—the portion of a powder composed of particles that are smaller than a specified size, currently less than 10 µm.

1615 fraction, n—the portion of a powder sample that lies between two stated particle sizes. Synonymous with cut.

1616 cut, n—see fraction.

1617 subsieve fraction, n—particles all of which will pass through a 44-µm (No. 325) standard sieve.

1618 sieve fraction, n—that portion of a powder sample that passes through a standard sieve of specified number and is retained by some finer sieve of specified number.

1619 particle size, n—the controlling lineal dimension of an individual particle as determined by analysis with sieves or other suitable means.

1620 particle size distribution, n—the percentage by weight, or by number, of each fraction into which a powder sample has been classified with respect to sieve number or microns. (Preferred usage: “particle size distribution by frequency.”)

1621 hydrogen loss, n—the loss in weight of metal powder or of a compact caused by heating a representative sample for a specified time and temperature in a purified hydrogen atmosphere—broadly, a measure of the oxygen content of the sample when applied to materials containing only such oxides as are reducible with hydrogen and no hydride-forming element.

³ See Test Method B 331.
1622 segregation, n—the separation of one or more constituents of a powder, for example, by particle size or chemical composition.

3.1.9 Procedures to Evaluate Powder:
1701 sieve analysis, n—particle size distribution; usually expressed as the weight percentage retained upon each of a series of standard sieves of decreasing size and the percentage passed by the sieve of finest size. Synonymous with screen analysis.
1702 screen analysis, n—see sieve analysis.
1703 sieve classification, n—the separation of powder into particle size ranges by the use of a series of graded sieves.

3.1.10 Equipment to Evaluate Powder:
1801 powder flow meter, n—an instrument for measuring the rate of flow of a powder according to a specified procedure.
1802 mesh, n—the number of openings per linear inch of screen.

3.2 Forming—Terms associated with consolidation of metal powders and mixes, including tooling, equipment, and characterization of sintered compacts.

3.2.1 General:
2001 green—unsintered (not sintered); for example, green compact, green density, green strength.
2002 preforming—the initial pressing of a metal powder to form a compact that is subjected to a subsequent pressing operation other than coining or sizing. Also, the preliminary shaping of a refractory metal compact after presintering and before the final sintering.
2003 blank, n—a pressed, presintered, or fully sintered compact, usually in the unfinished condition, requiring cutting, machining, or some other operation to give it its final shape.
2004 briquet, n—see compact.
2005 compact, n—an object produced by the compression of metal powder, generally while confined in a die, with or without the inclusion of nonmetallic constituents. Synonymous with briquet.
2006 pressed bar, n—a compact in the form of a bar; a green compact.
2007 rolled compact, n—a compact made by passing metal powder continuously through a rolling mill so as to form relatively long sheets of pressed material.
2008 composite compact, n—a metal powder compact consisting of two or more adhering layers, rings, or other shapes of different metals or alloys with each material retaining its original identity.
2009 compound compact, n—a metal powder compact consisting of mixed metals, the particles of which are joined by pressing or sintering or both, with each metal particle retaining substantially its original composition.

3.2.2 Processes for Compacting:
2101 molding, v—the pressing of powder to form a compact.
2102 press, v—to apply force to a mass of powder, generally while confined in a die or container, to form a compact.
2103 double press-double sinter, n—to repress and sinter a previously presintered or sintered compact.

Discussion—Used to describe a four-step manufacturing process.

2104 single-action pressing, n—a method by which a powder is pressed in a stationary die between one moving and one fixed punch.

Discussion—Only during ejection does either the stationary die or punch move.

2105 double-action pressing, n—a method by which a powder is pressed in a die between opposing moving punches.
2106 withdrawal pressing, n—a powder consolidation method in which the die moves downward in relation to the lower punch(es) during compaction. It further descends over the fixed lower punch(es) for ejection, so that the compact may then be pushed off the tooling at this point.
2107 multiple pressing, n—a method of pressing whereby two or more compacts are produced simultaneously in separate die cavities.
2108 roll compacting, n—the progressive compacting of metal powders by the use of a rolling mill. Synonymous with powder rolling.
2109 powder rolling, n—see roll compacting.
2110 cold pressing, n—the forming of a compact at room temperature.

3.2.3 Conditions for Compacting:
2201 die lubricant, n—a lubricant applied to the walls of the die and to the punches to facilitate the pressing and ejection of the compact.

Discussion—Contrast with powder lubricant. Synonymous with die-wall lubricant.
2202 Die-wall lubricant, n—synonymous with die lubricant.

3.2.4 Tools Used for Compacting:
2301 Mold, n—in metal or powder injection molding, the member of the tooling into which the powder and binder mixture is forced, and the configuration of which forms the surfaces of the green part. In isostatic compacting, a mold is also the confining form in which powder is isostatically compacted.
2302 Compacting tool set, n—an assembly of tooling items in which powder is pressed.

Discussion—May include a die, punches, and core rods.

2303 Die, n—a member of the compacting tool set forming the cavity in which the powder is compacted or a P/M compact is repressed.
2304 Die body, n—the stationary or fixed part of a die.
2305 Die set, n—the parts of a press that hold and locate the die in proper relation to the punches.
2306 Core rod, n—a member of the compacting tool set that forms internal features such as splines, diameters, keyways, or other profiles in a P/M compact.
2307 Punch, n—a member of a compacting tool set used to close the die cavity and transmit the applied pressure to the powder or P/M compact.

Discussion—Multiple upper or lower punches may be needed to compact multilevel parts.

2308 Stripper punch, n—a punch that, in addition to forming the top or bottom of the die cavity, later moves further into the die to eject the compact.
2309 Split die, n—a die made of parts that can be separated for ready removal of the compact.
2310 Die insert, n—a removable liner or part of a die body.
2311 Segment die, n—a die fabricated by the assembly of several die sections within a retaining bolster or shrinkage ring.
2312 Rotary press, n—a machine fitted with a rotating table carrying multiple dies in which a material is pressed.

3.2.5 Phenomena Resulting from Compaction:
2401 Bridging, v—the formation of arched cavities in a powder mass.
2402 Green, adj—unsintered (not sintered); for example, green compact, green density, green strength.
2403 Springback, n—see green expansion.
2404 Cold welding, n—cohesion between two surfaces of metal, generally under the influence of externally applied pressure, at room temperature.

Discussion—Often used to describe the mechanism by which powder particles develop initial bonds and a pressed compact develops green strength.

3.2.6 Properties of Compacts:
2501 Green density, n—the mass per unit volume of an unsintered compact.
2502 Pressed density, n—synonymous with green density.
2503 Green expansion, n—the increase in dimensions of an ejected compact relative to the die dimensions, measured at right angles to the direction of pressing. Synonymous with springback.

3.2.7 Forging:
2601 Powder forging (P/F), n—densification by forging of an unsintered, presintered, or sintered preform made from powder.

Discussion—in the case in which the preform has been sintered, the process is often referred to as “sinter forging.”

2602 P/F, v—the acronym for powder forging. See powder forging.
2603 P/M forging, v—see powder forging.
2604 P/M hot forming, v—see powder forging.
2605 Preform, n—a blank intended to be subject to deformation and densification involving a change of shape.
2606 Sinter forging, n—powder forging using sintered preforms.
2608 Hot forging, v—see powder forging.
2610 Hot upset powder forging, n—hot densification of a P/M preform by forging where there is a significant amount of lateral material flow.
2611 Hot repressed powder forging, n—hot densification of a P/M preform by forging where material flow is mainly in the direction of pressing.

3.2.8 Metal Injection Molding:
2701 Metal injection molding (MIM), n—a process in which a mixture of metal powders and a binder system is forced under pressure into a mold. See also powder injection molding.
2702 MIM—see metal injection molding.
2703 Powder injection molding (PIM), n—a process in which a mixture of powders and a binder system is forced under
pressure into a mold. See also **metal injection molding**. 
**2704 PIM**—see **powder injection molding**.

### 3.3 Sintering—Terms associated with forming a metallic bond among particles including processes, equipment, and characterization of sintered compacts.

#### 3.3.1 Processes for Sintering:

**3101 sinter, v**—to increase the bonding in a mass of powder or a compact by heating below the melting point of the main constituent.

**3102 solid-state sintering, v**—sintering of a powder or compact without formation of a liquid phase.

**3103 presintering, v**—the heating of a compact at a temperature below the normal final sintering temperature, usually to increase the ease of handling or shaping the compact, or to remove a lubricant or binder before sintering.

**3104 activated sintering, v**—a sintering process during which the rate of sintering is increased, for example, by addition of a substance to the powder or by changing sintering conditions.

**3105 continuous sintering, v**—presintering, or sintering, in such manner that the objects are advanced through the furnace at a fixed rate by manual or mechanical means. Synonymous with **stoking**.

**3106 stoking, v**—see **continuous sintering**.

**3107 liquid phase sintering, v**—sintering of a compact, or loose powder aggregate, under conditions in which a liquid phase is present during part of the sintering cycle.

**3108 infiltration, n**—a process of filling the pores of a sintered, or unsintered, compact with a metal or alloy of lower melting point.

**3109 nitrogen alloying, n**—the transfer of nitrogen from a furnace atmosphere to powder or a P/M part, in such a way as to increase the nitrogen content of the material within controlled limits.

**3110 cored bar, n**—a compact of bar shape heated by its own electrical resistance to a temperature high enough to melt its interior.

#### 3.3.2 Conditions During Sintering:

**3201 packing material, n**—any material in which compacts are embedded during the presintering or sintering operation.

**3202 sintering time, n**—the total elapsed time during which the P/M part/specimen is within (∓) a specified percentage of the stated sintering temperature.

**3203 dissociated ammonia, n**—a reducing gas produced by the thermal decomposition of anhydrous ammonia over a catalyst, resulting in a gas of 75 % hydrogen and 25 % nitrogen. Synonymous with cracked ammonia.

**3204 exothermic atmosphere (gas), n**—a reducing gas atmosphere used in sintering, produced by partial or complete combustion of hydrocarbon fuel gas and air with the associated generation of heat. The maximum combustible content is approximately 25 atomic percent.

**3205 endothermic gas, n**—a reducing gas atmosphere used in sintering, produced by the reaction of a hydrocarbon vapor and air over a catalyst with the use of an external heat source. It is low in carbon dioxide and water vapor while containing combustibles of about 60 atomic percent hydrogen and carbon monoxide combined.

#### 3.3.3 Phenomena Resulting from Sintering:

**3301 powder metallurgy part, n**—a shaped object that has been formed from metal powders and bonded by heating below the melting point of the major constituent. A structural or mechanical component, bearing, or bushing made by the powder metallurgy process. Synonymous with **P/M part**.

**3302 P/M part, n**—see **powder metallurgy part**.

**3303 fully dense material, n**—a material completely free of porosity and voids.

**Discussion**—This is a conceptual term. In practice, complete densification is difficult to achieve and some microporosity will generally be present. The measured density of a material depends on its specific chemistry, thermomechanical condition, and microstructure.

**3304 cake, n**—a bonded mass of unpressed metal powder.

**Discussion**—Often refers to the form of powder as it exits a furnace.

**3305 closed pore, n**—a pore not communicating or connected with an exterior surface.

**3306 open pore, n**—a pore communicating with an exterior surface.

**3307 communicating pores, n**—see **interconnected porosity**.

**3308 interconnected porosity, n**—a network of pores in and extending to the surface of a sintered compact. Usually applied to powder metallurgy materials in which the interconnected porosity is determined by impregnating the specimens with oil. Synonymous with **communicating pores**.

**3309 exudation, n**—the action by which all or a portion of the low melting constituent of a compact is forced to the surface during sintering. Sometimes referred to as “bleed out.” Synonymous with **sweating**.
sweating, n — see exudation.

infiltration erosion, n—the pitting, channeling, and coarsening of the surface porosity that results from the dissolution of the base metal by the liquid infiltrant, as the infiltrant flows into the matrix.

infiltration residue, n—material that remains on the surface of the part after infiltration.

blistered compact, n—a sintered object characterized by having blisters or eruptions on the surface.

Discussion—In ferrous materials, this effect is often caused by in situ gas decomposition and soot formation that forces particles apart and causes the compact to blister.

neck formation, n—during sintering, the development of a neck-like bond between particles.

slumping, n—the lack of shape retention of a molded part, during subsequent processing, because of the effect of gravity.

warping, n—distortion that may occur in a compact during sintering.

oxide network, n—continuous or discontinuous oxides that follow prior particle boundaries.

surface finger oxide, n—the oxide that follows prior particle boundaries into a part from the surface and cannot be removed by physical means, such as rotary tumbling.

pore, n—an inherent or induced cavity within a particle or within an object.

permeability, fluid, n—the rate of passage of a liquid or a gas through a porous body; determined under specified conditions.

fluid permeability, n—see permeability, fluid.

3.3.4 Properties of Sintered Parts:

dimensional change of a compact, n—the difference, at room temperature, between the size of the sintered specimen and the die size.

Discussion—The difference in dimensions is usually reported as a percentage of the die size. It should include a (+) when the sintered part is larger than the die size and a (−) when the sintered part is smaller than the die size.

growth, n—an increase in dimensions of a compact which may occur during sintering. (Converse of shrinkage.)

shrinkage, n—a decrease in dimensions of a compact which may occur during sintering. (Converse of growth.)

infiltrant efficiency, n—the ratio of the mass of infiltrant absorbed by the part to the mass of infiltrant originally used, expressed as a percentage.

infiltration loading density, n—infiltrant weight per unit area of contact between infiltrant and part.

metal filter, n—a metal structure having controlled interconnected porosity produced to meet filtration or permeability requirements.

porosity, n—the amount of pores (voids) expressed as a percentage of the total volume of the powder metallurgy part.

apparent porosity, n—specific to cemented carbides, microstructural features that appear to be pores in a properly prepared, unetched surface; these features may result from uncombined carbon or nonmetallic inclusions as well as actual porosity.

density (dry), n—the mass per unit volume of an unimpregnated powder metallurgy part.

density (wet), n—the mass per unit volume of a powder metallurgy part impregnated with oil or other nonmetallic materials.

density ratio, n—the ratio, often expressed as a percentage, of the density of a porous material to the density of the same material completely free of porosity. Synonymous with relative density.

relative density, n—see density ratio.

radial crushing strength, n—the relative capacity of a plain sleeve specimen of sintered metal to resist fracture induced by a load applied between flat parallel plates in a direction perpendicular to the axis of the specimen.

transverse rupture strength, n—the stress calculated from the flexure formula, required to break a specimen supported near the ends as a simple beam; the load is applied midway between the center lines of the supports.

permeability, n—a property measured as the rate of passage of a liquid or gas through a compact; measured under specified condition.

oil content, n—the measured amount of oil contained in an oil-impregnated object, for example, a self-lubricating bearing.

apparent hardness, n—the hardness of a P/M material (including the effects of porosity), measured using macroindentation hardness equipment.

Discussion—See general description of production, properties, and uses of sintered metal powder bearings and structural parts, paragraph on density and mechanical properties, information on hardness measurement, Volume 02.05, gray pages.

3.3.5 Procedure to Evaluate Sintered Parts:

rate-of-oil flow, n—the rate at which a specified oil will pass through a sintered porous compact under specified test
3.3.6 Removal of Binders:

3601 binder removal, n — the chemical or thermal extraction of binder from a compact. Synonymous with debinding.
3602 debinding, n — see binder removal.
3603 burn-off, n — removal of organic additives (binder or lubricant) from a compact by heating.

3.4 Postsinter Treatments—Terms associated with secondary operations including repressing, machining, and heat treatment that are performed on sintered compacts.

3.4.1 Processes:
4101 impregnation, n — a process of filling the pores of a sintered compact, with a nonmetallic material such as oil, wax, or resin.
4102 repress, v — to apply pressure to a previously pressed and either sintered or presintered compact. It includes restrike, coin, and size.
4103 restrike, v — to repress for the purpose of increasing the density of a sintered compact (not to be confused with coin or size).
4104 coin, v — to repress a sintered compact to obtain a definite surface configuration. Synonymous with emboss (not to be confused with restrike or size).
4105 size, v — to repress a sintered compact to decrease the dimensional variation (not to be confused with coin or restrike).
4106 steam blackening, n — the superheated steam treatment of a ferrous P/M component to form a thin, dark, oxide layer, primarily Fe$_3$O$_4$, on the outside surfaces of the component, and extending into the surfaces of the interconnecting porosity.
4107 steam treatment, n — see steam blackening.

3.5 Miscellaneous—Terms that do not belong under any other category.

3.5.1 Definitions:
5101 RSM—Rapidly Solidified Materials.
5102 RSP—Rapid Solidification Processing or Rapidly Solidified Powders.
5103 RST—Rapid Solidification Technology.
5104 lot, n — a specified quantity of product manufactured under traceable, controlled conditions as agreed between producer and user.

3.5.2 Processes:
5201 isostatic pressing, n — the pressing of a powder, compact, or sintered object by subjecting it to a nominally equal pressure from every direction.
5202 cold isostatic pressing, n — the pressing of a powder, compact, or sintered object by subjecting it, at ambient temperature, to nominally equal pressure from every direction.
5203 hot isostatic pressing, n — the pressing of a powder, compact or sintered object by subjecting it, at elevated temperature, to nominally equal pressure from every direction.
5204 hot densification, n — the consolidation, at elevated temperature and pressure, of an unsintered, presintered, or sintered powder preform, or encapsulated or loose powder, to reduce porosity.
5205 hot pressing, v — pressure-assisted, low strain rate uniaxial densification of a powder preform, compact, or encapsulated or loose powder at a temperature sufficient to induce diffusion or creep.

3.5.3 Materials:
5301 cemented carbide, n — sintered material characterized by high strength and wear resistance and comprising one or more carbides of refractory metals as the main component bonded by metallic binder phase.
5302 hardmetal, n — see cemented carbide.
5303 cermet, n — sintered material containing at least one metallic phase and at least one nonmetallic phase that is generally of a ceramic nature.
X1. ALPHABETICAL INDEX

X1.1 Index Numbers correspond to numbers assigned to terms within the standard.

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Standard Test Method for Measurement of Thickness of Anodic Coatings on Aluminum and of Other Nonconductive Coatings on Nonmagnetic Basis Metals with Eddy-Current Instruments

1 This method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on General Test Methods.

2 Annual Book of ASTM Standards, Vol 02.05.

1 This method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on General Test Methods.
5.5 Curvature—Measurements are affected by the curvature of the test specimen. The influence of curvature varies considerably with the make and type of instrument, but always becomes more pronounced as the radius of curvature decreases.

5.6 Surface Roughness:

5.6.1 Measurements are influenced by the surface topography of the substrate and the coating, and a rough surface will give individual instrument readings that will vary from point to point. In this case, it is necessary to make many readings at different positions to obtain an average value that is representative of the mean coating thickness.

5.6.2 If the basis metal is rough it may also be necessary to check the zero of the instrument at several positions on a sample of the uncoated rough substrate. If the roughness of the substrate surface is small, relative to the coating thickness, its effect will probably be negligible.

5.7 Foreign Particles—The probes of eddy-current instruments must make physical contact with the test surface and are, therefore, sensitive to foreign material that prevents intimate contact between the probe and the coating surface.

5.8 Pressure—The pressure with which the probe is applied to the test specimen affects the instrument readings, and should, therefore, be kept constant.

5.9 Number of Readings—The precision of the measurements can be improved by increasing the number of readings in accordance with statistical principles.

6. Calibration of Instruments

6.1 Before use, each instrument shall be calibrated in accordance with the instructions of the manufacturer, employing suitable thickness standards. During use, the calibration shall be checked at frequent intervals, at least once an hour. Attention shall be given to the factors listed in Section 5 and to the procedures described in Section 7.

6.2 Calibration standards of known thickness are available either as foils or as coated specimens.

6.2.1 Calibration Foils:

6.2.1.1 The calibration foils used for the calibration of eddy-current instruments are generally made of plastic. They are advantageous for calibration on curved surfaces, and are more readily available than coated standards.

6.2.1.2 To prevent measurement errors, it is necessary to ensure that intimate contact is established between foil and substrate. Resilient foils should be avoided if possible. Calibration foils are subject to indentation and should, therefore, be replaced frequently.

6.2.2 Coated Standards—These calibration standards consist of nonconductive coatings of known, uniform thickness permanently bonded to the substrate material.

6.3 The basis metal of the calibration standards shall have electrical properties similar to those of the basis metal of the coated test specimen. To confirm their suitability, a comparison of the readings obtained with the basis metal of the bare standard and that of the test specimen is recommended.

6.4 The basis metal thickness for the test and the calibration shall be the same if the critical thickness, defined in 4.3, is not exceeded. When possible, back up the basis metal of the standard or of the test specimen with a sufficient thickness of similar material to make the readings independent of the basis metal thickness. A way to determine if the basis metal thickness exceeds the critical thickness is to make measurements before and after backing up the basis metal with copper or aluminum at least 3 mm thick. If there is no difference between the readings, the critical thickness is exceeded.

6.5 If the test specimen is soft and thin, it is subject to indentation by the probe. Because of this, and despite the use of special probes or fixtures, measurements on such specimens are sometimes impossible to make.

6.6 If the curvature of the test specimen is to be measured is such as to preclude calibration on a flat surface, the curvature of the coated standard or of the substrate on which the calibration foil is placed shall be the same as that of the test specimen.

7. Procedure

7.1 Operate each instrument in accordance with the instructions of the manufacturer. Give appropriate attention to the factors listed in Section 5.

7.2 Check the calibration of the instrument at the test site each time the instrument is put into service and at frequent intervals during use to assure proper performance.

7.3 Observe the following precautions:

7.3.1 Basis Metal Thickness—Check whether the basis metal thickness exceeds the critical thickness. If not, either use the back-up method in 6.4, or make sure that the calibration has been made on a standard having the same thickness and electrical properties as the test specimen.

7.3.2 Edge Effects—Do not make readings close to an edge, hole, inside corner, etc., of a specimen, unless the validity of the calibration for such a measurement has been demonstrated.

7.3.3 Curvature—Do not make readings on a curved surface of a specimen unless the validity of the calibration for such a measurement has been demonstrated.

7.3.4 Number of Readings—Because of normal instrument variability, it is necessary to make several readings at each position. Local variations in coating thickness may also require that a number of measurements be made in any given area; this applies particularly to a rough surface (see 5.9).

7.3.5 Surface Cleanliness—Before making measurements, clean any foreign matter such as dirt, grease, and corrosion products from the surface without removing any coating material.

8. Accuracy

8.1 The instrument, its calibration, and its operation shall be such that the coating thickness can be determined within 10 % or 1 µm, whichever is greater, of the true thickness.

9. Report

9.1 The report shall include the following information:

9.1.1 Type of instrument used,
9.1.2 Size and description of test specimen,
9.1.3 Whether special jigs were used,
9.1.4 Type of calibration standard and the method used, and
9.1.5 Thickness of coating as determined from the measurements.

Note 1—Although, theoretically, this test method can be used for
measuring nonconductive coatings on a magnetic basis metal, its use for coatings below 25 µm is not recommended, and the magnetic method outlined in Method B 499 shall be used.

10. Precision and Bias

10.1 The precision of this test method is being determined.
Standard Guide for
Preparation of Zinc Alloy Die Castings for Electroplating
and Conversion Coatings

This standard is issued under the fixed designation B 252; the number immediately following the designation indicates the year of
original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A
superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide is intended as an aid in establishing and maintaining a procedure for preparing zinc alloy die castings for electroplating and conversion coatings. It is primarily intended for the preparation of Alloys UNS Z33521 (AG-40A) and UNS Z35530 (AC-41A) (Specification B 86) for electroplating with copper, nickel, and chromium (Specification B 456).

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards: 2
   B 6 Specification for Zinc
   B 86 Specification for Zinc and Zinc-Aluminum (ZA) Alloy
   Foundry and Die Castings
   B 456 Specification for Electrodeposited Coatings of Copper Plus Nickel Plus Chromium and Nickel Plus Chromium

2.2 Military Standard:
   MIL-S-13165C Shot Peening of Metal Parts 3

3. Summary of Practice

3.1 The normal sequence of preparation steps is as follows: (1) smoothing of parting lines; (2) smoothing of rough or defective surfaces, if necessary; (3) buffing, if necessary; (4) precleaning and rinsing; (5) alkaline electrocleaning and rinsing; (6) acid dipping and rinsing; and (7) copper striking.

4. Significance and Use

4.1 The performance and quality of electroplated or conversion-coated zinc alloy die casting depends upon the surface cleanliness and condition. Various metals are electroplated or conversion coatings are established on zinc alloys for decorative or engineering finish. The common electroplates applied are usually copper, nickel, and chromium for decorative and functional uses. The common conversion coatings applied are phosphates, chromates, and anodized coatings. Electroplated zinc die castings and conversion coatings on zinc die castings are used in many industries such as the marine, automotive, plumbing fixtures, and appliance industries.

5. Composition and Characteristics of Zinc Alloy Die Castings

5.1 The alloys used in the manufacture of zinc alloy die castings are made with special high-grade zinc conforming to Specification B 6, alloyed with about 4 % of aluminum, 0.04 % of magnesium, and either 0.25 (max) or 1.0 % copper (Alloys UNS Z33521 and UNS Z35530). Impurities such as lead, cadmium, tin, and iron are held at or below the specified low levels in Specification B 86.

5.2 Die castings made of Alloys UNS 233521 and UNS 235530 are usually dense and fine grained but do not always have smooth surfaces. Defects sometimes encountered in the surface layers include cracks, crevices (cold shut), skin blisters, and hemispherical pores. Burrs are usually left at parting lines where fins and gates are removed by die trimming.

5.3 Cast surfaces are frequently contaminated with parting compounds applied at frequent intervals to die surfaces to facilitate the ejection of the castings and with water-soluble oils added to quenching tanks for corrosion inhibition.

5.4 Zinc alloy die castings are chemically active and are dissolved or etched during prolonged contact with concentrated solutions of many mineral or organic acids or strongly alkaline solutions with a pH greater than 10. Immersion periods in such solutions should be of short duration to avoid roughening.

1 This guide is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.02 on Pre Treatment.


2 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard’s Document Summary page on the ASTM website.

3 Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.
6. Smoothing of Parting Lines

6.1 Parting lines are smoothed by (1) mechanical polishing with abrasive-coated wheels or belts, (2) tumbling with abrasive media, or (3) vibration with abrasives.

6.2 Abrasives with a size range of 220 to 300 mesh glued on cloth wheels or continuous cloth belts that run over flexible back-up wheels are usually used for mechanical polishing of parting lines. Wheel diameters range from 5 to 40 cm, depending on the complexity of the shape. Wheels are rotated with a minimum peripheral speed of 2500 m/min. A peripheral speed of 2100 m/min should not be exceeded with belts. Lower speeds of the order of 1100 to 1400 m/min are fairly common for small die castings polished on small diameter wheels. Abrasive belts should not be used dry but should be lubricated with a small amount of grease. Die castings usually are handled individually to polish parting lines smooth. This may require 30 s or less for small castings, and sometimes 5 or 6 min for larger ones.

6.3 Tumbling in horizontal barrels, loaded with abrasive stones such as limestone, preformed and fused aluminum oxide, ceramic shapes or abrasive-loaded plastic chips, and a lubricant such as soap or detergent solution, removes parting-line burrs from die castings in 4 to 12 h. The barrels may be rotated at 4 r/min. Higher speeds reduce the time cycles and costs, but also increase the danger of impingement of parts against zinc surfaces. A hexagonal barrel with a capacity of 0.5 m³ can be loaded with 450 kg of abrasive stones or chips and 90 kg of zinc die castings.

6.4 Vibration in a bed of resin-bonded abrasive chips removes parting-line burrs, typically in 1 to 4 h. Frequencies range from 700 to 2100 cpm and amplitudes from 0.8 to 6.4 mm. A vibrating tub with a capacity of 0.5 m³ can be loaded with about 900 kg of abrasive media and 180 kg of zinc die castings. A dilute solution of detergent or soap is continuously metered through the bed of media and parts to keep their surfaces clean and maximize surface smoothing. Parting lines may be mechanically polished before vibratory processing when a large amount of flash must be removed.

7. Smoothing of Rough or Defective Surfaces

7.1 Rough or defective surfaces are smoothed by (1) mechanical polishing on rotating wheels or continuous, abrasive-coated belts, (2) spin finishing, (3) vibratory finishing, or (4) controlled shot peening. Fissures, skin blisters, and other defects with a depth of 25 to 50 µm can usually be erased with these metal-removal methods. Deeper defects are infrequent.

7.2 Mechanical polishing for smoothing rough or defective surfaces is similar to mechanical polishing for smoothing parting line areas (see 6.2). Parting lines and rough or defective surfaces are frequently polished by the same operator. If polishing is mechanized to advance die castings attached to a conveyor through successive belts or wheels to polish different areas, a manual operation may be required later to complete the smoothing of parting lines if they are too curved. The finish ranges from 0.2 to 0.6 µm, depending on the abrasive and the pressure.

7.3 Smoothing by spinning in abrasives is accomplished by attaching die castings to spindles or drums rotated with a peripheral speed of about 600 m/min in a slurry of abrasive material such as ground corn cobs or nut shells mixed with a small amount of grease or other lubricant. Times usually range from 5 to 10 min and the finish from 0.1 to 0.2 µm, depending on the abrasive.

7.4 Vibrating tubs loaded with plastic chips (such as polyurethane) impregnated with an abrasive (such as aluminum oxide) smooth the surfaces of die castings in 2 to 4 h when frequencies are in the range of 1700 to 2100 cpm and amplitudes are adjusted to 3.2 to 6.4 mm. Vibratory machines produce a finish of 0.15 to 0.25 µm, with a cutting rate of 5 µm/h. A smoother finish of 0.075 to 0.125 µm can be obtained with plastic media containing finer abrasive, which removes metal at a slower rate. Media and zinc parts are usually loaded with a ratio of 5:1 or 6:1. Surface gouges may occur with a smaller ratio.

7.5 Controlled shot peening will plastically deform and densify the casting surface and near-surface layers. Shot peening can seal surface pores, which can create problems in electroplating and conversion coating. The process is described in MIL-S-13165C. The process is also effective in removing fins, burrs, and flash from the surface. The casting configuration, including the smallest size radii and wall thickness, as well as the required finish and contamination limits, will dictate the proper selection of peening media, shot size, intensity, and coverage, as is detailed in MIL-S-13165C.

8. Buffing

8.1 Die castings are buffed to produce a mirror-like finish, suitable for plating with conventional solutions, when good leveling plating solutions are not available. Buffing can be omitted, however, for die castings which have good surfaces or which can be uniformly polished to a finish of 0.25 µm, if solutions with good leveling power are used for plating copper and nickel.

8.2 Die castings are buffed on cloth wheels rotated at a peripheral speed not exceeding 2150 m/min. Slower speeds, of the order of 1100 to 1600 m/min, are used for small die castings. Buffing compounds should be made with a binder that is readily emulsified or saponified during alkaline cleaning. The abrasive may be tripoli (amorphous silica) or lime, mixed with about 25% of tallow or other lubricants. Compounds suspended in a liquid are preferred for automatic buffing machines that advance die castings through a succession of buffs of varying diameter and width, which individually smooth different surface areas. Buffs are usually made of cloth with a thread count of 34 to 37/cm. A finish of 0.025 to 0.05 µm can be produced by buffing. The smoothing rate is influenced by the temperature of the metal surface (faster at approximately 150°C than at lower temperatures).

8.3 After buffing, surfaces with impacted buffing compound can be improved by passing them over a dry wheel to remove buffing compound. This will reduce the demand placed on the precleaning solution.

9. Precleaning and Rinsing

9.1 It is strongly recommended that the preliminary removal of most of the buffing compound and other soil in a precleaning operation be done as soon as possible after buffing and
polishing. Most buffing compounds become substantially more difficult to remove after aging several days.

9.2 There are several methods by which soils can be removed from zinc die castings prior to final alkaline electrocleaning. Generally speaking, these fall into three main classes: solvent degreasing, emulsion cleaning, and cleaning with aqueous base detergents.

9.2.1 Solvent Degreasing—Before considering the use of solvent degreasing, federal and state safety and environmental laws and regulations should be consulted. Many of the commonly used solvents are now being banned from use. Exposure to their vapors (VOC) is being strictly regulated for health, safety, and environmental reasons. Current safe exposure levels for various solvents should be obtained before use. Cold solvents, such as mineral spirits, methylene chloride, trichloroethylene, perchloroethylene and trichloroethane, are used with brushing to loosen packed buffing compound, but this method usually is not practical for mass production conditions. Simple dipping in cold solvent is often ineffective. Vapor degreasing with trichloroethylene or perchloroethylene is widely practiced. Often the buffered die castings are sprayed with, or immerged in, hot solvent for mechanical removal of heavy soil deposits. This is followed by condensation of hot, clean solvent vapors on the work; this removes the last traces of grease and compound. The method is very effective, provided adequate measures are taken to remove the very fine abrasive and metallic particles from the work. Trichloroethylene and perchloroethylene are nonflammable as used in vapor degreasing and still must be used in systems designed to protect personnel from inhalation of vapors. Suppliers of solvents should be consulted as to the safety of a given installation.

9.2.1.1 All federal, state, and local regulations for the disposal of solvents should be followed.

9.2.2 Emulsion Cleaning:

9.2.2.1 Impacted buffing compound may be loosened, and to some extent removed, by immersion in various hydrocarbon-water emulsions. These emulsions are available in several forms, including unstable emulsions (diphase cleaners), invert type emulsions, mixtures of emulsions and alkaline cleaners, and stable emulsions. Such emulsion cleaners usually have a suitable hydrocarbon base such as kerosene or a higher flashpoint solvent to which is added emulsifiers, soaps, and inhibitors to prevent etching of the die castings. The pH of the emulsion cleaner should be kept between 7 and 10 to avoid damage to the castings.

9.2.2.2 These emulsions normally are used hot, about 80°C, as a soak, sometimes with agitation, for about 2 to 5 min. A warm water spray rinse should follow the emulsion soak cleaning. Buffing compound not removed in the emulsion soak is sufficiently softened so that it is easily removed in an alkaline spray wash operation that normally follows.

9.2.2.3 Emulsion cleaning is an effective method for removing buffing compound. Its principal disadvantage is the danger of carryover of hydrocarbon solvent into plating baths because of incomplete rinsing. For this reason, it is very important that proper alkaline cleaning and rinsing follow to ensure solvent removal from blind holes, defects in rack coatings, and recesses.

9.2.2.4 All federal, state, and local regulations for the use and disposal of solvents should be followed.

9.2.3 Aqueous Base Detergents—In recent years, hot mixtures of emulsifiers and surfactants (wetting agents), sometimes combined with mild alkaline phosphates or borates, are used for soak cleaning to soften and remove buffing compound. Combining soak cleaning with ultrasonics is particularly effective on impacted buffing compound. Such detergent soaks should be followed by spray cleaning with an alkaline cleaner. If a spray cleaning step is not needed, then the soak cleaning step should be followed by a spray rinse with warm water before electrocleaning. Sometimes conventional alkaline soak cleaners are used for precleaning die castings with little or no buffing compound on them. These alkaline cleaners must be mild and inhibited since strong alkali will attack the castings.

9.3 Power Spray Alkaline Washing—Alkaline spray cleaners are widely used, during the initial cleaning operation or following initial presoaks in emulsions, solvents, or detergents. This is accomplished with conveyerized units equipped with washing, draining, rinsing, and draining sections. The solution heated to a temperature range of 50 to 80°C is sprayed with a pressure of 170 to 205 kPa through nozzles on 20 to 30 cm centers in the washing area. A typical solution may contain 10 g/L of mixed alkalies such as trisodium phosphate, sodium tripolyphosphate, sodium metasilicate, and sodium bicarbonate and not more than 1 g/L sodium hydroxide. The solution should also contain not more than 0.2 g/L of a low-foaming or non-foaming surfactant. In a typical precleaning cycle, a 1 to 2 min washing period is followed with a ½ to 1 min draining period, a ½ to 1 min water rinse with spray nozzles and a final draining period of ½ to 1 min. Proprietary alkaline spray cleaners are available for this application.

10. Alkaline Electrocleaning and Rinsing

10.1 Electrocleaning is necessary for completing the removal of oil, grease, and soil, and to ensure good electrolead adhesion. Anodic cleaning is usually selected for zinc alloys in preference to cathodic cleaning. Anodic current densities usually range from about 1.6 to 3.2 A/dm². Time cycles vary from 25 to 45 s.

10.2 A typical solution for anodic cleaning contains 30 to 40 g/L of mixed alkalies such as trisodium phosphate and sodium metasilicate, 0.5 g/L of a low-foaming surfactant, and not more than 0.5 g/L of sodium hydroxide and is heated to 70 to 82°C. Proprietary products for anodic cleaning are available. Lower temperatures may be required if time cycles must be prolonged for more than 45 s, or if the transfer time from the electrocleaner to the first rinse is more than about 30 s. Operating an anodic electrocleaner at too dilute concentrations may over etch the castings.

10.3 A cycle including a warm water rinse, a cold water rinse, and a water spray rinse is recommended after alkaline electrocleaning. The rinses should be agitated with air to dilute, as much as possible, the concentration of alkaline solution in blind holes, grooves, and other surface indentations and

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* ASTM Committee D26 Manual on Vapor Degreasing, MNL2, ASTM, Philadelphia, PA.
cavities. Alkaline solution entrapped in surface crevices and pores will become sites for process blisters if alkaline cleaner concentrations are not reduced to very low levels by agitated cold water rinsing. Rinse water containing a high concentration of dissolved minerals should be avoided.

10.4 Electrocleaning may not be required for the application of conversion coatings.

11. Acid Dipping and Rinsing

11.1 An acid dip must follow alkaline cleaning, to remove zinc oxidation products and trace amounts of alkaline compounds carried over from the cleaning operations due to the inadequate rinsing. The strength must be adjusted to the time of immersion. A solution containing 0.25 to 0.75 % by weight of sulfuric acid is frequently used for time cycles of 25 to 45 s, at room temperature. A solution of citric acid is a safe alternative. All acids used should be removed by thorough rinsing before electroplating or applying a conversion coating. Excessive exposure to stronger acid solutions can cause etching and dissolution of the metal.

11.2 The acid dip should remove all traces of black films or loosely adherent smut. For die castings prone to smut, including alloys containing more than about 0.25 % copper, the acid solution can be ultrasonically agitated; this effectively prevents the retention of smut on the surface.

11.3 A succession of two agitated overflow rinses and a water spray rinse is suggested after acid dipping. A final water rinse should use deionized spray water, which can be recycled for use in an initial rinsing stage. Acid solution must be completely removed from crevices and pores, to avoid blisters that may otherwise occur during or soon after plating. Crevices and pores in grooves and other surface indentations are common sites for process blisters if rinsing after acid dipping is incomplete.

11.4 Electrocleaning may not be required for the application of conversion coatings.

12. Copper Striking

12.1 A copper strike applied in a cyanide copper solution is normally the first plating step for all die castings to be electroplated with nickel and chromium. The thickness of the strike should be at least 1.0 µm for die castings that will be subsequently plated with bright copper in high-temperature cyanide solutions and at least 5.0 µm for die castings that will be plated with nickel after copper striking. A thickness of 3.0 to 4.0 µm is recommended for die castings that will be subsequently electroplated with bright, leveling copper in copper sulfate-sulfuric acid solutions.

12.2 Solutions containing 20 to 25 g/L of copper cyanide, 10 to 20 g/L of free sodium cyanide, and 15 to 75 g/L of sodium carbonate are customary for strike solutions. A few formulations include the molar equivalents of potassium cyanide in place of sodium cyanide. Others contain 3.8 to 7.5 g/L of sodium hydroxide in addition to the major constituents. In many of the formulations, use is made of addition agents that reduce hexavalent chromium, aid in anode corrosion, and refine the grain structure. Cathode current densities normally range from 2.7 to 6.5 A/dm² and solution temperatures from 50 to 57°C. Cyanide solutions containing a higher concentration of copper cyanide and operated at a higher temperature should be avoided because of the danger of blister formation.

12.3 The average cathode current density must be balanced with the free sodium cyanide and the temperature of the solution to prevent burning at edges and other high-current-density areas. With an average cathode current density of 2.7 A/dm², the cathode current efficiency varies from 30 to 60 % for strike plating. A high temperature, a high copper cyanide concentration, and a low-free cyanide concentration, within the limits given in 12.2, favor high efficiency. Ultrasonic agitation has been proposed for increasing cathode efficiency, the covering power in recessed areas, and improving the density of the copper deposit. Ultrasonic agitation has been reported as an important condition for copper striking in cyanide-free solutions prepared with potassium pyrophosphate, copper pyrophosphate and potassium citrate, prior to bright copper plating in acid sulfate solutions.5

12.4 High-purity copper anodes are recommended for copper cyanide strike solutions. Solutions should be continuously filtered to avoid the inclusion of small particles that nucleate nodules during subsequent plating operations.

Standard Guide for Preparation of Aluminum Alloys for Electroplating

1. Scope

1.1 This guide covers cleaning and conditioning treatments used before metal deposition (Section 5), and immersion deposit/strike procedures (Section 6) that enhance the adhesion of metals that are subsequently applied to aluminum products by electrodeposition or by autocatalytic chemical reduction.

1.2 The following immersion deposit/strike procedures are covered:

1.2.1 Zinc immersion with optional copper strike (6.3).
1.2.2 Zinc immersion with neutral nickel strike (6.4).
1.2.3 Zinc immersion with acetate-buffered, nickel glycolate strike (6.5).
1.2.4 Zinc immersion with acid or alkaline electroless nickel strike.
1.2.5 Tin immersion with bronze strike (6.6).

1.3 From the processing point of view, these procedures are expected to give deposits on aluminum alloys that are approximately equivalent with respect to adherence. Corrosion performance is affected by many factors, however, including the procedure used to prepare the aluminum alloy for electroplating.

1.4 This guide is intended to aid electroplaters in preparing aluminum and its alloys for electroplating.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific precautionary statements see Section 7 and Appendix.

2. Referenced Documents

2.1 ASTM Standards:
B 85 Specification for Aluminum-Alloy Die Castings
B 179 Specification for Aluminum Alloys in Ingot Form for Castings from all Casting Processes
B 209 Specification for Aluminum and Aluminum-Alloy Sheet and Plate
B 209M Specification for Aluminum and Aluminum-Alloy Sheet and Plate [Metric]
B 221 Specification for Aluminum-Alloy Extruded Bars, Rods, Wire, Shapes, and Tubes
B 221M Specification for Aluminum-Alloy Extruded Bars, Rods, Wire, Shapes, and Tubes
B 322 Practice for Cleaning Metals Prior to Electroplating
B 431 Practice for Processing of Mandrels for Electroforming
E 527 Practice for Numbering Metals and Alloys (UNS)

3. Significance and Use

3.1 Various metals are deposited on aluminum alloys to obtain a decorative or engineering finish. The electroplates applied are usually chromium, nickel, copper, brass, silver, tin, lead, cadmium, zinc, gold, and combinations of these. Silver, tin, or gold is applied to electrical equipment to decrease contact resistance or to improve surface conductivity; brass, copper, nickel, or tin for assembly by soft soldering; chromium to reduce friction and obtain increased resistance to wear; zinc for threaded parts where organic lubricants are not permissible; tin or lead is frequently employed to reduce friction on bearing surfaces. Nickel plus chromium or copper plus nickel plus chromium is used in decorative applications. Nickel plus brass plus lacquer or copper plus nickel plus brass plus lacquer is also used for decorative finishes, sometimes with the brass oxidized and relieved in various ways.

3.1.1 Electroless nickel may be applied as a barrier layer prior to other deposits, or for engineering purposes.

3.2 The preparation of aluminum and aluminum alloy mandrels for electroforming is described in Practice B 431.

4. Nature of Aluminum and Its Influence on Preparation

4.1 Microstructure—It is difficult to find a preplating procedure that is equally satisfactory for all types and tempers of aluminum alloys because the various alloys and products behave differently electrochemically due to their different compositions and metallurgical structures. When elements are added for alloying purposes, they may appear in an aluminum alloy in several different forms: that is, they may be in solid solution in the aluminum lattice, be present as microparticles of
the elements themselves, or be present as particles of intermetallic compounds formed by combination with the aluminum. The several solid solution matrices and the 20 or more microconstituents that may occur in commercial alloys may have different chemical reactivities and electropotentials and their surfaces may not respond uniformly to various chemical and electrochemical treatments. In addition, the response may be influenced by variations in the microstructure of different lots of products of the same alloy. In some cases, these variations may be introduced or aggravated by preparation processes; for example, the heat generated in buffing. The electroplater needs to know the aluminum alloy that is to be processed in order to select the best electroplating procedure. In the absence of this information, there are so-called universal procedures that may be used. However these will not necessarily be the best or the most economical procedures for the alloy.

4.2 Oxide Film—In addition to differences in microstructure that may affect response to preplating treatments, all aluminum products have an ever-present natural oxide film. This oxide film can be removed by various acid and alkaline treatments and even though it reforms immediately on contact with aqueous solutions or air, it then is usually thinner and more uniform than the original film. The newly formed oxide film provides a more suitable surface for deposition of the first metallic layer.

5. Cleaning and Conditioning Treatments

5.1 To obtain consistent results for electroplating on aluminum alloys, it is essential that the various cleaning and conditioning treatments provide a surface of uniform activity for the deposition of the initial metallic layer. First, the surface should be free of any oil, grease, buffing compound, or other foreign material. For removing oil, grease, or buffing compound, use vapor degreasing,5 solvent washing, or solvent emulsion cleaning. For removing buffing compound, specially formulated detergent type or modified detergent type buffing compound removers may also be used. If the deposits of soil are relatively light and fairly uniformly distributed, a mild etching type cleaner may also be used. A convenient one is a hot, aqueous carbonate-phosphate solution (X1.1). Other types of cleaners are used; for example, mildly alkaline or acidic soak cleaners are used to remove gross soils. Also available are a wide range of proprietary cleaners of the “non-etching” type. Some of these are actually buffered mixtures, similar to the carbonate-phosphate mixture (X1.1) where the so-called non-etching characteristics are obtained by buffering the solution to pH levels where the etching action becomes minimal. Others are truly non-etching types where etching is prevented by using silicate inhibitors, such as sodium metasilicate (Na$_2$SiO$_3$). These inhibitors always leave a film of aluminum silicate on the surface. When these materials are used, subsequent deoxidizing solutions should contain controlled amounts of fluoride salts to insure complete removal of the film.

NOTE 1—General information on the cleaning of metals is given in Practice B 322.

5.2 After cleaning, a conditioning treatment of the surface is generally required. For this to be effective, it must accomplish two things: (1) remove the original oxide film and (2) remove any microconstituents that may interfere with the formation of a continuous deposited metallic layer or that may react with subsequent electroplating solutions.

5.2.1 An effective conditioning treatment is immersion of the work in a warm sodium hydroxide solution (Appendix X1.3) followed by water rinsing and immersion in a nitric acid-bifluoride desmutting solution (Appendix X1.4). An alternative desmutting solution is sulfuric acid-hydrogen peroxide (Appendix X1.5).

NOTE 2—When an unmodified sodium hydroxide solution is used, etching may become nonuniform and heavy concrete-like scales may form on tank walls and heating surfaces, their development becoming more rapid as the concentration of dissolved aluminum increases. The incorporation of controlled amounts of deflocculating complexors such as sodium gluconate, sodium glucoheptonate, certain sugar derivatives, and certain substituted sugar amines will eliminate this problem. Many proprietary etching materials are so modified.

NOTE 3—The universal acid mixture (Appendix X1.9) is applicable to almost all alloys, and is especially desirable for use with alloys containing magnesium.

5.2.2 For heat-treated alloys (alloys in a “T” temper), it is important to remove the relatively thick, heat-treated oxide film before proceeding with subsequent conditioning treatments. Normally, heat-treated films are removed by machining, or by the polishing action on metal surfaces that are buffed.

5.2.2.1 In the absence of machining or buffing, controlled abrasive blasting may be used to remove this oxide. Fine abrasives such as aluminum oxide, ceramic beads, or glass beads may be used. Silicon carbide abrasives should be avoided. If aluminum oxide, or glass beads are used, subsequent treatments should include the use of an acid fluoride to ensure that any embedded aluminum oxide or silica is removed. However, surfaces of heat-treated alloys that are not machined or buffed should have the heat-treated film removed with a deoxidizing etch to obtain uniform electroplating results. An effective deoxidizing etch is a hot sulfuric-chromic acid solution (Appendix X1.2). Suitable proprietary deoxidizing etches including some with no chromates are available. They should be used as recommended by the manufacturer.

5.2.3 For wrought alloys of the UNS A91100 and UNS A93003 types (see Specifications B 209 and B 209M) fairly good conditioning may be obtained by using the carbonate-phosphate cleaner (Appendix X1.1) followed by a nitric acid dip at room temperature (Appendix X1.6). These alloys do not contain interfering constituents and for some applications, this method of conditioning may be ample. If a silicate inhibited cleaner is used (see 5.1) the fluoride containing smut remover (Appendix X1.4) is preferred.

NOTE 4—In accordance with current ASTM practice and for international usage, the aluminum alloys have been classified in accordance with

5 For details on the proper operation and safety precautions to be followed in vapor degreasing, see Handbook of Vapor Degreasing, ASTM STP 310, ASTM, 1976.
5.2.4 Another effective conditioning treatment for removing the surface oxide film and any undesirable microconstituents comprises the use of a hot sulfuric acid etch (Appendix X1.7). The time of the dip depends on the alloy involved. Generally the shorter time is used on castings. This treatment is satisfactory for all aluminum-magnesium alloys, both wrought and cast. It not only leaves the surface in an excellent condition for the deposition of the first metallic layer, but it also eliminates the undesirable effects of the magnesium-containing constituents in alloys of the UNS A95052, UNS A96061, and UNS A96063 types (see Specifications B 221 and B 221M).

5.3 The following are types of casting alloys containing high percentages of silicon: UNS A04130, UNS A14130,UNS A03800, (see Specification B 85), UNS A03561, and UNS A13560, (see Specification B 179). A dip at room temperature in a mixed acid solution (Appendix X1.8) containing nitric and hydrofluoric acids is recommended for conditioning the surface of these alloys. This treatment also removes the heat-treated film from unpolished, heat-treated castings.

6. Immersion Deposit/Strike Procedures

6.1 Following the cleaning and conditioning treatments, it is necessary to further treat the surface to obtain adequate adhesion of an electrodeposited metal on aluminum alloys. This section describes five commercially used procedures:

6.1.1 Zinc immersion with optional copper strike (6.3).
6.1.2 Zinc immersion with neutral nickel strike (6.4).
6.1.3 Zinc immersion with acetate buffered, nickel glycolate strike (6.5).
6.1.4 Zinc immersion with an acid or alkaline electroless nickel strike (6.6).
6.1.5 Tin immersion with bronze strike (6.7).
6.2 The immersion deposit/strike conditions recommended for each procedure give good results with many alloys of aluminum. However, some alloys and tempers may require slight modification of the processing conditions for best results.

6.3 Zinc Immersion with Optional Copper Strike:

6.3.1 In the zinc immersion step, the oxide film is removed from the surface to be electroplated and is replaced by a thin and adherent layer of metallic zinc. This provides a surface that responds to most of the electroplating procedures for plating other metals on zinc.

6.3.2 For the immersion step, a highly alkaline solution containing the following components can be used at room temperature (15 to 27°C).

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium hydroxide (commercial)</td>
<td>525 g/L</td>
</tr>
<tr>
<td>Zinc oxide (technical grade)</td>
<td>100 g/L</td>
</tr>
<tr>
<td>Ferric chloride hexahydrate</td>
<td>1.0 g/L</td>
</tr>
<tr>
<td>Rochelle salt</td>
<td>10 g/L</td>
</tr>
</tbody>
</table>

6.3.2.1 For best results, the sodium hydroxide must be low in sodium carbonate content (preferably under 2 % by weight) and the zinc oxide must be free of contamination.

Note: 5—in the zinc immersion solutions in this standard, the purity of the ingredients often plays an important role in the successful operation of the process. This is particularly true of the zinc oxide used. Contamination of the zinc oxide with lead or arsenic can be especially troublesome. Proprietary, prepared powdered or liquid zincates are frequently used therefore, since they will have had all raw materials properly checked for purity.

6.3.2.2 The thickness and quality of the immersion film are influenced by the conditions of deposition. When deposition is too rapid, heavy, coarse, crystalline, and porous, nonadherent deposits are formed. Since the thinner zinc deposits give the best results, it is recommended that the temperature of the zincate solution be kept below 27°C and the immersion time be from 30 s to 1 min.

6.3.3 A modification of the basic zincate solution in most applications gives more uniform and satisfactory results. The modified zinc immersion procedure has the following advantages: (1) more uniform coverage by subsequent electroplating baths, (2) greater operating range for the “double immersion” version of the treatment (see 6.3.5), and (3) improved resistance to corrosion on all electroplated aluminum alloys except for the UNS A92024 and UNS A97075 alloys. The modified solution is prepared by dissolving the zinc oxide in a sodium hydroxide solution and cooling to room temperature. Before the bath is diluted to volume, a water solution of ferric chloride crystals and Rochelle salt (potassium sodium tartrate) is added. The bath should be stirred while the ferric chloride-Rochelle salt solution is added. The modified zincate solution is made up as follows:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium hydroxide</td>
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<tr>
<td>Zinc oxide</td>
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<td>1.0 g/L</td>
</tr>
<tr>
<td>Rochelle salt</td>
<td>10 g/L</td>
</tr>
</tbody>
</table>

6.3.3.1 This bath should also be operated under 27°C and for immersion times of the order of 30 s to 1 min. It is recommended that Bath II be utilized whenever the “double immersion” treatment is employed. Likewise, it will be found advantageous on all wrought and cast alloys, except the UNS A92024 and UNS A97075 types, for corrosion-resistant applications.

6.3.3.2 With both of the solutions (Baths I and II), the rinse immediately after the zinc immersion step is critical. The activity of the solution increases rapidly with dilution. Because of the high concentrations used, the solution is viscous. If this viscous layer is not promptly removed in the rinsing step, the diluted film may deposit a loose, spongy zinc film in the rinse, thereby destroying an otherwise acceptable zinc film. Therefore, rinses must be strongly agitated so that this film is rapidly and uniformly removed. Spray rinsing at moderate to high pressure is preferred where the part configuration is such that the sprays can impinge on all surfaces.

6.3.4 Dilute versions of the modified zinc immersion procedures have been developed for applications where rinsing and drag-out are problems. The bath viscosity is reduced by lowering the concentration of the principal components. In

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6. There are proprietary zincate solutions available containing cations other than iron (also various other additions such as complexing agents or chelating agents or both). A solution containing copper and nickel, as well as zinc, is described by Schaer, G., Plating and Surface Finishing, 68,51 (March 1981).
using the dilute baths, a low film weight must be maintained by a closer control of operating conditions and by addition agents. Two typical dilute baths are prepared as follows:

<table>
<thead>
<tr>
<th>Zinc Immersion Solution, Bath III</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium hydroxide 50 g/L</td>
<td></td>
</tr>
<tr>
<td>Zinc oxide 5 g/L</td>
<td></td>
</tr>
<tr>
<td>Rochelle salt 50 g/L</td>
<td></td>
</tr>
<tr>
<td>Ferric chloride hexahydrate 2 g/L</td>
<td></td>
</tr>
<tr>
<td>Sodium nitrate 1 g/L</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Zinc Immersion Solution, Bath IV</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium hydroxide 120 g/L</td>
<td></td>
</tr>
<tr>
<td>Zinc oxide 20 g/L</td>
<td></td>
</tr>
<tr>
<td>Rochelle salt 50 g/L</td>
<td></td>
</tr>
<tr>
<td>Ferric chloride hexahydrate 2 g/L</td>
<td></td>
</tr>
<tr>
<td>Sodium nitrate 1 g/L</td>
<td></td>
</tr>
</tbody>
</table>

6.3.1.1 Bath IV will provide a much greater zinc reserve for high-production work with only a small sacrifice in rinsing and drag-out properties. When using these dilute solutions, the temperature must be maintained between 20 to 25°C and the immersion time must not exceed 30 s.

6.3.1.2 A more highly modified zincate (modified with copper, nickel, and iron) has been described by Wyszynski. It has much greater tolerance for variations in operating conditions, especially temperature and time of immersion, and permits processing a wider variety of alloys without resorting to the double zincating treatment (6.3.5). Because the quaternary alloy deposited by immersion is much less active than the relatively pure zinc from many immersion baths, subsequent electroplated deposits are applied with less difficulty.

6.3.5 A variation of the zincate treatment that has considerable merit consists of a double zinc immersion treatment with the first zinc layer being removed by a dip in a room temperature solution of 500 mL of concentrated, nitric acid (67 mass %, density 1.40 g/mL) diluted to 1 L. With this procedure, the first immersion dip removes the original oxide film and replaces it with a zinc layer. Removal of the zinc layer by the nitric acid dip leaves the surface in suitable condition for deposition of the final zinc immersion layer.

6.3.6 The concentrated zincate solutions (Baths I and II) are very viscous and losses occur largely from drag-out. This is advantageous as it limits the accumulation of impurities resulting from attack on the aluminum. It has the disadvantage however in that it increases the load on the waste disposal system.

6.3.7 The specific gravity of the concentrated solutions should be checked occasionally and any loss made up by adding more of the components. Loss of volume by dragout should be corrected by the addition of more solution of the specific composition. The dilute solutions (Baths III and IV and those recommended by Schae and Wyszynski) should be controlled by chemical analysis of the caustic, zinc, and modifying metal concentration.

6.3.8 When a properly conditioned aluminum alloy article is immersed in the zincate solution, the thin natural oxide film that is present on the surface of the article dissolves and, as soon as underlying aluminum is exposed, it also starts to dissolve and is immediately replaced by an equivalent weight of zinc. When the aluminum surface is completely covered with an extremely thin layer of zinc, action in this solution virtually ceases.

6.3.9 With correct procedure, the resulting zinc deposit will be fairly uniform and firmly adherent to the surface. The appearance of the surface, however, will vary with the alloy being coated as well as the rate at which the coating forms. The weight of zinc deposit should be of the order 15 to 50 µg/cm², corresponding to a thickness of 20 to 70 nm. Generally, it is desirable to limit the weight of the deposit to not over 30 µg/cm². The thinner and more uniform zinc deposits are the most suitable for electroplating preparation and for the performance of electroplated coatings in service. Heavy zinc deposits tend to be spongy and less adherent and do not provide as good a surface for obtaining adherence as the thinner deposits. The weight of the zinc deposit will vary with the alloy and the conditioning treatment that is used.

6.3.10 After the surface of an aluminum alloy article has been conditioned and the zinc immersion deposit has been formed, other metals can be electroplated on this surface by any of the methods suitable for electroplating on zinc. Ordinarily, it is advisable to apply a suitable copper strike over the zinc-immersion layer before other metals are deposited. Silver, brass, zinc, nickel, or chromium, however, may be deposited on the zinc immersion layer provided the electroplating procedures are suitable for electroplating over zinc.

6.3.11 When a copper strike is to be used over the zinc immersion layer, a tartrate-type copper cyanide solution operated as follows is recommended:

<table>
<thead>
<tr>
<th>Tartrate-Type Copper Strike Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper cyanide 42.0 g/L</td>
</tr>
<tr>
<td>Total sodium cyanide 50.0 to 55.0 g/L</td>
</tr>
<tr>
<td>Sodium carbonate 30.0 g/L</td>
</tr>
<tr>
<td>Rochelle salt 60.0 g/L</td>
</tr>
<tr>
<td>Free sodium cyanide 5.5 to 10.5 g/L</td>
</tr>
</tbody>
</table>

The work is introduced with the electrical circuit connection made for “live” entry (cathodic).

6.3.11.1 Reduce cathode current density to 130 A/m² and electroplate for an addition 3 to 5 min.

6.3.12 After this strike, the work can be transferred to other standard electroplating solutions for further electroplating.

6.4 Zinc Immersion/Neutral Nickel Strike. Zinc Immersion/Neutral Nickel Strike:

6.4.1 Aluminum parts with cleaned and conditioned surfaces are given a double zinc immersion treatment as described in 6.3.3 and 6.3.5. Recommended times for the first and second zinc immersion are 45 s and 30 s, respectively.

6.4.2 After water rinsing, the zincated parts are given a nickel strike as follows:

| Proprietary chemical available from Atotech USA, Rock Hill, SC 29731.
|Proprietary chemical available from Atotech USA, Rock Hill, SC 29731.


10 US Patent 3,417,005 assigned to General Motors Corporation.


12 Proprietary chemical available from Atotech USA, Rock Hill, SC 29731.
Neutral Nickel Strike Treatment

**Electrolyte:**
- Nickel sulfate $7H_2O$: 142 g/L
- Ammonium sulfate: 34 g/L
- Nickel chloride $6H_2O$: 30 g/L
- Sodium citrate: 140 g/L
- Sodium gluconate: 30 g/L
- Temperature: 57 to 66°C
- pH at 60°C: 6.8 to 7.2
- Current density: 950 to 1300 A/m²
- Time: 30 to 45 s
- Agitation: Non-air type

6.4.2.3 Reduce cathode current density to 400 to 550 A/m² and electroplate for an additional 3 to 5 min.

6.4.3 After receiving the above neutral nickel strike, the aluminum parts can be electroplated with other metals using standard electroplating solutions.

**6.5 Zinc Immersion/Nickel Glycolate Strike:**

6.5.1 Aluminum parts with cleaned and conditioned surfaces are given a zinc immersion treatment as described in 6.3. A single or a double immersion treatment may be used.

6.5.2 After water rinsing, the zincated parts are given a nickel strike in the mildly acid electrolyte with the following process conditions:

<table>
<thead>
<tr>
<th>Acetate Buffered Nickel Glycolate Strike</th>
<th>Treatment Electrolyte</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel acetate $4H_2O$</td>
<td>65 g/L</td>
</tr>
<tr>
<td>Boric acid</td>
<td>45 g/L</td>
</tr>
<tr>
<td>70% Glycolic acid</td>
<td>60 mL/L</td>
</tr>
<tr>
<td>Saccharin</td>
<td>1.5 g/L</td>
</tr>
<tr>
<td>Sodium acetate</td>
<td>50 g/L</td>
</tr>
<tr>
<td>Temperature</td>
<td>Room</td>
</tr>
<tr>
<td>pH</td>
<td>5.5 to 6.8</td>
</tr>
<tr>
<td>Wetting agent</td>
<td>Optimum amount for surfactant used</td>
</tr>
<tr>
<td>Current density</td>
<td>250 A/m²</td>
</tr>
<tr>
<td>Time</td>
<td>2 min</td>
</tr>
<tr>
<td>Anode</td>
<td>Nickel or inert</td>
</tr>
<tr>
<td>Agitation</td>
<td>Work or solution movement</td>
</tr>
</tbody>
</table>

6.5.3 After receiving the above nickel glycolate strike, the aluminum parts can be electroplated with other metals, using standard electroplating solutions.

**6.6 Zinc Immersion/Electroless Nickel Strike:**

6.6.1 Aluminum parts with cleaned and conditioned surfaces are given a zinc immersion treatment as described in 6.3. A single or double immersion treatment may be used.

6.6.2 After water rinsing, the zincated parts are given an electroless nickel strike in the electroless nickel solution of choice. Because of the need for carefully buffered conditions and tolerance for dissolved zinc, these solutions are generally proprietary. The operating conditions recommended by the manufacturer should be followed carefully. In particular, it should be noted that these solutions may have deposition rates that vary with different sources, operating conditions and age. Immersion time must be adequate to ensure complete, pore-free coverage of all surfaces.

6.6.3 After receiving the electroless nickel strike, the aluminum parts can be electroplated with other metals using standard electroplating conditions, or transferred to a different electroless nickel bath for the application of electroless nickel deposits for engineering purposes.

6.7 Tin Immersion/Bronze Strike:

6.7.1 The aluminum parts should be cleaned as described in 5.1. They should then be conditioned preferably in an alkaline etch followed by rinsing and desmutting in a nitric acid plus ammonium bifluoride solution, as described in 5.2.1.

6.7.2 After water rinsing, the cleaned and conditioned aluminum parts are subjected to a tin activation treatment. This is accomplished either by simple immersion, or by “live” (current on) entry (cathodic) of the work into a proprietary aqueous stannate bath, for 30 s at 26 to 30°C.

6.7.3 Without rinsing and with minimum time delay, the tin-activated aluminum parts are transferred into a proprietary, aqueous bronze cyanide bath where they are given a strike of 3 to 4 min at 26 to 30°C with a cathodic current density of 320 to 540 A/m².

6.7.4 After the bronze strike and water rinsing, other metals can be electroplated on the aluminum parts using standard electroplating solutions.

7. Safety Precautions

7.1 Some chemical solutions are exothermic upon mixing or in use, thereby requiring cooling and proper containment to prevent injury to personnel.

**Note 6—Caution:** Care in the handling and use of all cyanide-containing salts and solutions must be exercised. Adequate rinsing between cyanide and acid process solutions must be performed.
XI. SOLUTIONS FOR CLEANING AND CONDITIONING ALUMINUM ALLOYS

X1. Carbonate-Phosphate Cleaner:
Sodium carbonate, anhydrous 25 g/L
Trisodium phosphate, anhydrous 25 g/L
Temperature 60 to 80°C
Time 1 to 3 min
Container steel

X1.1.1 Carbonate-Phosphate Cleaner:
Sodium carbonate, anhydrous 25 g/L
Trisodium phosphate, anhydrous 25 g/L
Temperature 60 to 80°C
Time 1 to 3 min
Container steel

X1.2 Deoxidizing Etch:
Sulfuric acid (density 1.83 g/mL) 100 mL
Chromic acid, CrO₃ 35 g
Water to 1 L
Time 2 to 5 min
Temperature 70 to 80°C
Container lined with lead

NOTE X1.1—Warning: Dissolve the chromic acid in approximately 800 mL of water, then slowly add the sulfuric acid with rapid mixing; when the solution has cooled to room temperature, dilute to 1 L. Fumes are toxic. Use exhaust.

X1.2.1 This solution may be used at room temperature for periods of 5 to 30 min to remove many types of oxides. Operation at this lower temperature offers greater safety and reduces the amount of hazardous fumes evolved.

X1.3 Caustic Dip:
Sodium hydroxide 50 g/L
Time 30 s to 1 min
Temperature 50°C
Container Steel

NOTE X1.2—Warning: Fumes are toxic. Use exhaust.

X1.4 Acid Desmutter:
Nitric acid (density 1.4) 500 to 700 mL/L
Ammonium bifluoride 30 to 120 g/L
Time 30 s
Temperature 20 to 25°C
Container steel with a suitable plastic lining

NOTE X1.3—Warning: Fumes are toxic. Use exhaust.

X1.4.1 The activity and aggressiveness of this desmutter may be controlled by varying the concentrations as indicated. Increasing the nitric acid concentration decreases activity; increasing the ammonium bifluoride concentration increases activity.

X1.5 Alternative Acid Desmutter:
Sulfuric acid (H₂SO₄ 93 mass %, density 1.83 g/mL) 100 mL
Hydrogen peroxide (H₂O₂ 32.5 mass %, stabilized for use with nonferrous metals) 50 mL
Water to 1 L
Time 15 s to 1 min
Temperature room
Container 300 series stainless steel or container with a suitable plastic lining

NOTE X1.4—Warning: The acid should be slowly added to 90 vol % of the water required with rapid stirring. When the solution cools to room temperature, add the hydrogen peroxide and dilute to exact volume. Fumes are toxic. Use exhaust.

X1.6 Nitric Acid Dip:
Commercial nitric acid (67 mass %, density 1.4) 500 mL
Water to 1 L
Temperature room
Container steel-lined with suitable plastic or UNS S30403, UNS S31603, or UNS S34700 stainless steel

NOTE X1.5—Warning: Fumes are toxic. Use exhaust.

X1.7 Sulfuric Acid Dip:
Sulfuric acid (H₂SO₄ 93 mass %, density 1.83 g/mL) 150 mL
Water to 1 L
Temperature 80°C
Time 2 to 5 min
Container lined with lead or a suitable plastic

NOTE X1.6—Warning: The acid should be slowly added to the approximate amount of water required with rapid mixing. When the solution cools to room temperature, dilute to exact volume.

X1.8 Mixed Acid Dip:
Commercial nitric acid, (67 mass %, density 1.4 g/mL) 750 mL
Commercial hydrofluoric acid (48 mass %, density 1.16 g/mL) 250 mL
Time 3 to 5 s
Container steel-lined with a suitable plastic or carbon brick or both

NOTE X1.7—Warning: Fumes are toxic. Use exhaust.

X1.9 Universal Deoxidizer:
Commercial nitric acid (67 mass %, density 1.4 g/mL) 500 mL
Sulfuric acid (H₂SO₄ density 1.84 g/mL) 250 mL
Water 250 mL
Ammonium bifluoride 60 g/L

NOTE X1.8—Caution: Add the nitric acid to the water slowly with vigorous agitation. Allow to cool to room temperature. Slowly add the sulfuric acid to the mixed acids with vigorous agitation. Allow to cool to room temperature. Dissolve required amount of ammonium bifluoride. Adjust to final volume with water if necessary. The operation may have to be interrupted several times to permit cooling! The temperature during mixing must never be allowed to exceed the safe operating limits of the lining or plastic container or irreparable damage may occur.

X1.9.1 This acid dip is applicable to almost all alloys. It is particularly useful on alloys containing magnesium.

NOTE X1.9—Warning: Fumes are toxic. Use exhaust.
Standard Practice for Preparation of and Electroplating on Stainless Steel

This standard is issued under the fixed designation B 254; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 Various metals are electrodeposited on stainless steel for color matching, lubrication during cold heading, spring-coiling and wire-drawing operations, reduction of scaling at high temperatures, improvement of wettability (as in fountain pens), improvement of heat and electrical conductance, prevention of galling, jewelry decoration, and prevention of superficial rusting.

1.2 This practice is presented as an aid to electroplaters and finishing engineers, confronted with problems inherent in the electrodeposition of metals on stainless steel. It is not a standardized procedure but a guide to the production of smooth adherent electrodeposits on stainless steel.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

A 380 Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems

3. Nature of Stainless Steel

3.1 Because previous metal treatment may have a more pronounced effect on the final finish when stainless steel is being electroplated, the metal finisher should become acquainted with the fabrication procedure, grade, and mill finish of the stainless steel with which he is working before outlining his electrodeposition procedure (see Appendix X1).

3.2 Stainless steel surfaces are normally resistant to a wide variety of corrosive elements. This property is the result of a thin transparent film of oxides present on the surface. Because this film rapidly reforms after it has been stripped off or penetrated, it protects stainless steel against corrosion. An adherent electrodeposits cannot be obtained over the oxide film normally present on stainless steel. However, once this film is removed by surface activation and kept from reforming while the surface is covered with an electrodeposits, any of the commonly electroplated metals may be electrodepositioned successfully on stainless steel.

3.3 Where the finished product is to be subjected to severe exposure, the deposit produced by the proposed electroplating sequence should be tested under similar exposure conditions before adoption, to determine whether the natural corrosion resistance of the stainless steel has been impaired by the presence of the electrodeposits.

4. Nature of Cleaning

4.1 The preparation of stainless steel for electroplating involves three basic steps in the following order:

4.1.1 Removal of scale. If scale removal is necessary, one of the methods outlined in Appendix X2 may be used (Note 1).

4.1.2 Removal of oil, grease, or other foreign material by cleaning, and

4.1.3 Activation immediately before electroplating.

4.2 Precleaning—Removal of fabricating lubricants and finishing compounds from the stainless steel may have to be undertaken immediately following the fabrication or finishing operation (Note 2).

4.3 Electrocleaning—Anodic cleaning is generally preferred (Note 3).

4.4 Metal Lubricants—Metal lubricants such as copper, lead, or cadmium, applied to stainless steel wire for cold heading, wire drawing, or spring forming are removed by immersion in a solution of 200 mL of concentrated, 67 mass %, nitric acid (density 1.40 g/mL) diluted to 1 L at 50 to 60°C. See Practice A 380.

NOTE 1—Oil, grease or other fabricating lubricants should be removed by cleaning before heat treating.

NOTE 2—Spray cleaning with a nozzle pressure of 200 to 400 kPa (30 to 60 psi) in a power washer, using an alkaline or emulsion-type cleaner,
is the generally preferred method, especially for the removal of heavy drawing, buffing, or polishing compounds. Soak cleaning or vapor degreasing may also be used. Extreme examples of such compounds are drawing or stamping lubricants containing unsaturated oils, which, if left on the surface, form by air-oxidation tenacious films that are very difficult to remove.

**Note 3**—When brightness is important, alkalinity, current density, and temperature should be kept as low as the part will permit. This is an essential requirement when cleaning work on racks bearing auxiliary lead anodes or when high chromium alloys (such as UNS Types S44200 and S44600) are being cleaned.

5. **Cleaning Solutions**

5.1 The types of solution control, electrodes, heating coils, and rinse tanks normally used for cleaning carbon steel are satisfactory for stainless steel. Equipment previously used for the cleaning or processing of carbon steel should not be used. See Practice A 380.

6. **Racking**

6.1 The general principles of good racking as used in chromium electroplating processes apply. However, the high electrical resistance of stainless steel requires rack construction methods that minimize potential contact problems and increase the number of contact points.

**Note 4**—Because of the high electrical resistance of stainless steel, especially in fine-coiled wire articles such as watch bands, chains, jewelry, etc., it is necessary to provide a larger number of contacts. As an example, a watch band 110 mm long made of 1.0-mm diameter wire has been found to require at least three contacts.

7. **Activation**

7.1 After the cleaning operation and before the electroplating operation, the parts must be completely activated, that is, the thin transparent film of oxides must be removed from the surface to be electroplated (Note 5). This film will reform if the parts are allowed to dry or are exposed to oxygen-containing solutions. For this reason, the shortest interval practicable should elapse between the time the parts are removed from the activating solution and covered by the electrodeposits, unless a simultaneous activation-electroplating procedure is used.

**Note 5**—The etching practice may be more severe for nondecorative applications than for decorative applications.

7.2 The following activating procedures have been used. The procedure selected will depend upon the nature of the part and preceding or subsequent processes (see 7.7). In the following solution formulas, the concentrations are expressed on a volume basis as follows:

**Liquids:** as volume per litre of solution

**Solids:** as mass per litre of solution

7.3 The commercial grade acids and salts used in the formulas include:

- **Sulfuric acid**: 93 mass %; density 1.83 g/mL
- **Hydrochloric acid**: 31 mass %; density 1.16 g/mL
- **Nickel chloride**: NiCl2·6H2O
- **Copper sulfate**: CuSO4·5H2O

**Warning**—Sulfuric acid should be slowly added to the approximate amount of water required with rapid mixing, and then after cooling, diluted to exact volume.

7.4 **Cathodic Treatments**

7.4.1 Sulfuric acid

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>50 to 500 mL/L</td>
</tr>
<tr>
<td>Temperature</td>
<td>to 1 L</td>
</tr>
<tr>
<td>Time</td>
<td>1 to 5 min</td>
</tr>
<tr>
<td>Current density</td>
<td>0.54 A/dm²</td>
</tr>
<tr>
<td>Anodes</td>
<td>Pure lead</td>
</tr>
</tbody>
</table>

7.4.2.4 **Hydrochloric acid**

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>50 to 500 mL/L</td>
</tr>
<tr>
<td>Temperature</td>
<td>to 1 L</td>
</tr>
<tr>
<td>Time</td>
<td>1 to 5 min</td>
</tr>
<tr>
<td>Current density</td>
<td>2.15 A/dm²</td>
</tr>
<tr>
<td>Anodes</td>
<td>Electrolytic nickel strip or nickel bar</td>
</tr>
</tbody>
</table>

**Note A**—See Patent No. 2,133,996.

7.4.3 After immersion in a solution containing 100 to 300 mL/L of hydrochloric acid diluted to 1 L at room temperature for 30 to 60 s, treat cathodically in:

- **Sulfuric acid**: 50 to 500 mL/L
- **Water**: to 1 L
- **Temperature**: room
- **Current density**: 0.54 to 2.7 A/dm²
- **Anodes**: Pure lead

7.5 **Immersion Treatments**

7.5.1 Immerse in a solution of sulfuric acid containing 200 to 500 mL of acid diluted to 1 L at 65 to 80°C (with the higher temperature for the lower concentration) for at least 1 min after gassing starts. If gassing does not start within 1 min after the parts have been immersed, touch them with a carbon-steel bar or rod. This activation treatment will produce a dark, adherent smut that is removed in the electroplating bath. A cathodic current of at least 0.54 A/dm² may be used to accelerate activation. Lead anodes are suitable for this solution.

7.5.2 Immerse in the following solution:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrochloric acid</td>
<td>1 mL</td>
</tr>
<tr>
<td>Sulfuric acid</td>
<td>10 mL</td>
</tr>
<tr>
<td>Water</td>
<td>to 1 L</td>
</tr>
<tr>
<td>Temperature</td>
<td>Room</td>
</tr>
<tr>
<td>Time</td>
<td>26 s</td>
</tr>
</tbody>
</table>

**Note 6**—This practice has been used with success for chromium electroplating on stainless steel automobile parts in a conveyorized process. It is not recommended before copper or nickel electroplating.

7.6 **Simultaneous Activation-Electroplating Treatments**

7.6.1 Nickel chloride

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>126 mL</td>
</tr>
<tr>
<td>Temperature</td>
<td>Room</td>
</tr>
<tr>
<td>Electrodes</td>
<td>Nickel</td>
</tr>
</tbody>
</table>


7.6.1.1 **Anodic Treatment**

<table>
<thead>
<tr>
<th>Current density</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.2 A/dm²</td>
<td>2 min</td>
</tr>
</tbody>
</table>

7.6.1.2 Followed by **Cathodic Treatment**

<table>
<thead>
<tr>
<th>Current density</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.2 A/dm²</td>
<td>6 min</td>
</tr>
</tbody>
</table>

7.6.2 Nickel chloride

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>to 1 L</td>
</tr>
<tr>
<td>Electrodes</td>
<td>Nickel</td>
</tr>
<tr>
<td>Temperature</td>
<td>Room</td>
</tr>
<tr>
<td>Current density (cathodic)</td>
<td>5.4 to 21.5 A/dm²</td>
</tr>
<tr>
<td>Time</td>
<td>2 to 4 min</td>
</tr>
</tbody>
</table>


7.6.2.1 Bath may require cooling or reduction in hydrochloric acid content if temperature exceeds 30°C.

7.6.3 Nickel chloride

<table>
<thead>
<tr>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>30 to 300 g/L</td>
</tr>
</tbody>
</table>
Hydrochloric acid 15 to 160 mL/L
Water to 1 L
Electrodes nickel
Temperature room
Current density 0.55 to 10.75 A/dm²
Time ½ to 5 min

7.6.4 Hydrochloric acid undiluted commercial grade (7.2)
Copper sulfate 0.4 g/L
Electrodes nickel
Temperature room
Current density 4.5 to 6.6 A/dm²
Time 1 to 5 min

Note 7—Nickel anode materials containing greater than 0.01 % sulfur are not recommended for use in acid nickel strike baths operated at pH 0.5, or lower, to avoid oxidation of sulfides by hydrochloric acid (see 7.6.1-7.6.4, and 7.7).

7.7 A combination of more than one type of treatment may be necessary to ensure a high degree of adhesion. For example, the following has been used in the automotive industry for nickel plating on UNS Type S30200 stainless steel:

Sulfuric acid 650 mL
Water to 1 L
Potential (cathodic) 10 V
Electrodes lead
Temperature room
Time 2 min

Followed by:

Nickel chloride 240 g
Hydrochloric acid 120 mL
Water to 1 L
Electrodes nickel
Temperature room
Time 2 min
Current density (cathodic) 16.2 A/dm²

This is followed by transfer without rinsing to a Watts (or higher chloride) nickel bath with a pH of 1.5 to 2.0.

8. Rinsing

8.1 The parts should be transferred to the cold-water rinse and to the plating solution as rapidly as practicable after the activating procedure; otherwise the surface will passivate itself and the electrodeposit will not be adherent.

8.1.1 The rinse water should be kept slightly acid (approximately pH of 2.5 to 3.5). The acid carryover from the activation operation will maintain this pH in many instances.

8.1.2 In conveyerized operations where trace contamination of plating solutions with chloride and sulfate from activating solutions will produce an unsatisfactory electrodeposit, spray-rinse operations subsequent to the activation treatment will remove these contaminants.

8.1.3 If the simultaneous activation-plating procedure is employed and nickel plating follows, the intermediate rinse need only be superficial and the length of transfer time is not so important.

9. Electroplating

9.1 An adherent electrodeposit of commonly electroplated metals (cadmium, copper, brass, chromium, gold, nickel, or silver) may be electrodeposited directly on stainless steel provided the surface of the stainless steel is active.

Note 8—Nickel may be electrodeposited at normal current densities directly on properly activated stainless steel from standard nickel-electroplating solutions if the pH of the solution is between 2 and 4. A pH of 2 is preferred.

Note 9—When a chromium-electroplating solution containing 400 g/L of chromic acid is used for decorative chromium electroplating, better coverage and a wider bright range is obtained by operating at a current density of 16.2 A/dm² and 49°C.

Note 10—A bright nickel electroplate under chromium, preceded by one of the simultaneous activation-electroplating treatments, may often be used to advantage for better color matching and elimination of chromium buffing.

9.2 Where practical, the parts should have the current applied during entry into the electroplating solution.

10. Stripping

10.1 Nitric acid is the preferred stripping solution.

10.2 Decorative chromium electrodeposits have been stripped in a solution of 500 mL of concentrated, 31 mass % hydrochloric acid (density 1.16 g/mL) diluted to 1 L at 45 to 50°C for 1 min.

Note 11—Overstripping will result in etching.

Note 12—Decorative chromium electrodeposits may also be stripped anodically in any alkaline solution.

10.3 Cadmium is stripped successfully without current by immersion in a solution of 120 g/L of ammonium nitrate.

11. Post Electroplating Operations

11.1 Post electroplating operations such as stress relieving, buffing or coloring, and forming or drawing may be applied to stainless steel in the same manner as to any other basis metal, as long as the natural differences in the characteristic of the stainless steel are taken into consideration. The stainless steel supplier should be consulted for guidance in regard to these characteristics.

12. Test Methods

12.1 The methods of testing for thickness, hardness, and adhesion of electrodeposits applied with the usual basis metals may be employed for similar tests on stainless steel.

Note 13—An exception to this is the determination of the thickness of chromium on stainless steel by the hydrochloric acid drop method. Because gassing continues after the chromium coating has been penetrated, the accuracy of this method may be questionable for this application.
APPENDIXES
(Nonmandatory Information)

X1. STAINLESS STEEL GRADES

X1.1 There are many standard grades and many more special grades of stainless steel. Each grade has a specific use, and each may present an individual finishing problem. The common grades are classified as:

X1.1.1 Martensitic, or hardenable magnetic chromium grades. UNS Types S40300, S40500, S40600, S41000, S41400, S41600, S42000, S43100, S44002, S44003, and S44004 are the more common alloys of this grade.

X1.1.2 Ferritic, or nonhardenable magnetic chromium grades. UNS Types S43000, S43020, and S44600 are alloys of this type.

X1.1.3 Austenitic, or nonhardenable, nonmagnetic chromium-nickel alloys. UNS Types S20100, S20200, S30100, S30200, S30400, S30500, S30800, S30900, S31000, S31600, S31700, S32100, and S34700 are the more common alloys of this type.

X1.1.4 Precipitation-hardening or hardenable chromium nickel-alloys. UNS Types S17400, S17700, S15700, and S35000, are some examples of this type (Note X1.1).

NOTE X1.1—The precipitation-hardening types of stainless steel should be electroplatable by one or more of the procedures in this practice, but difficulties may be encountered with types containing relatively high levels of manganese, molybdenum, and silicon. In the fully aged condition, these grades of stainless steel may be susceptible to hydrogen embrittlement due to electroplating (see X3.3).

X2. MILL FINISHES

X2.1 Stainless steels can be obtained with a variety of finishes.

X2.1.1 Bar and Wire Stock—The common finishes applied to bar and wire stock are:

X2.1.1.1 Hot-rolled (scale present),
X2.1.1.2 Hot-rolled and pickled (scale removed),
X2.1.1.3 Rough-turned (round bars only),
X2.1.1.4 Cold-drawn,
X2.1.1.5 Centerless-ground (round stock only), and
X2.1.1.6 Polished (rounded stock only).

X2.1.2 Sheet and Strip Stock—The common finishes applied to sheet and strip stock are:

<table>
<thead>
<tr>
<th>Finish Description</th>
<th>Sheet Finish Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hot-rolled, annealed and pickled (white-pickled finish)</td>
<td>No. 1 Hot-rolled, annealed and pickled (white-pickled finish)</td>
</tr>
<tr>
<td>Bright full cold-rolled finish</td>
<td>No. 2B Bright full cold-rolled finish</td>
</tr>
<tr>
<td>Dull full cold-rolled finish</td>
<td>No. 2D Dull full cold-rolled finish</td>
</tr>
<tr>
<td>Standard polish (bright finish with fine polishing lines)</td>
<td>No. 4 Standard polish (bright finish with fine polishing lines)</td>
</tr>
<tr>
<td>Standard polish tampico finish (soft luster, satin finish)</td>
<td>No. 6 Standard polish tampico finish (soft luster, satin finish)</td>
</tr>
<tr>
<td>High luster polish (glossy, bright, buffed finish)</td>
<td>No. 7 High luster polish (glossy, bright, buffed finish)</td>
</tr>
</tbody>
</table>

X3. CONDITION FOR ELECTRODEPOSITION

X3.1 All of the common grades of stainless steel with normal heat treatments, in the full range of hardness, and in the forms mentioned above, with any surface finish free of scale, and when properly activated may be covered with electrodeposits of commonly electrodeposited metals.

X3.2 The smoothness of stainless steel surface to be electroplated should be comparable with the requirement of the finished product. Grinding checks, glazing, and heat-treating stains on the stainless steel should be avoided.

X3.3 The high-carbon grades (UNS Types 42000, 44002, 44003, and 44004) may be subject to hydrogen embrittlement during electroplating operations. This may be overcome by heating the electroplated articles at 150 to 260°C for 1 to 3 h after electroplating.

X4. EFFECT OF FABRICATION

X4.1 Stainless steel alloys are susceptible to improper fabricating procedures in forming or heat-treating, therefore, an investigation of difficulties encountered in producing an adherent deposit on stainless steel should start with the procedures used for fabrication. Technical assistance for such investigations is furnished by the suppliers of stainless steel. Their assistance may often be helpful in planning for electroplating on stainless steel.
X5. METHODS OF SCALE REMOVAL

X5.1 Scale on stainless steel such as that resulting from treatment during or subsequent to fabrication or forging must be completely removed to obtain satisfactory adhesion of the electrodeposit. The scale formed on stainless steels is generally tenacious and may be somewhat more difficult to remove than scale on carbon steel. Mechanical methods, such as sandblasting and tumbling, and chemical methods, such as pickling in acid solutions or in molten salts include:

X5.1.1 Blasting:

X5.1.1.1 This treatment serves to break up the hard scale and render it more responsive to pickling operations. The scale that usually develops on the martensitic and ferritic grades because of high annealing temperatures and long annealing cycles can be broken up only by sandblasting. Blasting is not usually employed on products made of sheet, strip, or tubing. Even if all scale has apparently been removed by blasting, it is necessary to pickle the parts before electroplating.

X5.1.1.2 Air-driven or rotary-impelled iron-free sand, stainless steel shot or aluminum oxide grit may be used. Ordinary steel shot, shot turkish emery, natural corundum, and other iron-containing abrasives are undesirable because they are likely to embed rust-producing iron in the surfaces being processed. Fragments, deburred particles, or other fines must be kept out of the blasting medium. The operation must be controlled very carefully, and the force of the blasting stream must be kept diffused to prevent unevenness of the surface.

X5.1.2 Shot Peening or Wheel Abraiding—This cold-working process may also be used for scale removal.

X5.1.3 Tumbling—This far gentler method of scale removal is generally used on parts that have been stamped, machined, or drawn to a practically completed shape.

X5.1.4 Wet Tumbling—A final finish approaching that of a color-buffed finish may be obtained by the proper selection of abrasive, speed of rotation, and time. The tumbling media should be completely removed if the parts are to be stored in a humid atmosphere 24 h or more before activation. A solution consisting of 200 to 400 mL of concentrated 63 mass % nitric acid (density 1.40 g/mL) diluted to 1 L operated at 50 to 60°C, followed by a cold water rinse, will effectively remove the tumbling media.

X5.1.5 Wire Brushing and Grinding—These procedures may be used for scale removal under certain conditions. Only stainless steel wire brushes should be used for wire brushing scale from stainless steel. Particles of steel from a carbon steel brush will embed themselves in the stainless steel and show rust in a very short time. Pickling after wire brushing is not very effective in removing embedded particles. Stainless steel wire brushes are commercially available. Commercial grinding compounds free from iron are probably more economical to use than stainless steel wire brushes.

X5.1.6 Pickling—This procedure usually follows preliminary scale removal by other means.

X5.1.6.1 The scale formed on stainless steel during forging, heat treating, welding, and other high-temperature processing will vary greatly with the grade and heat-treating environment. If it is heavy, blasting or shot peening before pickling will prove economical and time-saving because it will remove much of the brittle scale and crack the underlying layers, thus affording a more uniform removal in the pickling baths.

X5.1.6.2 The martensitic and ferritic grades are the more difficult to pickle. Full annealing of these alloys causes a very tight scale to develop that usually requires blasting to break. Sandblasting should always be followed by pickling to remove mill scale or particles of iron embedded in the surface.

X5.1.6.3 The high-carbon grades (UNS Types S42000, S44002, S44003, and S44004) should never be acid pickled in the fully hardened condition. Stress relieving at 150 to 370°C for 1 to 2 h after hardening and before pickling is essential to avoid pickling cracks. These grades require careful watching during the pickling operation to prevent severe etching. Polishing with iron-free abrasive grit or sandblasting are preferred methods of scale removal to avoid etching.

X5.1.6.4 One recommended procedure for pickling consists of two steps: scale softening and partial removal in sulfuric or hydrochloric acid, followed by final scale removal in a nitric-hydrofluoric acid solution. In the following solution formulas, the concentrations are expressed on a volume basis employing the following commercial grade acids:

- Sulfuric acid: 93 mass %; density 1.83 g/mL
- Hydrochloric acid: 31 mass %; density 1.16 g/mL
- Nitric acid: 63 mass % HNO₃; density 1.38 g/mL
- Hydrofluoric acid: 52 mass %; density 1.20 g/mL

See 7.3.

(1) The first step employs an 80 to 110-mL solution of sulfuric acid diluted to 1 L at 65 to 70°C for 10 to 45 min or a 100 to 150 mL solution of hydrochloric acid diluted to 1 L at 50 to 60°C for 30 to 90 min. A suitable inhibitor should be used, and the parts should be rinsed thoroughly before they are advanced to the second step. (2) The second step consists of immersion in a solution containing 60 to 100 mL of nitric acid and 15 mL of hydrofluoric acid diluted to 1 L at room temperature for 10 to 30 min, or until the smut has been removed. The time, for smut removal, required for the austenitic grades (except the free-machining 18Cr-8Ni grade, UNS Type 30300) may be shortened by the use of a solution containing 100 mL of nitric acid and 15 mL of hydrofluoric acid diluted to 1 L at 60 to 70°C until the smut has been removed. This latter method should not be used on the martensitic or ferritic grades nor on UNS Type S30300 austenitic stainless steel.

X5.1.7 Molten Salt Treatment—Several molten salt treatments are suitable for scale removal.

X5.1.7.1 The molten sodium hydride bath—acid treatment³ for scale removal is widely used commercially. In this process, the reduction of the scale is accomplished by immersion of the work in a molten sodium hydroxide bath containing 1½ to 2 mass % of sodium hydride. The scale is reduced in a period of time varying from a few seconds to about 15 min after the work


9
has reached the operating temperature of 400°C. The work is then removed, quenched in water, given a brief dip in a sulfuric acid solution and then brightened in a solution containing 60 to 80 mL of nitric acid diluted to 1 L or a solution containing 100 mL of nitric acid and 20 mL of hydrofluoric acid diluted to 1 L. Although this process has been used primarily in large scale continuous units, intermittent use for smaller batch-type operations is practicable.

X5.1.7.2 Another molten-salt bath used successfully for scale treatment is the proprietary bath\(^4\) containing sodium hydroxide and oxidizing salts.

X5.1.7.3 Electrolytic molten baths may also be employed.

X5.1.7.4 Molten salt methods are especially suited for pickling stainless steel because the molten caustic does not attack the metal in any way.

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Standard Test Method for
Apparent Porosity in Cemented Carbides

This standard is issued under the fixed designation B 276; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method specifies procedures for the metallographic determination of apparent porosity in cemented carbides.

NOTE 1—The term “apparent porosity” is construed to mean all microstructures observed on a properly prepared, unetched surface, including structures resulting from uncombined carbon, non-metallic inclusions, etc., as well as true, inherent porosity.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   B 243 Terminology of Powder Metallurgy
   B 665 Practice for Metallographic Sample Preparation of Cemented Tungsten Carbides

2.2 ASTM Adjunct:
   ADJA0276 Apparent Porosity (5 prints of 5 photomicrographs each)

2.3 ISO Standard:
   ISO 4505 Hardmetals—Metallographic Determination of Porosity and Uncombined Carbon

3. Terminology

3.1 Definitions—Definition of powder metallurgy terms can be found in Terminology B 243.

4. Significance and Use

4.1 Cemented carbide materials may contain small voids that, depending on the application, may affect the performance of the product. To assist users in specifying the maximum acceptable level of porosity, this test method illustrates a broad range of porosity levels for each of three porosity types. This test method is not intended to be used as a specification, but the levels shown here may be cited in specifications written by producers and users of cemented carbides.

5. Interferences

5.1 Lack of adequate pressure on the specimen during polishing may result in material being torn from the surface of the specimen. This condition may be erroneously interpreted as porosity.

6. Apparatus

6.1 Ordinary laboratory apparatus.

6.2 A metallographic microscope permitting observation and measurement up to a magnification of 200 diameters.

6.3 Equipment for the metallographic preparation of test specimens.

7. Specimen Preparation

7.1 Where possible, specimens should be metallographically mounted in a plastic material, so that they can be polished without rounding the edges. Larger specimens may be polished without mounting. When the specimens are too large, they shall be sectioned using a diamond cut-off wheel or by fracturing (appropriate safety precautions shall be utilized when fracturing a specimen). The area selected for examination shall represent, as nearly as possible, the entire cross section.

7.2 The specimen shall be prepared for metallographic examination. The surface to be examined shall be unetched and free of grinding and polishing marks.

NOTE 2—A suitable procedure is described in Practice B 665.

8. Procedure

8.1 Pore size shall be defined as the maximum dimension of the pore. Make special reference to the presence of cracks and slits, as well as nonmetallic inclusions.

8.2 Classification of apparent porosity is based entirely on comparison of the microstructures found with the photomicrographs in Figs. 1-5. This can be accomplished by scanning the specimen surface under the microscope at the appropriate magnification, and noting which of the photomicrographs most

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1 This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.06 on Cemented Carbides.


2 Annual Book of ASTM Standards, Vol 02.05.

3 Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.
nearly represents the fields observed. Choose an area fully representative of the specimen surface for comparison with the photomicrographs.

8.2.1 If the porosity of uncombined carbon is not uniform over the area of the test-piece section being examined, the locations on the section to which the evaluation refers must be identified, for example as top, bottom, edge, rim (case), core, etc.

8.2.2 Classify pores up to 10 µm as Type A (see Fig. 1 and Fig. 2), and examine at a magnification of 100 or 200 diameters. Report the porosity level by reference to the appropriate photomicrograph, and designate as A02, A04, A06, or A08.

8.2.3 Classify pores in the range from 10 to 25 µm as Type B (see Fig. 3), and examine at a magnification of 100 diameters. Report the porosity level by reference to the appropriate photomicrograph, and designate as B02, B04, B06, or B08.

8.2.4 Count and report pores larger than 25 µm as the number of pores per unit area. Choose the size ranges as follows: 25 to 75 µm, 75 to 125 µm, and over 125 µm.

8.2.5 Classify porosity resulting from uncombined carbon as Type C (see Fig. 4 and Fig. 5), and examine at a magnification of 100 or 200 diameters. Report the porosity level by reference to the appropriate photomicrograph, and designate as C02, C04, C06, or C08.

8.3 If no porosity of either Type A, B, or C is detected, report it as A00, B00, or C00, as applicable.
9. Report

9.1 The report shall include the following:
9.1.1 Reference to this test method,
9.1.2 Complete identification of the test specimen,
9.1.3 Results obtained, and
9.1.4 Details of any occurrence that may have affected the result.

10. Precision and Bias

10.1 A precision and bias statement cannot be made for this test method.

11. Keywords

11.1 apparent porosity; cemented carbides; hardmetals; microstructure; powder metallurgy
FIG. 3 Type B Apparent Porosity (× 100)
FIG. 4 Uncombined Carbon (× 100)
FIG. 5 Uncombined Carbon (× 200)

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Standard Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings

This standard is issued under the fixed designation B 281; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope
1.1 This practice is intended to serve as a guide for the proper preparation of copper and its alloys for electroplating and conversion coating. This practice is also suitable for use before autocatalytic plating. Only alloys containing at least 50 mass % copper are considered within the scope of this practice.
1.2 The wide variety of methods of mechanical finishing are not considered strictly as preparation for electroplating or conversion coating and consequently are described only briefly.
1.3 Details of electroplating and subsequent treatments for applying conversion coatings are not within the scope of this practice.
1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For a specific hazard statement, see 6.5.2.

2. Referenced Documents
2.1 ASTM Standards:
B 322 Practice for Cleaning Metals Prior to Electroplating

3. Significance and Use
3.1 The proper preparation of copper and copper alloy surfaces for electroplating, conversion coating, or autocatalytic plating is often critical to the performance of the coatings.
3.2 This practice outlines procedures required to produce satisfactory coatings on surfaces of copper and copper alloy surfaces.

4. Process Chemicals
4.1 All process chemicals are of technical grade or better. Acid solutions are prepared from grade chemicals as listed in Appendix X1.
4.2 Purity of Water—High quality water is not normally required to make up and maintain the solutions utilized in this practice. If reused or recycled water from waste treatment processes or from other in-plant sources is to be used, it should be relatively free of chromium salts, oil, wetting agents, or insoluble materials. Excessively hard water can decrease the life and performance of many cleaning solutions and make parts more difficult to rinse completely.

5. General Considerations
5.1 Removal of Oxides—Oxides can be removed from as fabricated, annealed, or heat-treated alloys by abrasive methods such as tumbling, burnishing, and emery set-up wheel polishing and by chemical methods, such as deoxidizing solutions, bright dips, and cyanide dips. The choice of method is dependent on the resultant surface finish required, amount of oxide to be removed, and the end-use properties of the article finished.
5.2 Castings and Forgings—Castings and forgings requiring abrasive methods to produce a desired surface finish do not necessarily need pickling or bright dipping. If pickled, bright dipped, or deoxidized, however, castings and other porous parts should be thoroughly rinsed between operations to avoid or minimize staining or stain spots. Castings or forgings processed in solutions containing wetting agents, which are in many proprietary products or which may be added by the individual, usually require greater care in rinsing.
5.3 Stampings and Drawn Products—Stampings and drawn work follow the same rule as castings and forgings except, for economy considerations, it may be advisable to pickle or deoxidize before abrasive finishing if heavy oxides are present.
5.4 Cold-Headed and Progressive Die Products—Cold-head products and progressive die products often require relief annealing to avoid subsequent season cracking.
5.5 Screw Machine Products—Screw machine products may be readily electroplated with only mild cleaning and acid dipping as they are produced from the machines. Abrasive methods may be applied as appropriate before cleaning and acid dipping.

6. Preparation for Electroplating and Conversion Coating
6.1 Outlines of Typical Preparatory Cycles:
clean, or tumble clean,
   6.1.2 Rinse,
   6.1.3 Alkaline electroclean,
   6.1.4 Rinse,
   6.1.5 Acid dip,
   6.1.6 Rinse, and
   6.1.7 Electroplate or conversion coat in an acid solution.

Note 1—A bright dip, electropolish, or deoxidization may be added after step 6.1.4 or 6.1.2.4 followed by two agitated and running rinses prior to step 6.1.5.
Note 2—If chromium compounds are used in the bright dip or alternatives in Note 1, additional steps will be required to ensure complete chromium removal from surfaces before any plating process.
Note 3—Additional information on procedures for cleaning of copper or copper alloys prior to electroplating may be found in Practice B 322.

6.2 Precleaning—Solvent or solvent-alkali emulsion-soak cleaners can be used if the parts being electroplated can be rinsed easily and completely; otherwise, mild alkaline cleaners and vapor degreasing should be used.

6.3 Electrocleaning—to produce the chemically clean surface required for electroplating or subsequent coatings, an electrolytic cleaner may be used with the part as the anode or cathode. Anodic cleaning, particularly of brass, may cause slight tarnishing or etching if applied for a prolonged time (more than a few seconds) or at too high or too low an operating temperature. A contaminated cathodic cleaner may form a smut film on the work. The voltage at the source is usually 6 to 8 V. Separate solutions should be used for anodic and cathodic cleaning. When a part is properly cleaned, it will show a continuous liquid film upon removal from rinses after acid dip solutions.

Note 4—Proprietary cleaners which are used in accordance with the supplier’s recommendations are preferred.
Note 5—The formula of a typical electrolytic cleaner composition is given in Appendix X1.

6.4 Tumble Cleaning—Tumble cleaning is an alternative process that can be used as a substitute for precleaning or electrocleaning depending on the parts being processed. A typical tumble cleaner is listed in Appendix X1. Proprietary cleaners are available and are generally considered preferable.

6.5 Tarnish and Stain Removal, Deoxidizing, and Neutralizing:

6.5.1 Acid Dipping—After the work has been thoroughly cleaned and rinsed, it must be acidified to neutralize any residual alkali before it enters an electroplating bath. Thus, before nickel, copper, tin, chromium, and similar plating solutions, an acid dip is used. The most common dips used are sulfuric acid, 50 mL/L to 100 mL/L by volume, or hydrochloric acid, 100 mL/L to 200 mL/L by volume. Fluoboric acid, 50 mL/L to 100 mL/L by volume, can be used before fluoroborate electroplating solutions. These solutions are maintained at room temperature. Leaded copper alloys form insoluble salts in either sulfuric or hydrochloric acid, and therefore should be pickled in fluoboric acid, 20 mL/L to 50 mL/L by volume, or nitric acid, 100 mL/L to 200 mL/L by volume.

6.5.2 Cyanide Dipping—After the work has been cleaned, acid pickled, or acid dipped, and thoroughly rinsed, it is sometimes immersed in a 15 to 45 g/L sodium cyanide solution to remove slight tarnish. This step is more common if the first electroplating solution also contains cyanide. (Warning—One should provide adequate rinsing before and after any cyanide-containing solution before going into an acid solution.)

Note 6—When processing parts containing lead as an alloy constituent, such as free-machining brass, care should be taken not to allow lead to accumulate in a cyanide solution beyond 50 mg/L as Pb.

6.5.3 Deoxidizing and Bright Dipping:

6.5.3.1 Deoxidizing is usually performed to activate the surface of the part by the removal of the oxide coating. Strong oxidizing solutions such as mixtures of sulfuric acid and hydrogen peroxide are used as pickling agents. Most of these compositions are proprietary.

6.5.3.2 Bright dipping is primarily used to improve the surface luster of the work. It also serves as a deoxidizing solution. While proprietary processes are available one non-proprietary composition which can produce good surface luster (not mirror brightness) is included in Appendix X1. The bright dipped parts should be rinsed thoroughly by immersion in several water rinses with constant agitation. A mild alkaline solution may be employed to neutralize residual acids before immersion in any cyanide-containing solution. An excess of hydrochloric acid in the bright dip composition must be avoided; otherwise, a dull finish will result.

7. Striking

7.1 Copper Strike—In order to prevent peeling, a copper strike is used before silver or nickel electroplating of leaded copper alloys and work that has been soft soldered. A nickel strike (see Appendix X2) may be used in addition to the copper strike before silver electroplating. A standard cyanide copper strike may be employed (see Note 6). All soldered surfaces and the basis metal must be completely covered with copper. This requires up to 1 min at 3 to 6 V.

7.2 Silver Strike—It is always necessary to apply a silver strike to the work before it enters a silver electroplating solution. The power source should be on and the electrical circuit connection made (for “live” entry) before immersing the work in either the silver strike or silver electroplating solution.

7.3 Nickel Strike—In order to obtain adhesion on alloys containing nickel, or chromium and iron, or both, one of the nickel strike solutions described in Appendix X1 is used. Additional thicknesses of nickel for the purpose of diffusion control, etc., may be applied.

7.4 Gold Strike—It is always necessary to apply a gold strike to the work before it enters a good electroplating solution. A standard soft gold strike may be employed.

8. Keywords

8.1 activation; cleaning; copper; deoxidation; preparation; striking
X1. Process Chemicals—All process chemicals are of technical grade or better. Acid solutions are prepared from the following technical grade chemicals:

- Glycolic acid (CH₂OH COOH) 67 mass %, density 1.3 g/mL
- Sulfuric acid (H₂SO₄) 93 mass %, density 1.83 g/mL
- Hydrochloric acid (HCl) 31 mass %, density 1.16 g/mL
- Nitric acid (HNO₃) 67 mass %, density 1.40 g/mL
- Phosphoric acid (H₃PO₄) 75 mass %, density 1.5 g/mL
- Fluoboric acid (HBF₄) 48 mass %, density 1.38 g/mL

X1.2 Tumble Cleaning—A typical tumble cleaner is as follows:

<table>
<thead>
<tr>
<th>Mass %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mild soap chips or powder 12.5</td>
</tr>
<tr>
<td>Trisodium phosphate (Na₃PO₄·12H₂O) 12.5</td>
</tr>
<tr>
<td>Sodium carbonate (Na₂CO₃) 75</td>
</tr>
</tbody>
</table>

This mixture is used in a solution concentration of from 30 to 45 g/L at 80 to 90°C.

X1.3 Electrocleaning—A typical formula is as follows:

<table>
<thead>
<tr>
<th>Mass %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium carbonate (Na₂CO₃) 40 to 50</td>
</tr>
<tr>
<td>Trisodium phosphate (Na₃PO₄·12H₂O) 25 to 50</td>
</tr>
<tr>
<td>Sodium hydroxide (NaOH) 10 to 25</td>
</tr>
<tr>
<td>Surface active agent (Low foam or No Foam Type) 1</td>
</tr>
</tbody>
</table>

This mixture is used in a solution concentration of 30 to 45 g/L.

- Temperature 60 to 71°C
- Current density 1 to 3 A/dm²
- Time 1 to 3 min cathodic and 5 to 10 s anodic

X1.4 Acid Pickles:

X1.4.1 “Fire Off” or Scale Removal Dip

This dip is used to remove heavy oxide layers on small parts that can be handled in bulk. A typical example is the following mixture:

<table>
<thead>
<tr>
<th></th>
<th>Mass %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sulfuric acid (50 mL/L) to (300 mL/L)</td>
<td></td>
</tr>
<tr>
<td>Nitric acid (150 mL/L) to (600 mL/L)</td>
<td></td>
</tr>
<tr>
<td>Water</td>
<td>To balance</td>
</tr>
<tr>
<td>Temperature</td>
<td>18 to 20°C</td>
</tr>
</tbody>
</table>

Note X1.1—Lead sulfate may form on the small globules of lead present in leaded brass and can cause porosity and blistering of the subsequent electroplate. Diluted nitric acid (without sulfuric acid) has been used successfully, as has fluoboric acid, in scale removal.

X1.4.2 For copper and copper-base alloys (except beryllium copper) to remove oxides, sulfuric acid (100 mL/L to 400 mL/L) by volume is used. These solutions may be used from room temperature to 80°C. Time required may have to be 10 min or longer. In some instances the smuts developed in beryllium and tellurium copper can be easily and completely removed by a short immersion in 30 to 35 % by vol (22°Be) HCl at ambient temperature.

X1.4.3 To remove oxides from beryllium copper alloys, proprietary sulfuric acid/hydrogen peroxide mixtures have been found to be preferable to the conventional bright dip solutions described in X1.4.1 and X1.4.5. Reduced porosity of the electroplated coating has been found when these proprietary deoxidizing solutions are employed instead of bright dips.

X1.4.4 When heat treated, beryllium copper may form a beryllium oxide film. A 5-min dip in a solution of the following composition can be used to ensure complete removal of this film:

<table>
<thead>
<tr>
<th></th>
<th>Mass %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium hydroxide</td>
<td>500 g/L</td>
</tr>
<tr>
<td>Water</td>
<td>To balance</td>
</tr>
<tr>
<td>Temperature</td>
<td>190°C</td>
</tr>
</tbody>
</table>

Note X1.2—This procedure is normally used by the producers of beryllium copper alloys.

X1.4.5 Nitric-Phosphoric Acid Pickle

This pickle is also used to remove oxides and scale. The parts should be clean and dry before being placed in the solution.

<table>
<thead>
<tr>
<th></th>
<th>Mass %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitric acid</td>
<td>250 mL/L</td>
</tr>
<tr>
<td>Phosphoric acid</td>
<td>750 mL/L</td>
</tr>
</tbody>
</table>

X1.5 Electropolishing—Electropolishing is used to impart a bright smooth finish to the part. It is accomplished by making the work anodic at 3 to 6 A/dm² in a solution as follows:

<table>
<thead>
<tr>
<th></th>
<th>Mass %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Glycolic acid</td>
<td>350 mL/L</td>
</tr>
<tr>
<td>Phosphoric acid</td>
<td>550 mL/L</td>
</tr>
<tr>
<td>Sulfuric acid</td>
<td>190 mL/L</td>
</tr>
<tr>
<td>Water</td>
<td>Balance</td>
</tr>
</tbody>
</table>

X1.6 Bright Dip Solution—A formula for a bright dip is given as follows:

<table>
<thead>
<tr>
<th></th>
<th>Mass %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sulfuric acid</td>
<td>600 mL/L to 750 mL/L</td>
</tr>
<tr>
<td>Nitric acid</td>
<td>200 mL/L to 350 mL/L</td>
</tr>
<tr>
<td>Hydrochloric acid</td>
<td>1 mL/L</td>
</tr>
<tr>
<td>Water</td>
<td>To 1 L</td>
</tr>
</tbody>
</table>

Agitate the work constantly while immersed for 5 to 105 s. A mild alkaline solution can be used after rinsing the bright dip solution from the part to ensure neutralization if a cyanide plating solution follows.
X2. NICKEL STRIKE SOLUTIONS

X2.1 Woods Nickel Strike—A low pH nickel strike of the following composition may be substituted for a copper strike, especially prior to silver electroplating:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel chloride (NiCl₂·6H₂O)</td>
<td>240 g/L</td>
</tr>
<tr>
<td>Hydrochloric acid</td>
<td>120 g/L</td>
</tr>
<tr>
<td>Water</td>
<td>balance</td>
</tr>
<tr>
<td>Cathodic current density</td>
<td>5.4 to 10.8 A/dm²</td>
</tr>
<tr>
<td>Temperature</td>
<td>18 to 25°C</td>
</tr>
<tr>
<td>Time</td>
<td>2 min</td>
</tr>
</tbody>
</table>

pH (lowered with sulfamic acid) 1.5
Cathode current density 2–10 A/dm²
Temperature 18 to 25°C

X2.2 Low pH Sulfamate Nickel Strike—This solution has been useful when plating on assemblies of mixed alloy components where copper or copper alloys have been joined to high-nickel steels or other passive metals.

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel Sulfamate (Ni(SO₃NH₂)₂)</td>
<td>320 g/L</td>
</tr>
<tr>
<td>Nickel as metal</td>
<td>76 g/L</td>
</tr>
<tr>
<td>Boric acid (H₃BO₃)</td>
<td>30 g/L</td>
</tr>
<tr>
<td>Water</td>
<td>balance</td>
</tr>
<tr>
<td>pH (lowered with sulfamic acid)</td>
<td>1.5</td>
</tr>
<tr>
<td>Cathode current density</td>
<td>2–10 A/dm²</td>
</tr>
<tr>
<td>Temperature</td>
<td>18 to 25°C</td>
</tr>
<tr>
<td>Time</td>
<td>2 min</td>
</tr>
</tbody>
</table>

X2.3 Glycolate Nickel Strike—Glycolate nickel strike is used on complex parts to increase uniformity and coverage in low current density areas.

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel Acetate (Ni(CH₃COO)₂)·4H₂O</td>
<td>65 gm/L</td>
</tr>
<tr>
<td>Boric Acid</td>
<td>45 gm/L</td>
</tr>
<tr>
<td>Hydroxyacetic Acid (Glycolic Acid)</td>
<td>60 mL/L</td>
</tr>
<tr>
<td>Sacharin (stress reducer)</td>
<td>1.5 gm/L</td>
</tr>
<tr>
<td>pH</td>
<td>6.2</td>
</tr>
<tr>
<td>Temperature</td>
<td>Ambient</td>
</tr>
<tr>
<td>Current Density</td>
<td>2.7 A/dm²</td>
</tr>
<tr>
<td>Anodes</td>
<td>Nickel (inert)</td>
</tr>
<tr>
<td>Time</td>
<td>5 min</td>
</tr>
</tbody>
</table>

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Standard Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings

This standard is issued under the fixed designation B 281; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This practice is intended to serve as a guide for the proper preparation of copper and its alloys for electroplating and conversion plating. This practice is also suitable for use before autocatalytic plating. Only alloys containing at least 50 mass % copper are considered within the scope of this practice.

1.2 The wide variety of methods of mechanical finishing are not considered strictly as preparation for electroplating or conversion coating and consequently are described only briefly.

1.3 Details of electroplating and subsequent treatments for applying conversion coatings are not within the scope of this practice.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For a specific hazard statement, see Note 6.

2. Referenced Documents

2.1 ASTM Standards:

B 322 Practice for Cleaning Metals Prior to Electroplating

3. Significance and Use

3.1 The proper preparation of copper and copper alloy surfaces for electroplating, conversion coating, or autocatalytic plating is often critical to the performance of the coatings.

3.2 This practice outlines procedures required to produce satisfactory coatings on surfaces of copper and copper alloy surfaces.

4. Process Chemicals

4.1 All process chemicals are of technical grade or better. Acid solutions are prepared from grade chemicals as listed in Appendix X1.

4.2 Purity of Water—High quality water is not normally required to make up and maintain the solutions utilized in this practice. If re-used or recycled water from waste treatment processes or from other in-plant sources is to be used, it should be relatively free of chromium salts, oil, wetting agents, or insoluble materials. Excessively hard water can decrease the life and performance of many cleaning solutions and make parts more difficult to rinse completely.

5. General Considerations

5.1 Removal of Oxides—Oxides can be removed from as fabricated, annealed, or heat-treated alloys by abrasive methods such as tumbling, burnishing, and emery set-up wheel polishing and by chemical methods, such as deoxidizing solutions, bright dips, and cyanide dips. The choice of method is dependent on the resultant surface finish required, amount of oxide to be removed, and the end-use properties of the article finished.

5.2 Castings and Forgings—Castings and forgings requiring abrasive methods to produce a desired surface finish do not necessarily need pickling or bright dipping. If pickled, bright dipped, or deoxidized, however, castings and other porous parts should be thoroughly rinsed between operations to avoid or minimize staining or stain spots. Castings or forgings processed in solutions containing wetting agents, which are in many proprietary products or which may be added by the individual, usually require greater care in rinsing.

5.3 Stampings and Drawn Products—Stampings and drawn work follow the same rule as castings and forgings except, for economy considerations, it may be advisable to pickle or deoxidize before abrasive finishing if heavy oxides are present.

5.4 Cold-Headed and Progressive Die Products—Cold-headed products and progressive die products often require relief annealing to avoid subsequent season cracking.

5.5 Screw Machine Products—Screw machine products may be readily electroplated with only mild cleaning and acid dipping as they are produced from the machines. Abrasive methods may be applied as appropriate before cleaning and acid dipping.

6. Preparation for Electroplating and Conversion Coating

6.1 Outlines of Typical Preparatory Cycles:

6.1.1 Vapor degrease or alkaline clean or emulsion soak
clean, or tumble clean,
6.1.2 Rinse,
6.1.3 Alkaline electroclean,
6.1.4 Rinse,
6.1.5 Acid dip,
6.1.6 Rinse, and
6.1.7 Electroplate or conversion coat in an acid solution.

NOTE 1—A bright dip, electropolish, or deoxidization may be added after step 6.1.4 or 6.1.2.4 followed by two agitated and running rinses prior to step 6.1.5.

NOTE 2—If chromium compounds are used in the bright dip or alternatives in Note 1, additional steps will be required to ensure complete chromium removal from surfaces before any plating process.

NOTE 3—Additional information on procedures for cleaning of copper or copper alloys prior to electroplating may be found in Practice B 322.

6.2 Precleaning—Solvent or solvent-alkali emulsion-soak cleaners can be used if the parts being electroplated can be rinsed easily and completely; otherwise, mild alkaline cleaners and vapor degreasing should be used.

6.3 Electrocleaning—To produce the chemically clean surface required for electroplating or subsequent coatings, an electrolytic cleaner may be used with the part as the anode or cathode. Anodic cleaning, particularly of brass, may cause slight tarnishing or etching if applied for a prolonged time (more than a few seconds) or at too high or too low an operating temperature. A contaminated cathodic cleaner may form a smut film on the work. The voltage at the source is usually 6 to 8 V. Separate solutions should be used for anodic and cathodic cleaning. When a part is properly cleaned, it will show a continuous liquid film upon removal from rinses after acid dip solutions.

NOTE 4—Proprietary cleaners which are used in accordance with the supplier’s recommendations are preferred.

NOTE 5—The formula of a typical electrolytic cleaner composition is given in Appendix X1.

6.4 Tumble Cleaning—Tumble cleaning is an alternative process that can be used as a substitute for precleaning or electrocleaning depending on the parts being processed. A typical tumble cleaner is listed in Appendix X1. Proprietary cleaners are available and are generally considered preferable.

6.5 Tarnish and Stain Removal, Deoxidizing, and Neutralizing:

6.5.1 Acid Dipping—After the work has been thoroughly cleaned and rinsed, it must be acidified to neutralize any residual alkali before it enters an electroplating bath. Thus, before nickel, copper, tin, chromium, and similar plating solutions, an acid dip is used. The most common dips used are sulfuric acid, 50 mL/L to 100 mL/L by volume, or hydrochloric acid, 100 mL/L to 200 mL/L by volume. Fluoboric acid, 50 mL/L to 100 mL/L by volume, can be used before fluoborate electroplating solutions. These solutions are maintained at room temperature. Leaded copper alloys form insoluble salts in either sulfuric or hydrochloric acid, and therefore should be pickled in fluoboric acid, 20 mL/L to 50 mL/L by volume, or nitric acid, 100 mL/L to 200 mL/L by volume.

6.5.2 Cyanide Dipping—After the work has been cleaned, acid pickled, or acid dipped, and thoroughly rinsed, it is sometimes immersed in a 15 to 45 g/L sodium cyanide solution to remove slight tarnish. This step is more common if the first electroplating solution also contains cyanide.

NOTE 6—Precaution: One should provide adequate rinsing before and after any cyanide-containing solution before going into an acid solution.

NOTE 7—When processing parts containing lead as an alloy constituent, such as free-machining brass, care should be taken not to allow lead to accumulate in a cyanide solution beyond 50 mg/L as Pb.

6.5.3 Deoxidizing and Bright Dipping:

6.5.3.1 Deoxidizing is usually performed to activate the surface of the part by the removal of the oxide coating. Strong oxidizing solutions such as mixtures of sulfuric acid and hydrogen peroxide are used as pickling agents. Most of these compositions are proprietary.

6.5.3.2 Bright dipping is primarily used to improve the surface luster of the work. It also serves as a deoxidizing solution. While proprietary processes are available one nonproprietary composition which can produce good surface luster (not mirror brightness) is included in Appendix X1. The bright dipped parts should be rinsed thoroughly by immersion in several water rinses with constant agitation. A mild alkaline solution may be employed to neutralize residual acids before immersion in any cyanide-containing solution. An excess of hydrochloric acid in the bright dip composition must be avoided; otherwise, a dull finish will result.

7. Striking

7.1 Copper Strike—In order to prevent peeling, a copper strike is used before silver or nickel electroplating of leaded copper alloys and work that has been soft soldered. A nickel strike (see Appendix X2) may be used in addition to the copper strike before silver electroplating. A standard cyanide copper strike may be employed (see Note 7). All soldered surfaces and the basis metal must be completely covered with copper. This requires up to 1 min at 3 to 6 V.

7.2 Silver Strike—It is always necessary to apply a silver strike to the work before it enters a silver electroplating solution. The power source should be on and the electrical circuit connection made (for “live” entry) before immersing the work in either the silver strike or silver electroplating solution.

7.3 Nickel Strike—In order to obtain adhesion on alloys containing nickel, or chromium and iron, or both, one of the nickel strike solutions described in Appendix X1 is used. Additional thicknesses of nickel for the purpose of diffusion control, etc., may be applied.

7.4 Gold Strike—It is always necessary to apply a gold strike to the work before it enters a good electroplating solution. A standard soft gold strike may be employed.

8. Keywords

8.1 activation; cleaning; copper; deoxidation; preparation; striking
X1. CLEANING AND PICKLING SOLUTIONS

X1.1 Process Chemicals—All process chemicals are of technical grade or better. Acid solutions are prepared from the following technical grade chemicals:

- Glycolic acid (CH\textsubscript{2}OH COOH) 67 mass %, density 1.3 g/mL
- Sulfuric acid (H\textsubscript{2}SO\textsubscript{4}) 93 mass %, density 1.83 g/mL
- Hydrochloric acid (HCl) 31 mass %, density 1.16 g/mL
- Nitric acid (HNO\textsubscript{3}) 67 mass %, density 1.40 g/mL
- Phosphoric acid (H\textsubscript{3}PO\textsubscript{4}) 75 mass %, density 1.5 g/mL
- Fluoboric acid (HBF\textsubscript{4}) 48 mass %, density 1.38 g/mL

X1.2 Tumble Cleaning—A typical tumble cleaner is as follows:

<table>
<thead>
<tr>
<th>Mass %</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Mild soap chips or powder</td>
<td>12.5</td>
</tr>
<tr>
<td>Trisodium phosphate (Na\textsubscript{3}PO\textsubscript{4}·12H\textsubscript{2}O)</td>
<td>12.5</td>
</tr>
<tr>
<td>Sodium carbonate (Na\textsubscript{2}CO\textsubscript{3})</td>
<td>75</td>
</tr>
</tbody>
</table>

This mixture is used in a solution concentration of from 30 to 45 g/L at 80 to 90°C.

X1.3 Electrocleaning—A typical formula is as follows:

<table>
<thead>
<tr>
<th>Mass %</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium carbonate (Na\textsubscript{2}CO\textsubscript{3})</td>
<td>40 to 50</td>
</tr>
<tr>
<td>Trisodium phosphate (Na\textsubscript{3}PO\textsubscript{4}·12H\textsubscript{2}O)</td>
<td>25 to 50</td>
</tr>
<tr>
<td>Sodium hydroxide (NaOH)</td>
<td>10 to 25</td>
</tr>
<tr>
<td>Surface active agent (Low foam or No Foam Type)</td>
<td>1</td>
</tr>
</tbody>
</table>

Total of individual ingredients adjusted to give 100 %

This mixture is used in a solution concentration of 30 to 45 g/L.

X1.4 Acid Pickles:

X1.4.1 “Fire Off” or Scale Removal Dip

This dip is used to remove heavy oxide layers on small parts that can be handled in bulk. A typical example is the following mixture:

<table>
<thead>
<tr>
<th>Mass %</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Sulfuric acid</td>
<td>(50 mL/L) to (300 mL/L)</td>
</tr>
<tr>
<td>Nitric acid</td>
<td>(150 mL/L) to (600 mL/L)</td>
</tr>
<tr>
<td>Water</td>
<td>remainder</td>
</tr>
<tr>
<td>Temperature</td>
<td>18 to 20°C</td>
</tr>
</tbody>
</table>

Note: X1.1—Lead sulfate may form on the small globules of lead present in leaded brass and can cause porosity and blistering of the subsequent electroplate. Diluted nitric acid (without sulfuric acid) has been used successfully, as has fluoboric acid, in scale removal.

X1.4.2 For copper and copper-base alloys (except beryllium copper) to remove oxides, sulfuric acid (100 mL/L to 400 mL/L) by volume is used. These solutions may be used from room temperature to 80°C. Time required may have to be 10 min or longer. In some instances the smuts developed in beryllium and tellurium copper can be easily and completely removed by a short immersion in 30 to 35 % by vol (22°Be) HCl at ambient temperature.

X1.4.3 To remove oxides from beryllium copper alloys, proprietary sulfuric acid/hydrogen peroxide mixtures have been found to be preferable to the conventional bright dip solutions described in X1.4.1 and X1.4.5. Reduced porosity of the electroplated coating has been found when these proprietary deoxidizing solutions are employed instead of bright dips.

X1.4.4 When heat treated, beryllium copper may form a beryllium oxide film. A dip in a solution of the following composition can be used to ensure complete removal of this film:

<table>
<thead>
<tr>
<th>Mass %</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium hydroxide</td>
<td>500 g/L</td>
</tr>
<tr>
<td>Water</td>
<td>To balance</td>
</tr>
<tr>
<td>Temperature</td>
<td>130°C</td>
</tr>
</tbody>
</table>

Note: X1.2—This procedure is normally used by the producers of beryllium copper alloys.

X1.4.5 Nitric-Phosphoric Acid Pickle

This pickle is also used to remove oxides and scale. The parts should be clean and dry before being placed in the solution.

X1.5 Electropolishing—Electropolishing is used to impart a bright smooth finish to the part. It is accomplished by making the work anodic at 3 to 6 A/dm\textsuperscript{2} in a solution as follows:

<table>
<thead>
<tr>
<th>Mass %</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Glycolic acid</td>
<td>350 mL/L</td>
</tr>
<tr>
<td>Phosphoric acid</td>
<td>550 mL/L</td>
</tr>
<tr>
<td>Sulfuric acid</td>
<td>190 mL/L</td>
</tr>
<tr>
<td>Water</td>
<td>Balance</td>
</tr>
</tbody>
</table>

X1.6 Bright Dip Solution—A formula for a bright dip is given as follows:

<table>
<thead>
<tr>
<th>Mass %</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Sulfuric acid</td>
<td>600 mL/L to 750 mL/L</td>
</tr>
<tr>
<td>Nitric acid</td>
<td>200 mL/L to 350 mL/L</td>
</tr>
<tr>
<td>Hydrochloric acid</td>
<td>1 mL/L</td>
</tr>
<tr>
<td>Water</td>
<td>To 1 L</td>
</tr>
</tbody>
</table>

Agitate the work constantly while immersed for from 5 to 105 s. A mild alkaline solution can be used after rinsing the bright dip solution from the part to ensure neutralization if a cyanide plating solution follows.
X2. NICKEL STRIKE SOLUTIONS

X2.1 Woods Nickel Strike—A low pH nickel strike of the following composition may be substituted for a copper strike, especially prior to silver electroplating:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel chloride (NiCl₂·6H₂O)</td>
<td>240 g/L</td>
</tr>
<tr>
<td>Hydrochloric acid</td>
<td>120 g/L</td>
</tr>
<tr>
<td>Water</td>
<td>balance</td>
</tr>
<tr>
<td>Cathodic current density</td>
<td>5.4 to 10.8 A/dm²</td>
</tr>
<tr>
<td>Temperature</td>
<td>18 to 25°C</td>
</tr>
</tbody>
</table>

Cathodic current density 5.4 to 10.8 A/dm²
Time 2 min
Temperature 18 to 25°C

X2.2 Low pH Sulfamate Nickel Strike—This solution has been useful when plating on assemblies of mixed alloy components where copper or copper alloys have been joined to high-nickel steels or other passive metals.

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel Sulfamate (Ni(SO₃NH₂)₂)</td>
<td>320 g/L</td>
</tr>
<tr>
<td>Nickel as metal</td>
<td>76 g/L</td>
</tr>
<tr>
<td>Boric acid (H3 BO₃)</td>
<td>30 g/L</td>
</tr>
<tr>
<td>Water</td>
<td>balance</td>
</tr>
<tr>
<td>pH (lowered with sulfamic acid)</td>
<td>1.5</td>
</tr>
<tr>
<td>Cathode current density</td>
<td>2–10 A/dm²</td>
</tr>
<tr>
<td>Time</td>
<td>2 min</td>
</tr>
<tr>
<td>Temperature</td>
<td>18 to 25°C</td>
</tr>
</tbody>
</table>

Cathode current density 2–10 A/dm²
Time 2 min
Temperature 18 to 25°C

X2.3 Glycolate Nickel Strike—Glycolate nickel strike is used on complex parts to increase uniformity and coverage in low current density areas.

<table>
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<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel Acetate (Ni(CH₃COO)₂)₄H₂O</td>
<td>65 gm/L</td>
</tr>
<tr>
<td>Boric Acid</td>
<td>45 gm/L</td>
</tr>
<tr>
<td>Hydroxyacetic Acid (Glycolic Acid)</td>
<td>60 mL/L</td>
</tr>
<tr>
<td>Sacharin (stress reducer)</td>
<td>1.5 gm/L</td>
</tr>
<tr>
<td>pH</td>
<td>6.2</td>
</tr>
<tr>
<td>Temperature</td>
<td>Ambient</td>
</tr>
<tr>
<td>Current Density</td>
<td>2.7 A/dm²</td>
</tr>
<tr>
<td>Anodes</td>
<td>Nickel (inert)</td>
</tr>
<tr>
<td>Time</td>
<td>5 min</td>
</tr>
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Standard Test Method for
Hardness Testing of Cemented Carbides

This standard is issued under the fixed designation B 294; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the hardness testing of cemented carbides by use of the Rockwell hardness tester with the Rockwell A scale (diamond indenter and 588.4 N (60 kgf) load) in the range of Rockwell A80 and above. Also covered are the procedures for the testing and selection of diamond indenters, the management and traceability of the four levels of standard test blocks, the acquisition of secondary standard test blocks, and the making and calibration of working standard test blocks.

1.2 The Rockwell hardness tester is a convenient and reliable means of measuring the hardness of cemented carbides. A hardness value is obtained easily, but it is subject to considerable error unless certain precautions are observed.

1.3 Test Methods E 18 shall be followed except where otherwise indicated in this test method.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials
E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

2.2 ISO Standard:
3738-2 Hardmetals—Rockwell hardness test (Scale A)—Part 2, Preparation and calibration of standard test blocks

3. Significance and Use

3.1 Rockwell hardness is one of the more important properties used to evaluate cemented carbides. For compositional groups of cemented carbides, hardness is an indication of wear resistance and toughness. Lower hardness grades usually indicate less wear resistance but greater toughness. For a specific grade of cemented carbide, hardness is an indication of the metallurgical quality of the material. In no case is hardness the only property to be considered in evaluating cemented carbides.

4. Apparatus

4.1 Scale—All hardness tests shall be made on the regular (as opposed to superficial) Rockwell tester, using a 588.4 N (60 kgf) load (Rockwell A scale). A hardness value is obtained easily, but it is subject to considerable error unless certain precautions are observed.

4.2 Effect of Vibration—The Rockwell hardness tester should be located in a vibration-free area in order to avoid erroneous results. If this is not possible, the tester shall be mounted so as to minimize vibrations, since vibrations tend to cause erratic readings.

4.3 Indenter—The standard indenter shall be selected, in accordance with the Annex to this test method, from diamond cone indenters specified for Rockwell A scale use and in conformance with Test Methods E 18.

4.3.1 The indenter, and an indentation made with it, in hardened steel or cemented carbide should be examined optically at approximately 50-diameter magnification for defects, conformance to shape, and mounting of the diamond. Examination should be made when selecting an indenter, occasionally during use, and whenever some event may be suspected of having damaged the diamond or its mounting.

4.4 Anvils—Select an anvil suitable for the specimen to be tested. The shoulder of the screw and the mating surface of the anvil should be clean. Seat the anvil securely. For the best accuracy, flat test pieces should be tested on a flat anvil of approximately 6-mm (1⁄4-in.) diameter. The bearing surface of this anvil, with a Rockwell C hardness of at least 60, shall be polished smooth and be free of pits and heavy scratches. The test piece should be supported suitably, with the test surface perpendicular to the line of travel of the indenter. Dust, dirt, grease, or scale should not be allowed to accumulate on any part of the apparatus, as this will affect the results.

4.5 Test Blocks—Secondary standard test blocks or working standard test blocks that have been prepared and calibrated in
acCORDANCE WITH THE ANNEX TO THIS TEST METHOD SHALL BE USED.

5. Test Specimens

5.1 Size of Specimens—A minimum thickness of 1.6 mm (1/6 in.) is recommended. With thinner specimens, breakage may occur, resulting in damage to the anvil, the indenter, or both. Specimens that have enough overhang to cause imbalance shall be supported properly. The 6-mm (1/4-in.) anvil will support flat test specimens up to approximately 113 g (1/4 lb) and will also support the standard test blocks recommended previously.

5.2 Preparation of Test Specimens—The finish of the test surface is of major importance. The surface to be tested should be prepared to obtain a roughness of Ra ≤0.2 µm (8 µin.) A coarser finish will provide a wider range of readings. Preparation shall be conducted in such a way that alteration of the surface due to heat or cold-working is minimized. A 220-grit medium hardness resinoid bond diamond wheel, downfed 0.01 mm (0.0005 in.) per pass with abundant flow of coolant, should provide the desired surface. The thickness of the layer removed from an as-sintered surface to be tested shall be not less than 0.2 mm (0.008 in.).

5.3 The surfaces of the test specimen shall be flat and parallel within one part per hundred parts in general practice, but within one part per thousand parts when critical comparisons are being made. The surface in contact with the anvil shall be free of any irregularity (for example, a previous hardness indentation). Taper that results in the test surface not being normal to the axis of the indenter, or irregularity that causes instability during the test, will result in error.

5.4 When determining the hardness of a test specimen with a curved surface, the radius of curvature shall not be less than 15 mm (1/2 in.). If less, then a flat surface at least 3-mm (1/8-in.) wide shall be prepared on which to conduct the test, and there shall be an opposite flat surface such that the specimen conforms to the requirements of 5.2 and 5.3. If the test surface is curved or the opposite surface must be supported in a V-anvil, the repeatability and reproducibility limits of 8.2 and 8.3 may not apply.

5.5 Preparation of Mounted Carbides—Remove mounted carbides from the steel body by heating or some other convenient method. All braze metal or other bond material shall be removed from both the test surface and the opposite face. The specimen should then be prepared as described in 5.1 through 5.4.

6. Procedure

6.1 Procedures that are not described in this test method shall conform to those of Test Methods E 18.

6.2 Disregard the first two readings after an indenter has been newly mounted.

6.3 Limit the speed of applying the major load so that the movement of the weights is completed in 4 to 6 s, with no test piece on the testing equipment and with the machine set to apply a major load of 60 Kg. Verification should be by direct observation of the weight motion, if visible.

6.4 Do not permit the time of maintaining the major load after the motion of the needle or the changing of the digital readout has ceased to exceed 2 s. Removal of the major load should be gradual by operating lever in manual machines or by motor in automatic machines, and should not exceed two additional seconds. On manual machines, abrupt actuation of the major load trip lever may affect the hardness value obtained. Abrupt actuation of the major load removal lever will significantly affect the hardness value obtained.

6.5 The Rockwell A hardness value is read after the major load has been removed and while the minor load is still applied.

6.6 The distance between the centers of any two adjacent indentations, and the distance between the center of any indentation and the edge of a test specimen, shall be at least 1.5 mm (0.06 in.).

6.7 Hardness should be read or estimated to the nearest 0.1 HRA. Calculations should be carried to two decimal places.

6.8 Make two trial determinations of the hardness of the test specimen. This action also reassures that the indenter is seated properly.

6.8.1 Select the standard test block having a value closest to the trial hardness of the test specimen. Determine the Rockwell A hardness at three points on the block.

6.8.2 If the arithmetic mean of the three determinations differs from the certified hardness value of the standard test block by more than ±0.5 HRA, check the diamond indenter and the testing equipment, and eliminate the cause of the error. Repeat the determinations.

6.8.3 If the arithmetic mean of the three determinations differs from the certified hardness value of the standard test block by ±0.5 HRA or less, record the difference, giving due regard to the algebraic sign. This difference will be used to correct the arithmetic mean of the hardness of the test specimens.

6.8.4 Determine the Rockwell A hardness of the test specimen, with determinations at three or more locations chosen at random, or as dictated by the purpose of the test.

6.8.5 Calculate the arithmetic mean of the hardness determinations. Apply the correction determined as in 6.8.3, giving due regard to the algebraic sign.

6.8.6 Report the corrected arithmetic mean of the hardness determinations, rounded in accordance with Practice E 295 to the nearest 0.1 HRA.

7. Report

7.1 Report the following information:

7.1.1 All details necessary for identification of the test specimen,

7.1.2 The corrected mean hardness,

7.1.3 The range of hardness determinations,

7.1.4 The number of hardness determinations,

7.1.5 The smallest division of readout or graduation of the hardness test machine and whether it is digital or analog,

7.1.6 The identification and original source of calibration for the standard test blocks used.

5 When the second decimal place is less than 0.05, leave the first decimal place unchanged. When the second decimal place is more than 0.05, increase the first decimal place by 0.1. When the second decimal place is exactly 5 and the first decimal place is odd, increase the first decimal by 0.1. If the first decimal place is even, leave it unchanged.
7.1.7 A reference to this test method, and
7.1.8 Details of any deviations from this test method, of optional procedures used, and of any conditions and occurrences that may have affected the results.

8. Precision and Bias

8.1 The following statements regarding the repeatability and reproducibility of hardness (HRA) measurements of cemented carbide test specimens shall apply only within the hardness range established for the indenter in accordance with A1.8.2 or A1.8.3. See Table A1.1.

8.2 The repeatability limit ($r$) is 0.3 HRA. On the basis of test error alone, the difference in absolute value of two test results obtained in the same laboratory on the same test specimen will be expected to exceed 0.3 HRA only approximately 5% of the time. The repeatability standard deviation ($S_r$) is 0.1 HRA.

8.3 The reproducibility limit ($R$) between or among laboratories is 0.4 HRA when each has calibrated its machine, indenter, and operator system with a standard test block that has itself been calibrated to the same superior test block used to calibrate the test blocks of the other laboratories. On the basis of test error alone, the difference in absolute value of the test results obtained in different laboratories on the same test specimen will be expected to exceed 0.4 HRA only approximately 5% of the time. The reproducibility standard deviation ($S_R$) is 0.14 HRA.

8.4 Neither the data of the interlaboratory study nor theoretical considerations suggest a bias in this test procedure.

8.5 If the test specimens are of a hardness substantially outside the hardness ranges of the standard test blocks on which the indenter has been performance tested, and if interlaboratory reproducibility is critical, the same indenter and standard test blocks should be used by each laboratory.

9. Keywords

9.1 cemented carbides; hardness; indenters; Rockwell hardness test; Scale A; test blocks

ANNEX

(Mandatory Information)

A1. PREPARATION, CALIBRATION, AND CONTROL OF STANDARD TEST BLOCKS AND SELECTION OF SCALE A INDENTERS USED IN THE PERFORMANCE OF THE PROCEDURES OF THIS TEST METHOD

A1.1 Scope and Field of Application—This Annex specifies the control of master, primary, secondary, and working standard test blocks. It specifies the preparation and calibration of primary, secondary, and working standard test blocks. It also specifies the procedure for selecting indenters having the required precision from standard Scale A indenters. Both test blocks and indenters complying with this Annex are required for Rockwell hardness testing of cemented carbides by the procedures of this test method.

A1.2 Hierarchy and Availability of Standard Test Blocks:

A1.2.1 Secondary standard test blocks, and the calibration or recalibration services for secondary standard test blocks, are available from the authorized calibrating agency. To provide traceability to the master standard test blocks, the Cemented Carbide Producers Association (CCPA) has released Set 2, master standard test blocks, to the authorized calibrating agency. Both test blocks and indenters complying with this Annex are required for Rockwell hardness testing of cemented carbides by the procedures of this test method.

A1.3 Master Standard Test Blocks:

A1.3.1 Of three sets of five master standard test blocks, Set 1 is retained by the CCPA. Set 2 has been released by the CCPA to Wilson Instruments, so that Wilson Instruments may serve as the calibrating agency for secondary standard test blocks traceable through primary standard test blocks to the set of master standard test blocks. Set 3 is retained by the Secretariat of ISO/TC 119.

A1.3.2 The sets of master standard test blocks retained by the CCPA and ISO/TC 119 shall be kept as permanent standards and shall be used only when calibration of a new master standard is required.

A1.3.3 Blocks retained by ISO/TC 119 shall be retained as unused international standards. Only in the event of destruction of some or all of Set 1 retained by the CCPA, and then only by majority vote of the members of ISO/TC 119/SC4, shall they be released to be used for the calibration of new master standard test blocks. They shall then be returned to ISO/TC 119.

A1.3.4 The markings and internationally agreed upon hardnesses and standard deviations of three sets of the five master set blocks are given in Table A1.2, Table A1.3, and Table A1.4.

A1.3.5 The blocks shall not be reground, and, to ensure that this has not been done, their thickness is to be measured and recorded at the time of the calibration.

A1.4 Primary Standard Test Blocks:

<table>
<thead>
<tr>
<th>Table A1.1 Control of Test Blocks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Level</td>
</tr>
<tr>
<td>--------</td>
</tr>
<tr>
<td>1</td>
</tr>
<tr>
<td>2</td>
</tr>
<tr>
<td>3</td>
</tr>
<tr>
<td>4</td>
</tr>
</tbody>
</table>

7 The statements of repeatability and reproducibility in this section are based on an interlaboratory study conducted by the Cemented Carbide Producers Association.

8 The statements of repeatability and reproducibility in this section are based on an interlaboratory study conducted by the Cemented Carbide Producers Association.

7 The Cemented Carbide Producers Association has authorized Wilson Instruments, 6 Emma St., Binghamton, NY 13905, to be the calibrating agency.
TABLE A1.2 Set 1 Retained by CCPA

<table>
<thead>
<tr>
<th>Marking</th>
<th>Hardness</th>
<th>Standard Deviation</th>
<th>Thickness, in.</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 Series I</td>
<td>85.70</td>
<td>0.07</td>
<td>0.260</td>
</tr>
<tr>
<td>6 Series II</td>
<td>88.64</td>
<td>0.07</td>
<td>0.270</td>
</tr>
<tr>
<td>8 Series III</td>
<td>91.08</td>
<td>0.06</td>
<td>0.280</td>
</tr>
<tr>
<td>2 Series IV</td>
<td>91.59</td>
<td>0.04</td>
<td>0.304</td>
</tr>
<tr>
<td>8 Series V</td>
<td>92.80</td>
<td>0.05</td>
<td>0.280</td>
</tr>
</tbody>
</table>

TABLE A1.3 Set 2 Released to Calibrating Agency

<table>
<thead>
<tr>
<th>Marking</th>
<th>Hardness</th>
<th>Standard Deviation</th>
<th>Thickness, in.</th>
</tr>
</thead>
<tbody>
<tr>
<td>4 Series I</td>
<td>85.68</td>
<td>0.08</td>
<td>0.260</td>
</tr>
<tr>
<td>10 Series II</td>
<td>88.63</td>
<td>0.08</td>
<td>0.270</td>
</tr>
<tr>
<td>6 Series III</td>
<td>91.06</td>
<td>0.06</td>
<td>0.280</td>
</tr>
<tr>
<td>12 Series IV</td>
<td>91.62</td>
<td>0.04</td>
<td>0.304</td>
</tr>
<tr>
<td>5 Series V</td>
<td>92.81</td>
<td>0.08</td>
<td>0.280</td>
</tr>
</tbody>
</table>

Note 1—The standard deviation is calculated on 40 indentations made in such a way as to cover the whole test surface.

TABLE A1.4 Set 3 Retained by the Secretariat of ISO/TC 119

<table>
<thead>
<tr>
<th>Marking</th>
<th>Hardness</th>
<th>Standard Deviation</th>
<th>Thickness, in.</th>
</tr>
</thead>
<tbody>
<tr>
<td>6 Series I</td>
<td>85.69</td>
<td>0.07</td>
<td>0.260</td>
</tr>
<tr>
<td>5 Series II</td>
<td>88.56</td>
<td>0.07</td>
<td>0.270</td>
</tr>
<tr>
<td>1 Series III</td>
<td>91.06</td>
<td>0.06</td>
<td>0.280</td>
</tr>
<tr>
<td>14 Series IV</td>
<td>91.60</td>
<td>0.06</td>
<td>0.304</td>
</tr>
<tr>
<td>6 Series V</td>
<td>92.79</td>
<td>0.08</td>
<td>0.280</td>
</tr>
</tbody>
</table>

A1.4.1 Primary standard test blocks shall be prepared and calibrated in compliance with ISO 3738-2.
A1.4.2 Primary standard test blocks shall be reserved for use by the calibrating agency to calibrate secondary standard test blocks.

A1.5 Secondary Standard Test Blocks:
A1.5.1 Secondary standard test blocks shall be prepared and calibrated in compliance with ISO 3738-2.
A1.5.2 Secondary standard test blocks shall be used to calibrate working standard test blocks, or they may be used to verify Rockwell hardness testing machines (Scale A) and indenters for testing hard metals when a higher degree of confidence is desired than may be possible with working standard test blocks.
A1.5.3 Secondary standard test blocks shall have a nominal diameter of 45 mm and a nominal thickness of 8 mm. The bottom face shall be chamfered 0.8 mm by 45°.

A1.6 Preparation, Calibration, and Marking of Working Standard Test Blocks:
A1.6.1 Working standard test blocks may be prepared by the user.
A1.6.2 Working standard test blocks shall be prepared and calibrated against secondary standard test blocks. They should be used for routine hardness testing, so that the test surfaces of the secondary hardness test blocks are preserved for test block calibration and other critical hardness testing.
A1.6.3 Working standard test blocks may be prepared in sets of from two to five (depending on the hardness ranges of test pieces to be measured), so as to have some or all of the following nominal hardnesses: 93, 92, 91, 88.5, and 85.5 HRA.
A1.6.4 Each block shall be marked permanently on the periphery with the letter "W" and an appropriate code such that it can be related unmistakably to a record of its most recent calibration.
A1.6.5 Working standard test blocks shall conform to the following conditions: all working standard test blocks shall comprise hardmetals composed substantially of tungsten carbide and cobalt without other carbides or with less than a total of 1% (m/m) of other carbides (for example, of titanium, tantalum, and niobium); the composition and structure shall be chosen to provide the desired hardness; and free carbon and etaphase shall be absent.
A1.6.6 Working standard test blocks should be not more than 45-mm (1.75-in.) diameter and not less than 5-mm (0.2-in.) thickness. The bottom face shall be chamfered 0.8 mm (V/2 in.) by 45°.
A1.6.6.1 Working standard test blocks shall be ground on both flat faces.
A1.6.6.2 The face on which indentations are to be made shall have had a minimum of 0.35 mm (0.014 in.) ground off of the as-sintered surface and shall have a surface finish of Ra ≤0.2 µm (8 µin.), and it may be polished. The maximum deviation in flatness of the surfaces shall not exceed 0.010 mm (0.0004 in.). The bottom of the blocks shall not be convex. The maximum deviation in parallelism shall not exceed 1 part per 2500 parts.
A1.6.7 A secondary standard test block traceable to a CCPA master standard test block shall be chosen that has a hardness nearest to that of the working standard test block, and ten indentations shall be made on its test surface. The arithmetic mean of the ten results shall be calculated to the nearest 0.01 HRA and subtracted from the certified hardness of the secondary standard test block. The result is the correction for the given combination of testing machine and indenter.
A1.6.8 If the correction is greater than 0.3 HRA absolute, the machine and indenter shall be examined to ascertain the cause, and the test shall then be repeated.
A1.6.9 If the correction is ≤0.3 HRA absolute, ten indentations shall be made on the test surface of the candidate working standard test block, and the standard deviation of the results shall be calculated.
A1.6.10 Standard deviation, \(s\),
\[
\sigma = \sqrt{\frac{\sum(x - x)^2}{n - 1}} \tag{A1.1}
\]
where:
\(x\) = the individual hardness result,
\(\bar{x}\) = the arithmetic mean of hardness results in the sample, and
\(n\) = the number of hardness results in the sample.
A1.6.11 If the standard deviation is greater than 0.10, HRA, the block shall be discarded, but it shall be recorded if it is not greater than 0.10 HRA. The arithmetic mean of the ten results shall be calculated to the nearest 0.01 HRA and corrected for the error of machine and indenter obtained above. The results shall be rounded off to the nearest 0.01 HRA and shall be
recorded as the hardness of the working standard test block.

A1.6.12 When calibrating a number of working standard test blocks of the same nominal hardness in an uninterrupted series, the correction may be determined only at the commencement of the series, unless there is reason to believe that the correction may have altered.

A1.6.13 The thickness of the secondary standard test block shall be measured and recorded.

A1.6.14 The test surfaces of the working standard test blocks shall not be reground unless they are then recalibrated against secondary standard test blocks.

A1.7 Test Report:

A1.7.1 Supply a test report or certificate with every secondary or working standard test block, and include the following information:

A1.7.1.1 A reference to this test method, ASTM B 294,
A1.7.1.2 All details necessary for identification of the test block,
A1.7.1.3 The hardness as determined in accordance with this Annex,
A1.7.1.4 The standard deviation of hardness readings over the test surface,
A1.7.1.5 The thickness of the block and the date on which it was calibrated,
A1.7.1.6 Identification of the block from which it was calibrated,
A1.7.1.7 The name of the institution, association laboratory, or individual responsible for the calibration, and
A1.7.1.8 Any deviations from the procedures of this test method, or a statement that there have been no deviations.

A1.8 Indenter Selection:

A1.8.1 Select suitable indenters from standard Scale A indenters.

A1.8.2 Conduct a performance test of the indenter. Make at least three indentations on each of a series of five cemented carbide standard test blocks having the nominal hardnesses 85.5, 88.5, 91, 92, and 93 and conforming to the specifications of this Annex. Calculate the arithmetic mean of the hardness readings taken on each block. Determine the difference between the mean hardness of each block and its certified hardness, retaining the plus or minus signs. Tabulate these differences in the order of increasing hardness of the blocks. Calculate the algebraic difference between adjacent differences in the table and divide by 2. The absolute value of the resulting quotient is the maximum expected interpolation error when the indenter is used to measure the hardness of a test piece having a hardness between the hardnesses of the standard test blocks from which the quotient resulted. An indenter for which all of the quotients are 0.15 or less shall be accepted for use within a hardness range extending from the actual hardness of the hardest standard test block plus 0.3 HRA to that of the softest minus 0.3 HRA.

A1.8.3 An indenter discarded by the criteria of A1.8.2 may be accepted for use over a narrower hardness range. From the data generated in A1.8.2, select a subset of adjacent standard test blocks for which the acceptance criteria of A1.8.2 is met. The indenter may be accepted for use within a hardness range extending from the actual hardness of the hardest in the subset, plus 0.3 HRA, to that of the softest in the subset, minus 0.3 HRA.

A1.8.4 Include an example of the calculations and decisions for A1.8.2 and A1.8.3.

A1.8.5 Record the serial number of the indenter and the HRA range over which it has been approved for use, as well as the data of the selection test. The record should be posted, with indenters flagged in a manner such that they are used only within the approved range.
Test Method for Density Determination for Powder Metallurgy (P/M) Materials Containing Less Than Two Percent Porosity

This standard is issued under the fixed designation B 311; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1 NOTE—Paragraphs 3.1, 4.1, 4.2, 5.4, and 6.1 were revised editorially in June 2002.

1. Scope

1.1 This test method covers the determination of density for powder metallurgy (P/M) materials containing less than two percent porosity and for cemented carbides. This test method is based on the water displacement method.

NOTE 1—A test specimen that gains mass when immersed in water indicates the specimen contains surface-connected porosity. Unsealed surface porosity will absorb water and cause density values higher than the true value. This test method is not applicable if this problem occurs.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ISO Standard:

3369 Impermeable Sintered Metal Materials and Hardmetals—Determination of Density

NOTE 2—The water density table in ISO 3369 differs from the table contained in this test method.

3. Summary of Test Method

3.1 Using an analytical balance, the test specimen is first weighed in air and then in water. The density is determined by calculation.

4. Significance and Use

4.1 For P/M materials containing less than two percent porosity, a density measurement may be used to determine if the part has been densified, either overall or in a critical region, to the degree required for the intended application. Density alone cannot be used for evaluating the degree of densification because chemical composition and heat treatment affect the pore-free density.

4.2 For cemented carbides, a density measurement is normally used to determine if there is any significant deviation in composition of the carbide grade. For straight tungsten carbide-cobalt grades, the relationship is straightforward. For complex carbide grades (for example, grades containing tantalum carbide or titanium carbide, or both, in addition to tungsten carbide-cobalt), the situation is more complicated. If the measured density is beyond the specified limits, the composition is outside of the specified limits. A measured density within the specified limits does not ensure correct composition; compensation between two or more constituents could result in the expected density with the wrong composition. Density alone cannot be used for evaluating a cemented carbide grade.

5. Apparatus

5.1 Analytical Balance, precision single-pan analytical balance that will permit readings within 0.01 % of the test specimen mass. The analytical balance shall be supported in a manner to eliminate mechanical vibrations and be shielded from air drafts.

5.2 Weighing Liquid—Distilled or deionized water to which 0.05 to 0.1 volume percent of a wetting agent has been added to reduce the effects of surface tension.

NOTE 3—Degassing the water by evacuation, boiling, or ultrasonic agitation helps to prevent air bubbles from collecting on the test specimen and specimen support when immersed in water.

5.3 Water Container—A glass beaker or other suitable transparent container should be used to contain the water.

NOTE 4—A transparent container makes it easier to see air bubbles adhering to the test specimen and specimen support when immersed in water.

5.4 Water Container—A glass beaker or other suitable transparent container should be used to contain the water.

NOTE 5—For the most precise density determination, the water container should be of a size that the level of the water does not rise more than 2.5 mm (0.10 in.) when the test specimen is lowered into the water.
5.4 Test Specimen Support for Weighing in Water—Two typical arrangements are shown in Fig. 1. The suspension wire may be twisted around the test specimen or the test specimen may be supported in a wire basket that is attached to the suspension wire. For either arrangement, a single corrosion resistant wire—for example, austenitic stainless steel, copper, nichrome—shall be used for the basket and suspension wire. The maximum recommended diameter of suspension wire to be used for various mass ranges is:

<table>
<thead>
<tr>
<th>Mass, g</th>
<th>Wire Diameter, mm (in.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>less than 50</td>
<td>0.12 (0.005)</td>
</tr>
<tr>
<td>50 to less than 200</td>
<td>0.25 (0.010)</td>
</tr>
<tr>
<td>200 to less than 600</td>
<td>0.40 (0.015)</td>
</tr>
<tr>
<td>600 and greater</td>
<td>0.50 (0.020)</td>
</tr>
</tbody>
</table>

**NOTE 6**—For the most precise density determinations, it is important that the mass and volume of all supporting wires immersed in water be minimized.

5.5 Thermometer—A thermistor thermometer should be used to measure the temperature of the water to the nearest 0.5°C.

6. Preparation of Test Specimens

6.1 A complete part or a section of a part may be used for the test specimen. For the highest precision, the test specimen shall have a minimum mass of 5.0 g. If less precision can be tolerated, several test specimens may be used to reach the minimum mass, provided each test specimen has a mass of not less than 1.0 g.

6.2 All test specimen surfaces shall be thoroughly cleaned of all adhering foreign materials, such as, dirt, grease, oil, oxide scale, metal powders or assembly materials. For cut specimens, care must be used to avoid rough surfaces to which an air bubble can adhere. A100-grit sanding or abrasive grinding is recommended to remove all rough surfaces.

7. Procedure

7.1 Weigh the test specimen in air using an analytical balance. This is mass A. This and all subsequent weighings shall be to 0.01% of the test specimen mass, for example:

<table>
<thead>
<tr>
<th>Mass, g</th>
<th>Balance Sensitivity, g</th>
</tr>
</thead>
<tbody>
<tr>
<td>less than 10</td>
<td>0.0001</td>
</tr>
<tr>
<td>10 to less than 100</td>
<td>0.001</td>
</tr>
<tr>
<td>100 to less than 1000</td>
<td>0.01</td>
</tr>
<tr>
<td>1000 to less than 10 000</td>
<td>0.1</td>
</tr>
</tbody>
</table>

**NOTE 7**—For the most precise density determination, duplicate weighings should be made for all mass determinations. The analytical balance should be adjusted to zero prior to each weighing. Duplicate mass determinations should be averaged before calculating the density.

**NOTE 8**—For improved reproducibility, the analytical balance should be periodically calibrated with a standard mass that is approximately equal to the test specimen mass.

7.2 Support the container of water over the pan of the balance using an suitable bridge as shown in Fig. 2. The container of water may also be supported below the balance for weighing larger specimens if the balance has a lower beam hook for this purpose. See Fig. 2b. If this arrangement is used, it is important to shield the suspension wire between the container of water and the bottom of the balance from air drafts.

7.3 Suspend the test specimen support with the test specimen from the beam hook of the balance. The water should cover any wire twists and the specimen support basket by at least 6 mm (¼ in.) to minimize the effect of surface tension forces on the weighing. Care should be taken to ensure that the test specimen and specimen support hang freely from the balance beam hook, are free of air bubbles where immersed in the water and are at the same temperature as the water and balance. Care should also be taken to ensure the surface of the water is free of dust particles.

7.4 Weigh the test specimen and specimen support immersed in water. This is mass B.

7.5 Remove the test specimen. Weigh the test specimen support immersed in water at the same depth as before. This is mass C. Care should be taken to ensure that the suspension support is free of air bubbles and that the suspension wire is not immersed below its normal hanging depth as a change in depth will change the measured mass.

**NOTE 9**—Some balances are capable of being tared. This automatically removes the necessity of reweighing the specimen support every time. In this case, tare the specimen support alone, immersed in water to the same depth as with the specimen, before weighing the specimen support and specimen immersed in water. The mass of the specimen support and specimen immersed in water is mass F, which replaces mass B minus mass C.

7.6 Measure the temperature of the water to the nearest 0.5°C and record its density E, at that temperature, from Table 1.

8. Calculation

8.1 Calculate the density as follows:

\[
\text{Density} = D = \frac{\text{Mass}}{\text{Volume}} \tag{1}
\]

\[
D = \frac{A - (B - C)}{E} \tag{2}
\]

\[
D = \frac{(A \times E)}{(A - B + C)} = \frac{(A \times E)}{(A - F)} \tag{3}
\]
where:
\[ D = \text{density of test specimen, g/cm}^3, \]
\[ A = \text{mass of test specimen in air, g}, \]
\[ B = \text{apparent mass of test specimen and specimen support in water, g}, \]
\[ C = \text{mass of specimen support immersed in water, g}, \]
\[ F = \text{mass of test specimen in water with mass of specimen support tared, g}, \]
\[ E = \text{density of water in g/cm}^3. \]

9. Report

9.1 Report the density rounded to the nearest 0.01 g/cm³.

10. Precision and Bias

10.1 The following precision and bias data were developed using the procedures contained in Test Method B 311 – 86. An interlaboratory study is in-progress using the procedures in this revised test method.

10.2 For test specimens over 5 g mass, the repeatability interval, \( r \), is 0.025 g/cm³. Duplicate results from the same laboratory should not be considered suspect at the 95 % confidence level unless they differ by more than \( r \).

10.3 For test specimens of over 5 g mass, the reproducibility interval, \( R \), is 0.03 g/cm³. Test results from two different laboratories should not be considered suspect at the 95 % confidence level unless they differ by more than \( R \).

10.4 For test specimens of 1 to 5 g mass, the repeatability interval, \( r \), is 0.025 g/cm³.

10.5 For test specimens of 1 to 5 g mass, the reproducibility
interval, \( R \), is 0.05 g/cm\(^3\).

10.6 There is no estimate of bias because there is no accepted reference material.

11. Keywords

11.1 cemented carbides; density; hard metals; metal injection molded (MIM) parts; powder metallurgy (P/M); powder forged (P/F) parts; powder metallurgy

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Standard Test Method for
Green Strength for Compacted Metal Powder Specimens

1 This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.02 on Base Metal Powders.


1 Annual Book of ASTM Standards, Vol 02.05.
8. Procedure

8.1 For lubricated powders follow the instructions starting in Section 8.2. The method of lubrication of the powders shall be standardized since green strength, green density, and compactibility will vary with the method chosen and the care with which it is applied. The method of lubrication shall be a matter of agreement between the parties concerned. Unlubricated powder may be tested in a die with lubricated walls. Apply to the die walls a lubricant mixture (for example, a mixture of 100 g of zinc stearate in one liter of methyl alcohol. Warning: This mixture is flammable and should be used in a suitable, well ventilated area.) After any excess liquid has drained away, allow the die walls to dry, and fill with the powder being tested, as described in 8.2 and 8.3.

8.2 Determine from Table 1 the approximate mass of powder to be used to make a test specimen 0.250 ± 0.005 in. (6.35 ± 0.13 mm) or 0.500 ± 0.005 in. (12.7 ± 0.13 mm) thick by 0.500 in. (12.7 mm) wide by 1.250 in. (31.7 mm) long. See Fig. 4. Weigh this charge to ±0.02 g.

8.3 The specimen is prepared using a double action pressing process. One example of this type of compaction is as follows: With the lower punch inserted in the die cavity, pour the powder into the die cavity taking care that the powder is uniformly distributed. Apply pressure from both ends by means of the following arrangement: Insert the upper punch, and place the die, supported on two spacers, between the platens of the press. Apply a preliminary pressure of 5000 psi (34 MPa) to the upper punch, while the die barrel is supported by the spacers; then release the pressure and remove the spacers. With the spacers removed, apply the final compacting pressure at a rate of 60000 ± 5000 psi (414 ± 34 MPa)/min.

8.4 Use a compacting pressure that will produce the correct thickness of 0.250 ± 0.005 in. (6.35 ± 0.13 mm) or 0.500 ± 0.005 in. (12.7 ± 0.13 mm).

8.5 After ejection from the die, weigh the specimen to the nearest 0.01 g, measure the specimen dimensions to the nearest 0.001 in. (0.03 mm), and identify the top of the specimen. Calculate the density of the green specimen. If the specimen density is within the tolerance, place the specimen in the transverse rupture test fixture or the constant loading beam device perpendicular to the supporting rods with the top uppermost. In the case of the transverse rupture test fixture, place the loaded fixture between the platens of the compression testing machine set for an initial crosshead velocity of approximately 0.1 in./min and apply a uniformly increasing force at a rate of approximately 20 lbf (89 N)/min until rupture occurs. Record the breaking force in lbf (or N) to the nearest 0.1 lbf (0.5 N). When using the constant loading beam device (Fig. 2), shot is allowed to flow into a suitable container at a rate that will produce approximately 20 lbf (89 N)/min on the specimen. The mass of the shot is determined to the nearest gram (0.0022 lb) and the breaking force in lbf (or N) is calculated to the nearest 0.1 lbf (0.5 N) as follows:

$$ P = \frac{AX}{B} $$  \hspace{1cm} (1)

where:
- $P = \text{force on test specimen, lbf (N)}$,
- $A = \text{length} A, \text{in. (mm)}$,
- $B = \text{length} B, \text{in. (mm)}$, and
- $X = \text{mass of shot, lb (kg)}$ to the nearest 0.0022 lb (1 g) required to rupture.

9. Calculations

9.1 Calculate the green strength as follows:

$$ S = \frac{3PL}{2w^2} $$  \hspace{1cm} (2)

where:
- $S = \text{green strength, psi (MPa)}$,
- $P = \text{force required to rupture, lbf (N)}$,
- $L = \text{length of specimen span of fixture, in. (mm)}$,
- $w = \text{width of specimen, in. (mm)}$, and
- $t = \text{thickness of specimen, in. (mm)}$.

10. Report

10.1 The green strength shall be reported in pounds-force per square inch (or megapascals) as the average of three individual results to the nearest 100 psi (or .5 MPa). The average green density shall always be reported with the green strength. The nominal thickness of the test specimen should be indicated where necessary for clarity. The lubrication method shall also be reported.

11. Precision and Bias

11.1 The following criteria should be used to judge acceptability of individual results at the 95 % confidence level for the
0.250 in. thick specimen. Repeatability and reproducibility data for the 0.500 in. specimen are not available.

**NOTE 1**—This precision statement is different from the statements in other B09 test methods because it describes the repeatability and reproducibility of individual values, not the averages of groups of test values.

11.1.1 The repeatability interval, \( r \), is estimated by the following equation: 
\[
r = 0.037 S + 78, 
\]
where \( S \) = green strength, (psi). Duplicate individual results by the same operator should be considered suspect if they differ by more than \( r \).

11.1.2 The reproducibility interval, \( R \), is estimated by the following equation: 
\[
R = 0.13 S + 220. 
\]
The individual results obtained by each of two laboratories should not be considered suspect unless they differ by more than \( R \).

11.2 The bias of the green strength test cannot be established because there is no standard available for comparison.

**12. Keywords**

12.1 constant loading beam device; green strength; P/M; powder metallurgy; unsintered compact
1. Scope

1.1 This guide provides methods for preparing lead or lead alloy products for the application of electroplated or autocatalytic coatings.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

2.1.1 B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings

3. Significance and Use

3.1 The preparation of lead and lead-alloy surfaces for electroplating is often critical to the successful performance of electrodeposited and autocatalytic metallic coatings.

3.2 This standard outlines the process operation procedures and processing solutions required, that lead to satisfactory electrodeposited metallic coatings (including undercoating) on surfaces of lead and lead-alloys.

4. Nature of Lead

4.1 The tensile strength of lead and lead alloys ranges from 15 to 35 MPa (2000 to 5000 psi), therefore, the measured adhesion of electroplated coatings cannot be greater than these values.

4.2 Difficulties in applying high-quality electroplated coatings to lead are due to the following properties of lead:

4.2.1 The very active chemical nature of lead, leading to the formation of oxide films in air,

4.2.2 The fact that the lead surface will form films of insoluble lead salts with most acids used in pickling,

4.2.3 The ease with which lead diffuses in contact with nonferrous metals, and

4.2.4 The poor resistance to plastic deformation during polishing.

5. Process Precautions

5.1 The following process precautions should be observed:

5.1.1 Precleaning of raw castings is sometimes necessary to remove mold parting compounds, surface oxides, and residues from recessed areas which are never reached by polishing-wheel or scratch-brush operations (Section 6).

5.1.2 In high-speed type cyanide electroplating solutions, the initial current density must be controlled and kept low enough so that no gassing occurs to cause poor adhesion. This is revealed as groups of blisters in the high-current-density areas of the electroplate.

5.1.3 If a strike electroplate is used, it should be thick enough to prevent the next electroplating solution from attacking the basis lead. A copper or nickel strike 2.5 µm thick should be used, but because there are so many variables involved, no specific recommendations can be made.

5.1.4 Preplates should be of such thickness that complete alloying with the lead does not take place, an occurrence that causes poor adhesion of subsequent deposits. This defect is indicated by blistering after prolonged storage or after an accelerated aging test.

5.1.5 The lead compounds formed by the action of acids and alkalies most often used in electropolating are not water soluble. Caution must be taken to remove or prevent the formation of these to eliminate subsequent adhesion failure. Acids that cannot be used are sulfuric, hydrochloric, and hydrofluoric. (Acids that can be used are sulfamic and fluoboric.) Alkalies should not be high in caustic content. Mild or buffered cleaners are preferred in order to minimize attack on the basis lead surface.

5.1.6 Engraving of electroplated finishes on lead cannot be performed on deposits over 5 µm thick as the deposit will tear away from the lead at cross cuts. Engine turning by burnishing can be done on any thickness of deposits.
5.1.7 Polishing and coloring of the deposit must be performed at slow speeds, and with loose or cooled buffs to eliminate overheating and flowing of basis metal.

6. Precleaning

6.1 Remove fins and parting lines by use of trimming dies or by scraping, filing, or grinding. Some machining may be done at this point, such as drilling holes, or milling or cutting slots, grooves, flats, or squared surfaces.

6.2 Clean in alkaline, emulsion type, or other standard cleaning material to remove surface materials (5.1.5).

6.3 After rinsing, transfer the parts into one of the following pickling solutions, the function of which is to remove surface oxides, without significant attack on the lead surface.

6.3.1 An aqueous solution containing 250 mL of 48 mass %, fluoboric acid with or without 45 mL of 30 mass % hydrogen peroxide diluted to 1 L. The addition of hydrogen peroxide will increase the aggressiveness of the pickling solution.

6.3.2 An aqueous solution containing 80 mL of glacial acetic acid and 45 mL of 30 mass % hydrogen peroxide diluted to 1 L.

6.3.3 An aqueous solution containing 100 g of sulfamic acid diluted to 1 L.

Note 1—These pickling solutions should be held in tanks having suitable nonmetallic linings.

6.4 After rinsing, an immersion in a water-displacing, film-forming material is advantageous. Sometimes a neutral soap, oil, or synthetic emulsion film may be used according to the type of polishing compound to be used. These materials are removed in subsequent operations.

7. Assembly of Parts

7.1 Precleaned parts then are ready for further assembly, such as soldering to other metals for ornamentation or mechanical reasons.

8. Polishing or Buffing

8.1 After parting lines, fins, and gate marks are removed, greaseless compounds on loose cloth wheels are used for rough smoothing followed by a soft leather, chamois, or sheepskin wheel operating at 15 to 25 surface m/s. These are used with white lime compounds of various grease concentrations. Cloth wheels can be used on highly ornamented parts, but not on any surface having a smooth area, as drag marks will occur.

9. Preparation for Electroplating

9.1 Precleaning:

9.1.1 Solvent or solvent emulsion cleaners can be used if the parts can be rinsed easily and completely; otherwise alkaline cleaners and ammonia, or wetting agent soaks should be used. Spray washing with alkaline or solvent-type cleaners may be used before the electrocleaning cycle instead of soak cleaning.

9.2 Electrocleaning:

9.2.1 Cathodic electrocleaning usually is used, especially for decorative bright finishes. Anodic electrocleaning dissolves the lead and is used sometimes in industrial or special applications if stressed or distorted surface layers must be removed to expose the natural understructure.

9.2.2 There are proprietary cleaners designed for special conditions; however, a cleaner made up using 23 g/L of sodium carbonate and 23 g/L of trisodium orthophosphate, anhydrous operated at 60 to 80 °C with 6 to 8 V with the work cathodic for 30 to 60 s is advantageous. Hand cleaning by mopping and brushing is performed, but the trend is away from hand operations.

9.3 Acid Pickle:

9.3.1 An acid pickle of one of the following types is used to remove all oxide residues and insoluble compounds left from cleaning:

9.3.1.1 An aqueous solution containing 120 to 250 mL of 48 mass % fluoboric acid diluted to 1 L used at 20 to 25°C for 30 to 60 s. See Practice B 281.

9.3.1.2 An aqueous solution containing 100 g of sulfamic acid dissolved in water and diluted to 1 L used at 20 to 25°C for 30 to 60 s. A blend of 75 to 85 % by weight sulfamic acid and 15 to 25 % by weight ammonium bifluoride may be dissolved in water at 60 to 120 g/L to form an equivalent solution.

10. Typical Cycles

10.1 Stereotype Metal (80 to 84 % Lead, 11 to 13 % Antimony, 4 to 6 % Tin)

10.1.1 Preparation for Nickel or Iron Stereotypes:

10.1.1.1 Electroclean cathodically and then anodically with the following solution or a proprietary equivalent:

<table>
<thead>
<tr>
<th>Solution</th>
<th>Temperature</th>
<th>Voltage</th>
<th>Anodic</th>
<th>Cathodic</th>
</tr>
</thead>
<tbody>
<tr>
<td>60 to 90 g/L trisodium orthophosphate crystals (Na₃PO₄·2H₂O)</td>
<td>40°C</td>
<td>6 V</td>
<td>3 to 4 min</td>
<td>3 to 4 min</td>
</tr>
</tbody>
</table>

10.1.1.2 Cold water rinse and spray.

10.1.1.3 Acid dip with the following solution:

<table>
<thead>
<tr>
<th>Solution</th>
<th>Temperature</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>25 volume % of 42 % fluoboric acid</td>
<td>20 to 25°C</td>
<td>10 to 15 s</td>
</tr>
</tbody>
</table>

10.1.1.4 Cold water rinse and spray.

10.1.1.5 Nickel or iron electroplate, 20 µm thick.

Note 2—A small amount of current must be applied prior to and while the article is entering the plating solution. After entry, the current should be adjusted to its normal value. Additionally, a nickel strike (see Table X1.1) may be used prior to the nickel or iron electroplate.

10.1.2 Alternative Preparation for Iron Electroplating Stereotypes:

10.1.2.1 Vapor degrease or wash in Stoddard solvent to remove proof-printing ink.

10.1.2.2 Electroclean (see 10.1.1.1).

10.1.2.3 Scrub with tampico brush and cleaning solution used in 10.1.2.2.

10.1.2.4 Thoroughly rinse with water.

10.1.2.5 Acid dip as in 10.1.1.3 (see 9.3.1, section 9.3.2, and section 9.3.3).

10.1.2.6 Thoroughly rinse with water.

10.1.2.7 Iron electroplate, 20 µm thick. (See 10.1.1.5.)

Note 3—The iron may be electrodeposited from the following solution:
Ferrous ammonium sulfate \((\text{FeSO}_4\cdot(\text{NH}_4)_2\text{SO}_4\cdot6\text{H}_2\text{O})\) 340 g/L
Boric acid \((\text{H}_3\text{BO}_3)\) 40 g/L
pH 3.1 to 3.4 (adjusted with sulfuric acid)
Temperature 60°C
Current density 1 to 5 A/dm²

11. Decorative Applications

11.1 Preparation for Copper Striking:
11.1.1 Preclean and water rinse (see 9.1).
11.1.2 Electroclean (see 9.2).
11.1.3 Water rinse.
11.1.4 Dip in acid (see 9.3).
11.1.5 Thoroughly water rinse.
11.1.6 Cyanide copperstrike to give complete coverage (see Table X1.3). Other proprietary copper strikes may be used.
11.1.7 Water rinse thoroughly to remove all traces of cyanide.
11.1.7.1 Prior to further electroplating, use a 1 to 2% sulfuric acid rinse.
11.1.8 Follow with other electrodeposits as required.

11.2 Preparation for Nickel Striking:
11.2.1 Preclean and water rinse (see 9.1).
11.2.2 Electroclean (see 9.2).
11.2.3 Water rinse.
11.2.4 Acid dip (see 9.3).
11.2.5 Water rinse.
11.2.6 Nickel strike to give complete coverage (see Table X1.1 and Table X1.2).
11.2.7 Water rinse.
11.2.8 Follow with other electrodeposits as required.

12. Small Parts

12.1 Preparation for Barrel Planting:
12.1.1 See Section 6 for precleaning steps.
12.1.2 Acid dip (see 9.3).
12.1.3 Thoroughly water rinse.
12.2 Cyanide copperstrike to give complete coverage (see Table X1.3). Other proprietary copper strikes may be used.
12.2.1 Water rinse thoroughly to remove all traces of cyanide.
12.2.2 Prior to electroplating, use a 1 to 2% sulfuric acid rinse.

APPENDIX

(Nonmandatory Information)

X1. STRIKE SOLUTIONS

X1.1 Table X1.1, Table X1.2, and Table X1.3 contain the composition of the solutions and the operating conditions for nickel and copper strikes.

<table>
<thead>
<tr>
<th>TABLE X1.1 Nickel Sulfamate Strike Solution</th>
<th>Quantity, g/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel sulfamate, Ni(SO₃NH₂)₂</td>
<td>250</td>
</tr>
<tr>
<td>Nickel chloride ((\text{NiCl}_2\cdot6\text{H}_2\text{O}))</td>
<td>25</td>
</tr>
<tr>
<td>Boric acid ((\text{H}_3\text{BO}_3))</td>
<td>30</td>
</tr>
<tr>
<td>pH</td>
<td>3.5</td>
</tr>
<tr>
<td>Temperature</td>
<td>30 to 35°C</td>
</tr>
<tr>
<td>Current density</td>
<td>1 to 2 A/dm²</td>
</tr>
</tbody>
</table>
### TABLE X1.2 Nickel Fluoborate Strike Solution

<table>
<thead>
<tr>
<th></th>
<th>Quantity, g/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel fluoborate (Ni(BF&lt;sub&gt;4&lt;/sub&gt;)&lt;sub&gt;2&lt;/sub&gt;)</td>
<td>250</td>
</tr>
<tr>
<td>Boric acid (H&lt;sub&gt;3&lt;/sub&gt;BO&lt;sub&gt;3&lt;/sub&gt;)</td>
<td>30</td>
</tr>
<tr>
<td>pH (paper)</td>
<td>3.5</td>
</tr>
<tr>
<td>Temperature</td>
<td>30 to 60°C</td>
</tr>
<tr>
<td>Current density</td>
<td>1 to 2 A/dm&lt;sup&gt;2&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

### TABLE X1.3 Copper Strike Solution

<table>
<thead>
<tr>
<th></th>
<th>Quantity, g/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper cyanide (CuCN)</td>
<td>22.5</td>
</tr>
<tr>
<td>Sodium cyanide (NaCN)</td>
<td>40</td>
</tr>
<tr>
<td>Sodium carbonate (Na&lt;sub&gt;2&lt;/sub&gt;CO&lt;sub&gt;3&lt;/sub&gt;)</td>
<td>15</td>
</tr>
<tr>
<td>Total copper (Cu)</td>
<td>16</td>
</tr>
<tr>
<td>Free cyanide (CN&lt;sup&gt;-&lt;/sup&gt;)</td>
<td>15</td>
</tr>
<tr>
<td>Temperature</td>
<td>40 to 50°C</td>
</tr>
<tr>
<td>Current density</td>
<td>1 to 1.5 A/dm&lt;sup&gt;2&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

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Standard Practice for Preparation of Iron Castings for Electroplating

This standard is issued under the fixed designation B 320; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice is intended to assist electroplaters in establishing and maintaining a satisfactory pre-electroplating cycle for malleable iron, gray iron, nodular iron, and white iron castings. It is also intended to indicate certain foundry practices which will facilitate subsequent finishing. Most of the practices that follow have been based on experience with malleable and gray iron. However, since they are related to the other forms, the same practices will probably apply. Nodular iron is also known as spheroidal or ductile iron, which is defined as cast iron with the graphite substantially in spherical shape and substantially free of flake graphite.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in 2.1.

2. Reagents

2.1 Purity of Reagents—All acids and chemicals used in this practice are technical grade. Diluted acid solutions are based upon the following assay materials:

- Hydrochloric acid (HCl): 31 mass %, density 1.16 g/mL
- Hydrofluoric acid (HF): 47 mass %, density 1.186 g/mL
- Sulfuric acid (H₂SO₄): 93 mass %, density 1.83 g/mL

(Warning—Use hydrofluoric acid with extreme care.)

(Warning—Sulfuric acid should be slowly added to the approximate amount of water required with rapid mixing, and then after cooling, diluted to exact volume.)

2.2 Purity of Water—Ordinary industrial or potable water may be used for preparing solutions and rinsing.

3. Foundry Practices

3.1 The seacoal content of the molding and facing sands should be maintained at the maximum practicable limits to minimize the occlusion of sand in the surfaces of the castings.

3.2 Upon removal from the molds, castings should be subjected to an abrading action (such as tumbling, grit blasting, or shot blasting) to remove as much as practicable of the occluded molding sand. Residual sand and scale may be removed, if necessary, by treatment in various proprietary descaling baths. These are usually based on fused caustic soda, some of which use chemical oxidizing or reducing agents and others employ electrochemical action as well. This is particularly important in the case of castings that will be annealed, to prevent the burning on of sand during this operation. Castings that will be warped or damaged by a blasting operation may be pickled in a solution containing 200 to 250 mL/L of sulfuric acid to remove occluded molding sand. See Warning statement in 2.1.

3.3 Annealed castings should be given an additional abrading as described in 3.2 to remove any scale that may have been formed, as well as graphicitic carbon that may be present at the surface.

4. Nature of Cleaning

4.1 The preparation of ferrous castings for electroplating involves the following basic steps in the order named:

4.1.1 The removal of oils, greases, residual polishing and buffing compounds (if any), and shop dirt by cleaning,

4.1.2 The removal of oxide films and scales and the loosening of surface carbon by pickling or by salt bath treatment (see 3.2),

4.1.3 The removal of smut caused by 4.1.2, and

4.1.4 Activation for electroplating.

4.2 Where excessive amounts of cutting oils used in machining operations are present, it may be necessary to preclean the parts before they reach the electroplating room. This may require the use of organic solvents, vapor degreasers, washing machines of the power-spray type, emulsion cleaners, or simple alkaline soak tanks. As short a time as possible should elapse between this precleaning and the preplating cleaning...
cycle so as to prevent rusting of the parts. Where control of the interval is not possible, parts should be left with a slightly alkaline or very thin organic film.

5. Cleaning Solutions and Equipment

5.1 The various solutions used for the treatment of malleable and gray iron castings should be maintained by chemical analysis so far as is practicable, such as determining the free acid and iron concentrations of the acid baths and using tests recommended by the manufacturer, the effective components of the proprietary cleaning solutions.

5.2 All solutions should be discarded before they lose their effectiveness, based on tests and experience.

5.3 When the amount of soil is excessive, particularly where no precleaning is done, it may be desirable to double the cleaning and pickling facilities. Thus, while the first of any two particular solutions becomes heavily contaminated, the second remains relatively clean and effective for further use. When the first of a pair of solutions is discarded, it is replaced by the second solution and a fresh second solution is prepared. This system also reduces the possible carry-over of contaminants such as oil and grease into subsequent solutions.

5.4 Where doubling the facilities is impossible or impracticable, similar economies may be obtained to a degree by providing cleaner and pickle tanks with overflow dams, sumps and pumps with which the solution may be recirculated. The pump intake should be located approximately half-way down the sump to preclude returning either settled-out solid dirt or surface oil and grease to the processing tank. The outlet should be near the bottom of the processing tank at the end opposite to the overflow dam so as to create some solution turbulence (for mechanical scrubbing benefits) and to ensure flow of contaminated solution to the dam.

5.5 In electrified tanks removable electrodes should be employed in preference to using the tank as an electrode, to facilitate inspection and cleaning. To ensure good circuitry, positive contacts such as an inverted V hook for round bars should be used. In alkaline cleaner tanks, where clean contact is often a problem, submerged oversized steel tank rods are effective.

5.6 All immersion rinse tanks should be equipped with dam-type overflows to ensure skimming of oil, grease, and light dirt from the surface of the water. Water inlets should be at the bottom of the tank, and should be of a size sufficient to provide an adequate flow of water. It is desirable that submerged inlet pipes be equipped with syphon-breakers (as required by law in many areas) not only to prevent the backflow of contaminated water into the mains, but also to produce a beneficial turbulence or scrubbing action due to the air introduced with the water. Agitation by means of low-pressure, oil-free air through perforated pipes at the bottom of the tank may also be used.

5.7 Having the heating coils on the working side of tanks will assure a working surface which is free of accumulated grease and oil.

6. Procedure for Racked Parts

6.1 The following cycle may generally be used for racked parts which will subsequently be electroplated in still tanks, semiautomatic equipment, and full-automatic equipment:

6.1.1 Precleaning—When castings have been subjected to machining, polishing, buffing, or similar finishing processes, it is desirable and frequently essential that lubricants and finishing compounds be removed by precleaning immediately following such operations. This is especially important when the lubricants contain unsaturated oils which, upon air oxidation, form films which are extremely difficult to remove. Precleaning methods as listed in 4.2 may be employed.

6.2 Soak Cleaning—In the event precleaning of a heavily soiled part is impossible or impracticable, soak cleaning to loosen oils and greases is recommended. The bath may be either an alkaline solution of such concentration as recommended by the supplier, and operated at a temperature as close to boiling as possible, or an emulsion-type cleaner operated as specified by the supplier. In either case, agitation of the solution by air or solution pumping, or movement of the part, will prove beneficial. The time may be 5 min or more.

6.3 Rinse—If the soak cleaner used is incompatible with the subsequent cleaner, a rinse is indicated. The supplier will normally suggest whether it is to be warm or cold, although a warm rinse (60°C) is usually desirable following alkaline soak cleaning. In any case, agitation of the rinse water is desirable; and, in the case of cold-water rinses, a spray upon leaving the tank is beneficial. The time of rinsing depends in part upon the shape of the part, but should be no less than 10 s.

6.4 Anodic Cleaning—The part is made the anode in a solution of a properly compounded alkaline cleaner of a concentration recommended by the supplier. The cleaner should be free-rinsing, and of high conductivity to permit a current density of 6 to 10 A/dm² at a tank potential of 6 to 9 V. The solution temperature should be from 90 to 100°C, and the cleaning time from 1 to 2 min.

6.5 Rinse—The supplier of a proprietary cleaner will usually indicate whether his product rinses more freely in warm or cold water. In general, rinsing should be done as described in 6.3, but preferably in a separate tank. Where practicable to do so, all rinses should be double rinses; that is, two separate rinses in succession, with the second cascading into the first for water economy.

6.6 Acid Pickling—This stage of the cycle is the most critical, and its operating conditions are dependent on the type of electroplating to follow. Most of the difficulties in electroplating of gray iron and malleable iron castings are caused by the free graphitic carbon, flake or nodular, which is present at the surface of the part. If the subsequent electroplating will be done under conditions causing sufficiently high hydrogen overvoltage (most acid solutions, and such alkaline solutions as copper, cadmium, or tin), a brief dip (less than 15 s) in a room temperature solution containing 200 mL/L of hydrochloric acid or 50 to 100 mL/L of sulfuric acid is usually adequate. See Warning in 2.1. If the electroplating will be done in an alkaline solution of low hydrogen overvoltage such as cyanide zinc, anodic treatment in acid to remove surface carbon is preferred. This is done by making the part the anode in a solution.
containing 250 to 350 mL/L of sulfuric acid for at least 30 s, preferably more, at a voltage sufficient to provide a current density of at least 10 A/dm². See Warning in 2.1. A black film of carbon smut will form during the first 15 to 30 s, then the part will become passive and the oxygen evolved on the part will remove the carbon by a combination of scrubbing and oxidation, leaving the casting relatively clean.

6.7 Rinse—The part should be rinsed in cold water as described in 6.3, but in a separate tank.

6.8 Anodic Cleaning—The casting should be subjected to anodic cleaning as described in 6.4, using either the same type of proprietary cleaner or a room temperature solution consisting of sodium cyanide (30 to 45 g/L) and sodium hydroxide (30 to 45 g/L).

6.9 Rinse—The part should be rinsed in cold water as described in 6.3, but in a separate tank.

6.10 Activation—If electroplating is to be done in alkaline solutions, such as cadmium, copper, tin, or zinc, no further treatment should be necessary. For nearly neutral or acid electroplating processes, however, parts should be immersed for 5 to 15 s in a room-temperature solution containing 50 to 100 mL of sulfuric acid. See Warning in 2.1.

6.11 Rinse—Activated parts should be rinsed in cold water as described in 6.3, but in a separate tank.

7. Variation in Procedure for Parts to be Racked

7.1 Where parts are subjected to several operations before electroplating, such as machining, forming, polishing, etc., thought should be given to possible precleaning between operations.

7.2 Mineral oils, particularly in recesses, are best removed by vapor degreasing.

7.3 Lubricating oils, buffing compounds greases, and the like, are best removed by anodic electrocleaning as described in 6.4. Heavy deposits of dried-on buffing compound may be more effectively removed by cathodic cleaning under similar operating conditions.

7.4 Castings that are heavily rusted can be pickled in a solution containing 50 to 100 mL/L of sulfuric acid and a suitable inhibitor to prevent excessive attack of the basis metal. See Warning in 2.1. Preferably, the solution should be operated at 65 to 95°C, and the time should be as required to remove the rust.

7.5 Very heavy scale can be removed by making the work anodic in a solution as described in 7.4, but with a suitable wetting agent instead of the inhibitor.

7.6 For castings from which molding sand has not been completely removed by the abrading processes described in 3.2 and 3.3, a dip in a water solution consisting of 125 mL/L of sulfuric acid and 125 mL/L of hydrofluoric acid at 20 to 30°C may be employed. See Warning in 2.1.

7.7 A suggested alternative for most of the cycle described in Section 6 is the use of one of the several proprietary processes involving molten caustics, hydrides, or alkaline salts, both with and without electrolysis. The suppliers of these processes should be consulted for details.

8. Procedure for Parts to Be Processed in Bulk

8.1 Castings to be cleaned in cylinders other than those in which the electroplating will be done should be processed through a cycle as follows:

8.1.1 Tumble Cleaning—Parts are cleaned by tumbling without current in a solution of properly compounded alkaline cleaner of 45 to 90 g/L at a temperature of 90 to 100°C for at least 5 min.

8.1.2 Rinse—Rinse for at least 1 min by tumbling in warm water, preferably agitated and overflowing.

8.1.3 Acid Pickle—Tumble the parts for at least 1 min in a solution of 250 to 350 mL/L of sulfuric acid at 65°C.

8.1.4 Rinse—Tumble in cold water, preferably agitated and freely overflowing, for at least 1 min.

8.1.5 Activation—Parts to be electroplated in acid solutions may be transferred to electroplating cylinders without further processing. Parts to be electroplated in cyanide solutions should be tumbled for at least 1 min in a room temperature solution consisting of 15 to 30 g/L of sodium cyanide and 15 to 30 g/L of sodium hydroxide.

8.1.6 Rinse—Parts activated as in 8.1.5 should be tumbled in cold water as described in 8.1.4, but in a separate tank.

8.1.7 Storage—If some time is to elapse between cleaning and electroplating, parts may be stored in a solution as described in 8.1.5. Castings to be subsequently electroplated in acid baths after storage must first be rinsed as described in 8.1.4, then tumbled for 1 min in a solution containing 30 to 50 mL of sulfuric acid, and rinsed again as described in 8.1.4 before being transferred to the electroplating cylinders. At best, it is not advisable to store castings for any length of time in such cyanide solutions, as salts may be retained in the pores and cause eventual spotting out.

9. Variations in Procedure for Parts to Be Processed in Bulk

9.1 Where both cleaning and electroplating will be done in the same barrel, using insulated cylinders, a cycle as described for racked work (Section 6) may be used, except that solution temperatures must be kept within the limits prescribed by the manufacturer of the barrels, and times increased by a factor of two or three to compensate for any lowering of temperature.

9.2 Electrolytic processes such as anodic cleaning and pickling should be carried out at a minimum of 12 to 15 V, so as to get the maximum current possible on the work within the temperature and current limits of the cylinder construction. Too low a current may not remove the smut and too high may damage the cylinder.

9.3 Where practicable, precleaning should be done as prescribed for racked work in 4.2.

10. Variations in Procedure for Various Types of Electroplating

10.1 Zinc Electroplating—Occasionally there is difficulty in the electroplating of certain castings which may be overcome by preceding the zinc electroplating with a strike such as cadmium, tin-zinc, tin, and acid zinc.

10.2 Chromium Electroplating—Variations in cleaning and pickling times should be investigated to find the optimum. In
certain cases it may be desirable to omit any preliminary wet processing and to resort to grit blasting.

11. Test for Effectiveness of Cleaning

11.1 The most reliable test of the effectiveness of the preplating cleaning cycle is the appearance of the electroplated part and its performance in service.

11.2 A commonly used test for the removal of greases and oils is the inspection for water breaks. This is best done after pickling or acid dipping, as an alkali film may mask a water break. The test is not always reliable.

11.3 Wiping a part with a clean white cloth just before entry into the electroplating bath will reveal whether the smut has been removed.

11.4 An inspection of parts after just a few seconds of electroplating can reveal either a uniform color, indicating a clean surface, or blotchy areas, indicating incomplete cleaning.
INTRODUCTION

This guide is intended to illustrate general principles of cleaning prior to electroplating. It is not meant to apply to every specific application. In specific cases, cleaning practice may depart from the general principles given in this guide.

1. Scope

1.1 This guide describes the procedure for cleaning metal surfaces to obtain good adhesion of electrodeposited metals. The degree of cleanliness required for metals to be electroplated is greater than for most other finishes. Methods of removal of heat-treat or mill scale are not included in these methods, because they are covered in practices referring to specific metals. It should also be understood that while these procedures are broadly applicable, particular substrates may require certain specific cleaning procedures.

1.2 Adequate cleaning requires a proper combination of cleaning procedures. The choice of these procedures must be based on a knowledge of the metals to be cleaned and of the soils to be removed. Because most experience and knowledge in cleaning have been obtained by suppliers of proprietary processes and formulations, these sources should be consulted before setting up a cleaning process.

1.3 A treatment to remove tarnish, light rust, fingerprints, or oxides is usually provided before immersion of the piece in the electroplating tank. This treatment activates the metal and is usually accomplished in acid baths which also serve to neutralize the residual alkaline film from alkaline cleaning. Alkaline chelated derusting and cleaning solutions, alone or with sodium cyanide, used as a soak or electrocleaner, are often preferred before electroplating on ferrous alloys.

1.4 Invariably several stages are necessary to provide adequate cleaning. These stages are discussed in three parts:

   Part I—Precleaning (use of a solvent, emulsion, or alkaline spray) to remove the bulk of the soil.

   Part II—Intermediate (alkaline) cleaning.

   Part III—Final electrocleaning, to remove trace solids and especially adherent impurities.

   Part IV—Trouble shooting.

Often, depending largely on the amount and type of soil on the workpieces as received, one or more of these stages may be eliminated or modified. Usually, even with light soils, it is advisable to retain multistage cleaning, thereby increasing the life and efficiency of the cleaning solutions.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. (For more specific safety precautionary statements see Sections 11 and 16.)

2. Significance and Use

2.1 The performance and quality of electroplated articles depend upon the surface cleanliness and condition. Various metals are electroplated for decorative or engineering finishes. The common electroplates applied are usually copper, nickel, and chromium for decorative and functional uses. Electroplated articles are used in many industries such as the marine, automotive, plumbing fixtures, and appliance industries.

3. Nature of the Soil

3.1 Some of the soils commonly encountered in electroplating are:

   3.1.1 Solid buffing compounds containing waxes, fatty acids, and abrasives.

   3.1.2 Liquid buffing compounds.

   3.1.3 Drawing and stamping compounds including those containing fillers (pigments).

   3.1.4 Machining oils.

   3.1.5 Rust-preventive slushing oils or greases.

   3.1.6 Electroplater’s stop-off residues.

   3.1.7 Fingerprints.

   3.1.8 Dry dirt from storage or dry pickling smut formed during derusting by pickling.

   3.1.9 Rust or oxide scales, especially admixed with oil, including heat-treat scales after oil quenching.

   3.1.10 Phosphate coating with or without lubricant.
3.1.11 Smut resulting from improper vapor degreasing of heavily buffed work.
3.1.12 Smut resulting from annealing parts without pre-cleaning between drawing operations.
3.1.13 Heat-treating salts, with or without quenching oils.
3.2 Consideration should be given to control of the soil. For example, efforts should be made to avoid overbuffing, leaving excessive compound on the work, or aging of the compound on the part before cleaning. Substitution of liquid for solid buffing compound, if work permits, often gives easier cleaning, if properly applied, but may require use of a different type of cleaner. Drawing compounds with polymerizing oils or white lead pigment are to be avoided because of difficulty in cleaning. Additives for lubricating and sulfurized cutting oils are chosen for their ability to adhebe tenaciously and are difficult to remove. Prolonged storage or drying of emulsion drawing compounds after metal working should be avoided so that slimy water-in-oil emulsions do not form. In-process cleaning or even a hot-water flush before storage is helpful. Emulsion machining lubricants (soluble oils) should be used in place of sulfurized cutting oils if operations permit. Lower-viscosity machining and rust-preventive oils are more easily removed. Stop-off materials, when used, should be applied carefully in order to avoid contaminating significant surfaces. The use of clean gloves should be mandatory after buffing or polishing to avoid fingerprints on the work. Airborne contaminants can be avoided by using covers over stored work. It is desirable to perform a cleaning operation as soon as possible after metal forming, polishing, or buffing to reduce the demands on subsequent cleaning operations, because many soils are more easily removed when fresh.

4. Metal
4.1 The properties of the metal and the method of fabrication and handling of parts play a role in cleaning. The softness and surface finish of the metal are factors in selecting handling methods. The chemical activity of the metal is an important and determining factor in cleaner selection. Aluminum requires care to avoid overetching in alkaline cleaners; both aluminum and zinc are sensitive to pitting attack, zinc and brass to tarnishing. Zinc die castings have surfaces that require special care because of sensitivity to attack by cleaning solutions. If possible, design of parts should avoid small indentations that tend to trap solid particles or buffing compositions. With die castings, care must be exercised to avoid cutting through the surface by excessive buffing. The subsurface is usually more sensitive than the “skin” of the casting. Some surface defects may not show up until cleaning and electroplating cycles are completed.

5. Cleaner
5.1 It is essential that proper cleaners and operational conditions be selected. Attention should be given to proper procurement since, even in the same category, not all cleaners are equally effective. A cleaner may be very effective for one group of soils, yet poor for other soils. This is true of electrocleaners as well as soak or spray cleaners. Soil, type of water, available time, rinsing facilities, type of metal, heating, and agitation available, facilities for disposal of cleaner, and type of personnel and equipment all influence the selection of cleaners. Obviously, economics must be considered but an initial or per pound cost must be balanced against other factors.
5.2 Cleaners do not work effectively indefinitely. The effective life of a cleaning bath must be estimated and baths discarded when exhausted. Bath life is influenced by some of the factors mentioned above as well as by the volume of work processed. The concentration of the cleaner should be controlled by analysis at regular intervals.

6. Rinses
6.1 Water hardness, acidity or alkalinity, and impurities are important factors in rinsing (1). Distilled or demineralized water is preferred where impurities in rinse water must be kept to a minimum. Boiler condensate may also be used advantageously. If the plant conditions water for acidity or alkalinity care must be taken to be sure the solids content is not too high (Note 1). Impurities derived from processing cannot be ignored; that is, rinse waters must be changed frequently or overfl owed continuously (Note 2). Counterflowing rinses are a distinct advantage in obtaining good rinsing with economical use of water.

NOTE 1—Boiler waters which contain cationic corrosion inhibitors may be quite detrimental to the plating process.

NOTE 2—Floating oil on water can cause poor adhesion.

7. Equipment
7.1 It is important to provide enough room in the plant for an adequate cleaning cycle. A discussion of equipment is beyond the scope of this practice (2, 3).

8. Criteria of Cleanliness
8.1 This subject has been treated exhaustively in the literature (4). The atomizer test is the most sensitive one, but the water-break test is most commonly used. This involves visual observation after a final rinse in clear, cool water. A continuous sheet of water on the part usually indicates a clean surface. (Certain precious-metal surfaces, such as gold, may exhibit water break, even though clean.) Some experience is necessary to judge the appearance of a break in the film of water. A specific drainage time, about 30 s, should be used before observation.
8.2 A dip in clean, dilute acid and reexamination are desirable to avoid false water-film continuity due to adsorbed soaps. Other methods, including electroplating and testing of the electroplate, should be used occasionally to confirm visual examination. (One procedure involves scrubbing with pumice and then comparing the surface produced by this method with that produced under production conditions.)

PART I—PRECLEANING

9. Purpose
9.1 Pre-cleaning is designed to remove a large excess of soil, especially deposits of buffing compound or grease. It is also

2 The boldface numbers in parentheses refer to the reports and papers appearing in the list of references at the end of this practice.
useful in reducing the viscosity of waxes and heavy oils, to enable later cleaning stages to be more effective, or to surround fingerprints and dry dust with an oily matrix to facilitate removal by alkaline cleaners.

10. Types

10.1 Cold solvent, vapor degreasing, emulsifiable solvent, solvent emulsion spray, invert-type emulsion cleaners, or hot alkaline spray with or without solvent emulsion can be used (5).

10.2 Cold Solvent (6)—Mineral spirits; trichloroethylene; perchloroethylene; 1,1,1-trichloroethane (methylchloroform); methylene chloride; or trichlorotrifluoroethane can be used for cold cleaning. Combining these with hand brushing is excellent but does not lend itself to production conditions. On the other hand, simple dipping in solvent is frequently ineffective. The chlorinated solvents are very effective for many soils, but not as effective in removing soap-based or other solvent-insoluble soils. Before electroplating, cold cleaning with solvents must be followed by additional cleaning such as alkaline cleaning to remove slight oily residues.

10.3 Vapor Degreasing (7)—Trichloroethylene and, to a lesser extent, perchloroethylene, trichlorotrifluoroethane, and methylene chloride are used for vapor degreasing. In vapor degreasing, the work is usually sprayed with clean solvent or given a thorough immersion in boiling or warm solvent for mechanical removal of tenacious soil or solids. This is followed by immersion in cold solvent to cool the parts. Then follows exposure to condensation of hot, clean solvent vapors on the work. This final step also removes any last traces of oil and grease and dries the part. For removal of caked-on oils and compounds, a predip in cool solvent can be used to wet and loosen the soil before the degreasing operation.

10.3.1 Vapor degreasing can be used to clean all types of metal, including steel, steel alloys, light metal alloys, special bronze, nonferrous metals, nickel, and titanium. This method simplifies the cleaning of parts containing several metals because it cleans by solvent action instead of chemical action; there is no danger of over-cleaning or under-cleaning because of any difference in chemical activity of the metals present. Because of the rapid penetrating action of the solvent and solvent vapor, this method is effective in cleaning parts containing recesses, blind holes, perforations, crevices, and welded seams. Where the soils are present on surfaces that are not readily accessible, the process is sometimes supplemented by ultrasonic cleaning in the solvent rinse chamber.

10.3.2 Vapor degreasing is effective on solvent-soluble soils and chemically active lubricants. Insoluble soils (buffing grits, metal chips and dust, etc.) are flushed away as the soluble soils (greases and oils) dissolve in the solvent. It is not effective on metallic salts, scale, carbon deposits, many inorganic soldering or welding fluxes, and fingerprints unaccompanied by oil or grease. This process is frequently competitive in cost with wet cleaning methods. Its lower equipment, floor space, and heat requirements offset the higher cost of solvent.

10.3.3 For some applications (steel stampings, buffed zinc-base die castings, etc.), the degreased work can go directly to mild electrolytic cleaning and subsequent electroplating without the need for an intermediate alkaline cleaning step.

10.4 Emulsion Cleaners—Oils and high-boiling hydrocarbons such as kerosene have the ability to dissolve most greases, particularly at high temperatures. The addition of emulsifiers, soaps, and wetting agents enhances the penetrating power of the organic solvent and permits removal of the latter and associated soil by power flushing. Further, intimate contact of the metal surface with the aqueous phase permits removal of materials not soluble in the hydrocarbon phase.

10.4.1 The principle of emulsion cleaning can be applied in a variety of ways including the use of straight emulsifiable solvents, unstable emulsions (diphase cleaners), invert-type emulsion cleaners, and stable emulsions. Additions of rust inhibitors or of alkali cleaners can be made to the water phase. Since agitation is important to good cleaning, the power-spray cleaners find wide applications.

10.4.2 Emulsion cleaners are used at temperatures up to 82°C. The higher temperatures remove soils more quickly and effectively, but caution must be used with cleaners containing organics of low flash point. Some cleaners containing chlorinated solvents are used above the flash point of some of the components since the chlorinated portion will volatilize to extinguish flashes.

10.5 Biological Cleaners (14)—Highly emulsifying soak cleaners are combined with living microorganisms to permit the removed oils, greases, and other complex organic compounds to undergo a natural process known as bioremediation. Living microbes break down organic compounds, such as oil and grease into carbon dioxide and water and the cleaners, if properly maintained, may run for years without changing the bath at all. Since the cleaning fluid is kept free from contaminants, the such systems allow more effective cleaning for a greater length of time.

10.5.1 In order to maintain a healthy biosystem, operating conditions are critical. Typically, optimum pH range for these types of cleaning systems is 8.5 to 9.5. Too high a pH will result in lowering of the bacteria action, and oil will be built-up. Too low a pH will render the bacteria too active resulting in consumption of the wetter and other organics necessary for proper cleaning. Temperature also is a critical operating parameter. Optimum metabolism of oil and grease is achieved around 40 – 50°C.

10.5.2 Air agitation is critical to maintaining an oxygenated environment to maintain sufficient biological activity and only aerobic bacteria. Without air during operation, anaerobic bacteria are produced and the cleaner will take on a noticeable, unpleasant odor. Air sparging also improves overall cleaning efficiency by promoting transfer of oil and grease particles from part surface into the cleaning solution. In order for bioremediation to proceed, particles must be detached from the part surface.

11. Precautions

11.1 The use of solvents and emulsions of diphase cleaners requires special attention to safety hazards. Petroleum and aromatic solvents of low flash point, for example less the 55°C, must be used with caution. Underwriters Laboratories-approved containers and adequate ventilation should be provided to avoid the accumulation of fumes in explosive concentrations. Diluted emulsion cleaners usually have flash points
above 70°C and emulsifiable solvents of high flash point are now available.

11.2 Trichloroethylene and perchloroethylene are nonflammable under the conditions of the vapor degreasing process and are among the least toxic of the chlorinated hydrocarbons; up to 100 ppm of either is tolerable in the working atmosphere for a normal 8-h working day. Trichlorotrifluoroethane (1000 ppm tolerable limit) is also used. With proper equipment design and operation, solvent vapors in the working area are easily maintained well below recommended safe limits. Degreaser operation, solvent vapors in the working area are easily tolerable limit) is also used. With proper equipment design and operation, solvent vapors in the working area are easily maintained well below recommended safe limits. Degreaser tanks should preferably be cleaned and maintained from outside the tank. Entry into a tank should be made only after all tanks should preferably be cleaned and maintained from outside the tank. Entry into a tank should be made only after all outside the tank. Entry into a tank should be made only after all solvent and vapors have been removed and then only with an observer on the outside. Proper ventilation cannot be overstressed because workmen will often discard a recommended gas mask. For cold-solvent operations, adequate ventilation must be provided in the work area.

11.3 Because soils accumulate in solvents, the solvents must be discarded or purified by distillation. In vapor degreasing equipment, the solvent is recovered by distillation and the soil discarded. The use of automatic auxiliary stills in conjunction with the degreaser allows continuous cleaning operation and solvent recovery.

11.4 Emulsifiable solvents must be discarded occasionally, although frequently most of the soil is flushed off in the rinse. Emulsion cleaners represent a particular problem of bath contamination because of the lack of adequate analytical controls to determine bath life. Because emulsion cleaners yield a water-shedding surface, the effect on water-break due to accumulated oils is difficult to differentiate from that due to the solvent. Because of the low cost of diluted emulsion cleaners, it is economical to discard these baths at frequent intervals. Soap-base emulsion cleaners can cause difficulties where acidic soils are introduced; here mixed alkalies and emulsion cleaners can also require water conditioning in hard-water areas to avoid precipitation of hard-water soaps. Good housekeeping is desirable to avoid bacterial contamination of emulsion cleaners. Bacteriostats can be included in the formulations of cleaners to prevent the unpleasant odors that result from bacterial action.

11.5 As indicated in 14.7.8, disposal of emulsion cleaners can present problems.

**PART II—INTERMEDIATE (ALKALINE) CLEANING**

12. **Purpose**

12.1 Intermediate alkaline cleaning removes solvent residues and residual soil which has been softened or conditioned by precleaning. Spray or soak alkaline cleaning also can be used as a precleaning stage followed by additional alkaline cleaning, if the soil and metal lend themselves to this treatment. This is not so for metals that are sensitive to alkaline cleaning, such as zinc, because the time in the alkaline cleaner should be minimal. Some electroplaters use the term precleaning for alkaline cleaning before electrocleaning, especially when solvent cleaning is carried out at a different part of the plant.

12.2 Although industrial practice is limited, vapor degreasing alone is sometimes used before electrocleaning. Most oils and greases and some buffing and drawing compounds are effectively removed and contamination of the electrocleaning bath is lessened. The specific applications will not be given in detail here (5, 6, 7). Manufacturers of degreasing solvent or equipment should be consulted for details.

13. **Types**

13.1 Soak alkaline cleaning is carried out at 30 to 120 g/L of alkaline cleaner at temperatures of 82°C to boiling, for periods of 3 to 15 min. If used ultrasonically, temperatures may be 70°C to boiling. The cleaners usually contain surface-active, soap-like materials which foam if agitated vigorously.

13.2 Spray alkaline cleaning is usually carried out at 4.0 to 15 g/L at temperatures of 50 to 82°C for 1 to 3 min with spray pressures of 69 to 345 kPa (10 to 50 psi). Foaming may be a problem, unless the cleaner is properly designed.

13.2.1 Foam is also a major problem because of accumulation of soaps in the cleaner from the action of the alkali on some organic soils and drag-in of wetters from precleaners. For this reason, it frequently is desirable to use low concentrations of cleaner, for example, 4.0 g/L, and to discard the solution often, even though cleaning is adequate. For the same reason, it is sometimes necessary to operate at lower pressures even though higher pressures give better cleaning.

13.3 Barrel alkaline cleaning is usually carried out at 7.5 to 45 g/L. Temperatures are usually lower than for soak cleaning because of mechanical factors. Although agitation is better than in some cleaning, control is frequently not as good.

14. **Factors Influencing Good Alkaline Cleaning**

14.1 **Concentration**—The optimum concentration of the cleaning solution should be determined by actual tests because many factors are involved.

14.2 **Temperature**—Best results are obtained near the boiling point if other conditions permit. The high temperature reduces the viscosity of the soil. A rolling boil provides agitation. In some cases, cleaner formulation may be such as to make lower temperatures optimal.

14.3 **Time**—Alkaline cleaners, operating by the mechanism of lifting the oil film, require a reasonable time to permit the surface-active materials to act on the surface. This time is shortened if agitation is vigorous, temperatures high, and concentration high. Age of solution and contamination retard cleaning.

14.4 **Agitation**:

14.4.1 **Spray Cleaning** (2)—As in emulsion cleaning, much of the effectiveness of spray cleaners in removal of solids is due to the mechanical action of the solution sprayed on the surface. Hence, every effort should be made to obtain efficient impingement at high pressure without pushing the work out of the spray area (or off the racks). Foaming after soap accumulation also limits spray pressure. The action of spray alkaline cleaners depends on detergency as well as mechanical action; the proper materials and conditions should be used. Spray nozzles should not be allowed to clog with solids and vesti- bules should be provided at each stage of the machine to avoid contamination by overspray. Agitation is very good in spray cleaning (if the sprays hit the solid surfaces directly).

14.4.2 **Soak Cleaning** (3)—Suggested methods of agitation in soak cleaning are as follows:
14.4.2.1 Complete withdrawal and reimmersion, especially at about half the total time allotted in the cleaner,
14.4.2.2 Pumping the solution over the work with proper intake and adequate pressure,
14.4.2.3 Rapid motion of the work in the cleaner,
14.4.2.4 Maintenance of a rolling boil,
14.4.2.5 Use of an air line or a mixer,
14.4.2.6 Ultrasonic agitation, and
14.4.2.7 Use of an overflow weir assists in separating unemulsified oils, grease, and other solids.

Note: Electrocleaning represents a special method of agitation and is considered in 15.4.

14.5 Age and Degree of Contamination—A long bath life is desirable, not only for economy but also to avoid the necessity for constant supervision. Alkaline cleaners vary in this respect; much depends on the amount of soil present on the work being cleaned. Care must be taken to avoid redeposition of the soil as parts exit from the cleaner. Cleaners should be discarded periodically because of accumulation of debris, etc. Surface oil should be skimmed frequently, preferably by overflowing into a properly designed skim trough. The cleaner should be buffered to have a high tolerance for acidic soils. Tolerance for soaps, formed by reaction with the soils, can be designed into the cleaner when necessary.

14.6 Rinsing (see Section 6)—Double rinses are desirable to reduce the concentration of cleaner in the rinse. A first hot rinse gives more efficient cleaner removal, while a second cold rinse reduces the tendency to rusting and tarnishing during transfer to the next stage. If rinsing is inadequate, the components of the cleaner must be selected to promote easy rinsing. Selection of a free-rinsing cleaner may require some sacrifice in cleaning properties. Rinse tanks should have adequate overflow rate, skimming troughs of good design, and proper positioning of the intake water line.

14.6.1 Agitation of work in the rinse tank is desirable. Water must also flow through hot rinses, although a reduced rate is often desired for economy. Spray rinsing is very effective, especially if coupled with soak rinsing by having a spray of clean water hit the work as it exits from the rinse tank. Drying of the cleaner on the work before rinsing should be avoided by having fog nozzles after cleaning tanks, if necessary, or by decreasing transfer time or operating temperatures. With some cleaners, adequate rinsing is difficult to obtain after the cleaner has dried on the work.

14.7 Selection of the Cleaner—Important factors to be considered are as follows:
14.7.1 Soil (see 3.1 and 3.2)—If the soil is rich in soaps, the cleaner should be lean in this respect and should have built-in high soap tolerance. Because of the specific action of the individual cleaners on certain soils, this is a prime factor in cleaner selection.
14.7.2 Metal (see 4.1)—Special cleaners are designed to avoid harmful effects on zinc, aluminum, and brasses. Iron, steel, magnesium, and copper have better tolerance to alkaline cleaners.
14.7.3 Water—Hard water frequently requires cleaners rich in water softeners, such as sequestering and chelating agents, largely to prevent difficulties during rinsing.

14.7.4 Method of Application (4)—There is no universal cleaner that can be efficiently applied by all methods of cleaning. The cleaner should be formulated for its method of application. Cooperation of purchaser with vendor in permitting study of equipment, and availability of heating facilities will minimize problems such as incomplete cleaning, excessive foaming, dry down of cleaning solution before rinsing, etc.

14.7.5 Degree of Cleanliness Required (5)—The degree of cleanliness required for the next operation should be determined by the purchaser and explained to the vendor. If parts are to be held over between operations, a light oil or alkaline protective film may be advantageous to prevent rusting. Parts going immediately to electroplating should be free from any objectionable films that might prevent adhesion of electrodeposits.

14.7.6 Unit Cost—Many factors enter into the cost of a cleaning operation. Among them are: yearly cost of space occupied by the equipment, capital costs and amortization of equipment, utilities (water, heat, power, etc.), maintenance by laboratory control, make-up, additions, and disposal of spent solutions, labor, cost of cleaning compounds, and cost of reprocessing rejects. To obtain true costs per thousand square feet of work processed all these factors must be included.

14.7.7 Safety—Consideration must be given to the safety of operators and equipment and parts being cleaned (see 11.1, 11.2, 11.3, and Section 16).

14.7.8 Disposal of Spent Solutions and Rinse Waters—Local, state, and Federal regulations must be consulted before final selection of a cleaning material can be made. Increasing attention to reduction of stream pollution throughout the country means that this factor is a more decisive one in selection of cleaning materials. Examples of materials that come under such regulations are: chromates, cyanides, nonbio-degradable detergents, phenolics, spent solvent emulsions, and phosphates.

15. Formulations of Alkaline Cleaners (8)

15.1 General—Actual compositions of cleaning materials for various metals, soils, methods of application, degree of cleanliness required, and other specific conditions are beyond the scope of this document. The fact that individual suppliers of proprietary cleaning materials have hundreds of standard products available indicates the complexity of this field. In like manner, the recommendations for their application vary widely. Some requirements for formulations of cleaning materials are listed below:

15.2 Soak Cleaners:
15.2.1 Ability to saponify animal and vegetable oils, if conditions such as paint stripping require it on steel parts. Otherwise, this requirement can be undesirable where sensitive metals are involved, because attack on the metal may occur thereby.
15.2.2 Wetting and emulsifying action supplied by soaps or synthetic surface agents to wet oily and greasy deposits and, with agitation, suspend the deposits in an emulsified state until rinsing is accomplished.
15.2.3 Deflocculating action or colloidal properties to attract solid particle soils and suspend them in a free rinsing condition, so that redeposition of the soil is prevented.
15.2.4 Water-softening ability is required to prevent formation of insoluble soaps by combination of hard-water salts with soap films formed by reaction of soils and alkaline constituents. Such insoluble soaps adhering to surfaces decompose in subsequent acid dips and leave objectionable films of fatty acids on parts to be electroplated. The sequestering polyphosphates are used for this purpose where operating temperatures do not exceed their decomposition range, about 80°C. The organic chelating agents are stable and offer another advantage over the polyphosphates in that they solubilize light oxide films on the metal parts and provide a more active surface for subsequent electroplating.

15.4.5 Buffering action is required in electrocleaners as in other types to maintain the optimum pH range, despite introduction of acidic soils.

15.4.6 Inhibition to prevent attack of sensitive metals is required for the undesirable condition where a single electrocleaning solution is used as a precleaner and final cleaner. On ferrous alloys no inhibition is required. Where sensitive metals have been precleaned, a slight attack or oxidation of the surface, when anodically cleaned, is considered beneficial. This action, plus subsequent acid dip, before electroplating, removes disturbed metal surfaces and promotes better adhesion.

16. Precautions (9)

16.1 Some alkaline cleaners generate considerable heat when dissolved in water. Rate of solution, however, is frequently very slow in cold water. It is most advantageous to have the water at a temperature of about 50°C while the cleaner is carefully sprinkled in small quantities over the surface of the tank, as with a shovel. Others prefer the safer but slower process of adding the cleaner directly to the tank of water at room temperature with provision for agitation to avoid caking. A caked cleaner dissolves slowly. If alkali gets on the skin, it should be washed profusely with water and medical attention received promptly.

PART III—FINAL ELECTROCLEANING

17. Purpose

17.1 The objectives of metal cleaning before electroplating have been summarized as follows (10):” In electrodeposition a surface is required which will receive a smooth, adherent metal deposit, but this is not necessarily an absolutely clean surface. In general, an acceptable surface is one on which objectionable surface films have been replaced by films more suitable and acceptable for electroplating.”

17.2 Assuming that the metal parts have been precleaned by methods described above, the “objectionable surface films” are precleaner residues, minor amounts of soils such as oils and solid particles not completely removed by previous cleaning treatments. Thus, the final electrocleaning process is insurance that only films remain that are not more suitable and acceptable” for the electroplating operations.

18. Types of Electrocleaning

18.1 Electrocleaning is soak cleaning with agitation provided by the upward movement of bubbles of hydrogen or
oxygen formed by the electrolytic decomposition of water in the solution. Because of this electrolytic action special types of cleaners are required (see 15.4).

18.2 Cathodic—Parts negatively charged in the electrocleaner tank are cathodically or direct current cleaned. Hydrogen gas is evolved on the surface. Positively charged ions and colloids are attracted to the cathode.

18.2.1 Cathodic cleaning provides greater agitation because two volumes of hydrogen are evolved on the surface, as compared with one volume of oxygen at the anode. Anodic cleaning, little or no tarnish or attack of nonferrous metal occurs.

18.2.2 Cathodic cleaning attracts positively charged metallic ions, soaps, and other colloidal materials in the solution causing them to “deposit out” as loose smuts on parts being cleaned. Since hydrogen is evolved on the parts it may penetrate and become occluded in hardened steel parts, causing embrittlement. Cathodic electrocleaning solutions are more sensitive to chromic acid contamination than anodic electrocleaning solutions.

18.3 Anodic—Parts positively charged in the electrocleaner solution are anodically or reverse current cleaned. Oxygen gas is evolved on the surface. Negatively charged ions and colloids are attracted to the parts.

18.3.1 A higher degree of cleanliness is obtained owing to the “deplating” action resulting from the positively charged surface repelling positively charged metallic ions, soils, and smuts. Oxygen gas does not enter metals as hydrogen does. Anodic electrocleaning solutions have much more tolerance for chromic acid contamination.

18.3.2 Less agitation occurs at the anode surface because only one volume of oxygen gas is evolved from the decomposition of the water.

NOTE 5—Nonferrous metals are tarnished or attacked if anodic cleaning is prolonged in an unhindered electrocleaning solution. However, this action may be beneficial as discussed in 15.4.6. Alloys of lead, nickel, or silver and nickel, and silver electroplated surfaces should not be anodically cleaned. Lead alloys are rapidly attacked and nickel and silver surfaces become passivated, requiring special activating treatments for subsequent electrodeposits.

18.4 Periodic Reverse (PR) Current Cleaning—A modification of normal electrocleaning methods is the use of a number of cycles of periodic reverse current to assist in removal of soils, instead of a single application of direct or reverse current cleaning, or a combination of them. Details of electrical equipment for production of periodic reverse current are outlined in a recent book (11). Users of PR cleaning, unlike PR electroplating, prefer a PR cycle in which the cathodic (direct-current) time equals the anodic (reverse-current) time. To prevent deposition of loose metallic smuts, the work should be removed from the electrocleaner during the deanodic portion of a cycle. PR cleaning gives improved smut removal, accelerates cleaning operations, and provides a more active surface for subsequent electroplating (12). PR cleaning improves electrolytic removal of rust and scale in alkaline chelating solutions (13).
20.2 Spray Washing Machines:
20.2.1 Heating—Check thermostats, scale on heating coils or tubes, and solution temperature. Descaling the machine, if necessary, with inhibited hydrochloric acid or a proprietary descaling chemical to improve heating efficiency.
20.2.2 Spray Risers—There should be enough spray risers, properly positioned and directed to contact all surfaces. The nozzles should be periodically cleaned.
20.2.3 Pumps and Lines—Check on the return side to be sure no air is being sucked into the system causing excessive foaming.
20.2.4 Baffles—The cleaning and rinsing stages must be properly baffled to prevent one from contaminating or diluting the other.
20.2.5 Contamination—Excessively dirty cleaning solutions cannot be expected to function properly. Grease overflow dams must function properly. Production must not be excessive before a new cleaner is made up.
20.3 Electrocleaners:
20.3.1 Current Density—Too high a current density when cleaning metals anodically may etch a buffed surface or cause excessive tarnishing. Too low a current density when electrocleaning cathodically will cause electroplating out of dissolved metals and some colloids as a smut. Too low a current density (cathodic or anodic) can be the cause of poor cleaning. Check the current with tong ammeters if available and calculate current density. Good steel electrocleaning requires a minimum of 5 A/dm²; 8 to 10 is better. Brass and zinc die castings and nickel deposits normally are cleaned at 2 to 4 A/dm².
20.3.2 Bus Bars—Be sure that the bus bars are of ample size to carry the current required for a work load (normally 158 A/cm² cross section of copper bar). Be sure that the bus bars are clean to ensure good contact.
20.3.3 Polarity—Be sure that the polarity is proper for the job. Cathodic cleaning should seldom be used as a final electrocleaning step because of the danger of “depositing out” dissolved metals. There are exceptions to this rule, as in electrocleaning lead, magnesium, nickel, silver, and some stainless steels. If doubt exists as to polarity, check with a voltmeter, or trace back the conductors to the current source.
20.3.4 Insulation—Be sure that the tank is insulated from the floor, and that the work, electrode rods, and electrodes are insulated from the tank. Insulated joints on steam, drain, and water lines should be provided.
20.4 Rinses—Rinses should be kept clean and overflowing. Some cleaners require warm rinses for maximum efficiency. The cleaner supplier’s instructions should be followed. It is poor practice to use a common rinse tank for several purposes, such as following a soak cleaner, electrocleaner, and an acid dip. Air agitation in rinses is beneficial.

21. Failures Attributable to Cleaning

Note 6—There are a number of other reasons for these failures, not associated with cleaning.

21.1 Blisters, Peeling, or Poor Adhesion—These defects may be caused by improper polarity in the electrocleaner, overcleaning (current density too high or cleaning time too long), no current in the electrocleaner, and oil and grease not completely removed. In certain circumstances, especially with materials used in the electronic industry, such electroplating defects may arise from other factors as well.

21.2 Pitting—Pitting may be due to oil and grease not being completely removed. If pitting is general and on all parts, the trouble is probably in the electroplating tanks.

21.3 Strains—This is a problem associated with bright-electroplated work. The most common cause is cleaner drying on the work during transfer. It can be eliminated by lowering the cleaner temperature, decreasing transfer time, installing fog nozzles to keep parts wet until they reach rinse tanks, or by using cleaners designed to prevent staining of sensitive nonferrous metals. It also can be caused by incomplete soil removal, particularly tightly adherent oils of a polar type almost impossible to remove in alkaline cleaners. Solvent degreasing or emulsifiable solvent predips may be required.

21.4 Roughness—This is caused by failure to remove smut or other solid particles. Sometimes this can be traced back to vapor degreasing which removes the oil but not the solid particles. If this is a cause, a high-pressure solvent spray will often help. Roughness also may be caused by incomplete rinsing of alkaline soak or electrocleaners due to excessive water hardness or an exhausted cleaner bath. Magnetically charged particles can cause roughness on electroplated steel parts.
REFERENCES

(6) ASTM Committee D-26 *Cold Cleaning with Halogenated Solvents, ASTM STP 403*, ASTM, Philadelphia, PA 19103.

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Standard Test Method for Density, Oil Content, and Interconnected Porosity of Sintered Metal Structural Parts and Oil-Impregnated Bearings


2 Annual Book of ASTM Standards, Vol 02.05.

3 Annual Book of ASTM Standards, Vol 05.01.

1 This standard is issued under the fixed designation B 328; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers determination of the density, oil content, and interconnected porosity of sintered bearings and structural parts with or without oil impregnation.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 243 Terminology of Powder Metallurgy
D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer
D 1298 Test Method for Density, Relative Density (Specific Gravity), or API Gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method

3. Terminology

3.1 Definitions of powder metallurgy (P/M) terms can be found in Terminology B 243. Additional descriptive information is available in the Related Material section of Vol 02.05 of the Annual Book of ASTM Standards.

4. Significance and Use

4.1 The volume of an arbitrary P/M shape cannot be accurately measured by standard techniques such as by micrometers or calipers. Since density is mass/volume, a precise method to measure the volume is needed. For nonporous objects, the volume of water displaced by the immersed object is determined by Archimedes principle. For porous P/M parts, a method is required to seal surface connected pores. If the pores are not sealed or the part is not oil impregnated, the part will absorb some of the water and decrease its buoyancy and exhibit an erroneously high density.

4.2 Density and oil content values are generally contained in the specifications for oil-impregnated bearings and other self-lubricating P/M parts. Desired lubrication requires sufficient interconnected porosity and satisfactory oil impregnation of the porosity.

4.3 For a particular P/M material, the mechanical properties of P/M structural parts are directly related to their density. Density values are therefore generally contained in the specifications for P/M parts.

5. Apparatus

5.1 Analytical Balance, of sufficient capacity and accurate to 0.01 % of the test specimen mass.

5.2 Device for weighing the test piece in air and in liquid (water); the water is distilled or deionized and preferably degassed. A wetting agent is added to the water, 0.05 to 0.1 % by weight, to reduce surface tension effects.

5.3 Soxhlet Extractor, with oil solvent. Extractors may be purchased from most laboratory supply companies.

5.4 Apparatus for vacuum impregnation of the test piece with oil.

5.5 Beaker and Wires, of various sizes. A wire basket may be used in place of wires (see Figs. 1 and 2).

5.6 Thermometer—Capable of reading temperature in the range of 10 to 38°C (50 to 100°F) to an accuracy of 0.5°C (1°F).

4 Kodak Photo-Flo 200, available from Momentum Graphics, 400 D. Pierce St., Somerset, NJ 08873, or its equivalent, has been found suitable.

5 Extractors may be purchased from Fisher Scientific, 585 Alpha Drive, Pittsburgh, PA 15238; Cole-Parmer, 7425 North Oak Ave., Niles, IL 60714; V.W.R., P.O. Box 15646, Philadelphia, PA 19105-5645; or Thomas Scientific, P.O. Box 99, Swedesboro, NJ 08085-6099.
5.7 Lubricant, of 20 to 65 cSt (100 to 300 SSU) at 38°C (100°F).

6. Test Specimen

6.1 The specimen mass shall be a minimum of 1.0 g. Several specimens may be used to reach the minimum mass.

7. Procedure

7.1 Using an analytical balance, obtain the mass of the as received oil-containing specimen (Mass J), the oil-free specimen (Mass A), and the fully impregnated specimen (Mass B). These, and all subsequent weighing, should be to 0.01 % of the mass of the part, for example:

<table>
<thead>
<tr>
<th>Specimen Mass, g</th>
<th>Balance Sensitivity, g</th>
</tr>
</thead>
<tbody>
<tr>
<td>less than 10</td>
<td>0.0001</td>
</tr>
<tr>
<td>10 to less than 100</td>
<td>0.001</td>
</tr>
<tr>
<td>100 to less than 1000</td>
<td>0.01</td>
</tr>
<tr>
<td>1000 to less than 10 000</td>
<td>0.1</td>
</tr>
</tbody>
</table>

7.2 To determine Mass A, remove any oil from the samples by extracting it in a Soxhlet apparatus of suitable size using toluol or petroleum ether as a solvent. After extracting for approximately 1 h, remove the residual solvent by heating samples 1 h at 120°C (250°F) and weigh upon cooling. Continue alternate extractions and drying until the dry mass in air is constant to 0.05 % of the mass of the part.

7.2.1 For large parts or for a faster method, but not as accurate and with no concern for subsequent metallurgical properties, the oil can be removed by heating the specimen in a protective atmosphere in the temperature range of 430 to 650°C (800 to 1200°F). This method may be used if agreed upon by both parties. The selection of a proper burnout temperature may be critical in the case of sintered 90/10 Cu/Sn materials, depending upon the sintering temperature and time used by the fabricator. The normal sintering temperature range for bronze is 815 to 870°C (1500 to 1600°F), depending on the desired shrinkage, strength, and porosity relationships. This method is also applicable to sintered aluminum materials if the temperature does not exceed 540°C (1000°F).

7.3 For the purpose of determining the mass of oil-impregnated specimens in air (Mass B) or in water (Mass C), either of the following two methods may be used to impregnate the test specimen. The vacuum method is preferred.

7.3.1 At room temperature, using a suitable evacuating method, reduce the pressure over the immersed specimen to not more than 7 kPa (2 in. mercury) pressure for 30 min. Then permit the pressure to increase to atmospheric pressure and the specimen to remain immersed in oil 20 to 65 cSt (100 to 300 SSU) 38°C (100°F) at room temperature and pressure for 10 min.

7.3.2 Immerse the specimen in oil, viscosity of 20 to 65 cSt (100 to 300 SSU) at 38°C (100°F), hold at a temperature of 82°C ± 5°C (180°F ± 10°F) for at least 4 h, and then cool to room temperature by immersion in oil at room temperature.

7.4 To weigh the specimen in water, select a fine wire for supporting the specimen. Suspend the wire from the beam hook, while the specimen is immersed in a beaker of distilled water. Support the beaker of water over the pan of the balance, using a suitable bridge. The container of water may also be supported below the balance for weighing specimens if the balance has a lower beam hook for this purpose. See Fig. 2b. Use a wetting agent (in the amount of 0.05 to 0.1 % by weight) to reduce the effects of surface tension. The recommended diameter of wire (copper or stainless steel) to be used for various weight range is as follows:

- less than 50 g – 0.12 mm (0.005 in.)
- 50 to less than 200 g – 0.25 mm (0.010 in.)
- 200 to less than 600 g – 0.40 mm (0.015 in.)
- 600 g and greater – 0.50 mm (0.020 in.)

In place of attaching the specimen on a wire, the use of a wire basket suspended in water may be used as an alternate method (see Fig. 1b).

7.5 Twist the wire around the specimen and suspend it from the beam hook so that the specimen is completely immersed in the water. The water should cover the specimen by at least 6 mm (0.25 in.) and the wire twist should be completely submerged. Immersion should be to the same point each time. Take care to ensure that no air bubbles adhere to the specimen or to the wire. If a wire basket is used as an alternate method, completely immerse the wire basket in the water.

7.6 Weigh the specimen and wire in water. This is Mass C. If a wire basket is used as an alternate method, weigh the specimen and wire basket in water.

7.7 Remove the specimen and reweigh the wire in water immersed to the same point as before. This is Mass E. Some balances are capable of being tared automatically, which eliminates the need for reweighing the wire for correction. The mass of the oil impregnated specimen in water with the mass of wire tared is Mass F. Excess oil should be removed from the surface of the specimen before weighing. Care should be taken not to remove oil from the porosity of the part. Water density, Dw, is found in Table 1. Measure the temperature of the water to the nearest 1°C.

7.7.1 If a wire basket is used as the alternate method, follow the same procedure but substitute the basket for the wire.

7.8 For oil content and interconnected porosity, determine the density of the impregnant, which is (D_o). Oil content is defined as the percent oil content by volume in the part as received. The interconnected porosity is the percent oil content by volume in the part as it is impregnated under specific laboratory conditions.

Note: 1—Typical density of petroleum-type lubricants is 0.880 g/cm³ and for synthetic lubricants it ranges from 0.910 to 1.000 g/cm³. Refer to
Test Method D 1217 or Practice D 1298 for determining the density of the lubricants.

8. Calculation

8.1 Calculate the density of structural parts using Eq 1 or Eq 2 (alternative):

\[
D = \left( \frac{A}{B - C + E} \right) D_w = \left( \frac{A}{B - (C - E)} \right) D_w
\]

\[
D = \left( \frac{A}{B - F} \right) D_w
\]

where:
- \( D \) = density, g/cm\(^3\),
- \( A \) = mass in air of oil-free specimen, g,
- \( B \) = mass of oil-impregnated specimen, g,
- \( C \) = mass of oil-impregnated specimen and wire in water, g,
- \( E \) = mass of wire in water, g,
- \( D_w \) = density of water at the immersion temp, g/cm\(^3\), and
- \( F \) = mass of oil-impregnated specimen in water, with mass of wire tared.

8.2 Calculate the wet density of bearings supplied fully impregnated with lubricant using Eq 3 or Eq 4 (alternative):

\[
D = \left( \frac{B}{B - C + E} \right) D_w
\]

\[
D = \left( \frac{B}{B - F} \right) D_w
\]

where:
- \( D \) = density, g/cm\(^3\),
- \( B \) = mass of oil-impregnated specimen, g,
$C$ = mass of oil-impregnated specimen and wire in water, g,
$E$ = mass of wire in water, g,
$D_w$ = density of water at the immersion temp, g/cm$^3$, and
$F$ = mass of oil-impregnated specimen in water, with mass of wire tared, g.

8.3 Calculate the interconnected porosity and the oil content by volume using Eq 5, Eq 6, or Eq 7 (alternative).

8.3.1 Interconnected Porosity:

$$P = \left[ \frac{B - A}{(B - C + E) \times D_o} \times 100 \right] \times D_o$$  \(5\)

$$P = \left[ \frac{B - A}{(B - (C - E)) \times D_o} \times 100 \right] \times D_w$$  \(6\)

$$P = \left[ \frac{B - A}{(B - F) \times D_o} \times 100 \right] \times D_w$$  \(7\)

where:
$P$ = interconnecting porosity by volume, %,
$A$ = mass in air of oil-free specimen, g,
$B$ = mass of oil-impregnated specimen, g,
$C$ = mass of oil-impregnated sample immersed in water, g,
$E$ = mass of wire in water, g,
$D_o$ = density of the oil, g/cm$^3$,
$F$ = mass of oil impregnated specimen in water with mass of wire tared, g, and
$D_w$ = density of water at the immersion temperature, g/cm$^3$.

8.3.2 Oil Content by Volume as Received—Use Eq 8 and 9, or Eq 10 (alternative):

$$P_1 = \left[ \frac{J - A}{(B - C + E) \times D_o} \times 100 \right] \times D_o$$  \(8\)

$$P_1 = \left[ \frac{J - A}{(B - (C - E))} \times D_o \right] \times D_w$$  \(9\)

$$P_1 = \left[ \frac{J - A}{(B - F) \times D_o} \times 100 \right] \times D_w$$  \(10\)

where:
$P_1$ = oil content by volume, as received, %,
$J$ = mass of oil-impregnated specimen as received, g,
$D_o$ = mass of oil-impregnated specimen, g,
$D_w$ = density of water at the immersion temperature, g/cm$^3$,
$A$ = mass in air of oil-free specimen, g,
$B$ = mass of oil impregnated specimen, g,
$C$ = mass of oil-impregnated sample immersed in water, g,
$E$ = mass of wire in water, g, and
$F$ = mass of oil impregnated specimen in water with mass of wire tared, g.

9. Precision and Bias

9.1 For ferrous and copper base sintered parts, the repeatability interval, $(r)$, is 0.05 g/cm$^3$ for dry or wet density. It is 2.0 percentage points for porosity. Duplicate results from the same laboratory should not be considered suspect at the 95% confidence level unless they differ by more than $(r)$.

9.2 For ferrous and copper base sintered parts, the reproducibility interval $(R)$ is 0.06 g/cm$^3$ for dry or wet density, and 4.0 percentage points for porosity. These results from two different laboratories should not be considered suspect at the 95% confidence level unless they differ by more than $(R)$.

9.3 There is no estimate of bias because there is no accepted porous reference material. A test program using fully dense silicon as a reference is being undertaken.

9.4 These data are based upon the identical sample checked at six different laboratories.

10. Keywords

10.1 density; interconnected porosity; oil content; oil-impregnated bearings; P/M; powder metallurgy; sintered metal; structural parts

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Standard Test Method for Apparent Density of Metal Powders and Compounds Using the Scott Volumeter

This standard is issued under the fixed designation B 329; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

ε1 NOTE—Figure 2 was editorially updated in June 2003.

1. Scope
1.1 This test method covers determination of the apparent density of metal powders and related compounds using the Scott Volumeter, also known as the Paint Pigment Volumeter.
1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.
1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents
2.1 ASTM Standards:
B 215 Practices for Sampling Finished Lots of Metal Powders
B 243 Terminology of Powder Metallurgy
B 873 Test Method for Measuring Volume of Apparent Density Cup Used in Test Methods B 212, B 329 and B 417

3. Terminology
3.1 Definitions—For definitions of terms used in this test method see Terminology B 243.

4. Significance and Use
4.1 This test method covers the evaluation of the apparent density physical characteristic of powders. The degree of correlation between the results of this test and the quality of powders in use will vary with each particular application and has not been fully determined.

4.2 The apparent density measured via this test method is often referred to as the “Scott Density.”

5. Apparatus
5.1 Fig. 1 shows the Scott Volumeter consisting of the following parts:
5.1.1 Top Brass Funnel—A large funnel with a 16-mesh brass screen and a small conical funnel for directing the powder into the baffle box (Note 2).
5.1.2 Baffle Box—A box with two glass sides and two wooden sides containing a series of four glass baffle plates.

Note 1—The wooden baffle box may be substituted by a water-resistant material if washing is more desirable than air cleaning.

5.1.3 Bottom Brass Funnel—A small brass funnel directly beneath lower baffle box opening for directing the powder into the density cup (Note 2).
5.1.4 Brass Density Cups—A cylindrical cup having a capacity of 25.00 ± 0.03 cm³, with an inside diameter of 28.00 ± 0.50 mm; or a square cup with a capacity of 16.39 ± 0.05 cm³ (1.000 ± 0.003 in.³). ³

Note 2—Replacement parts for 5.1.1, 5.1.3, and 5.1.4 may be of stainless steel.

5.1.5 Stand—A 90° pivoting wooden stand to support the funnels and the baffle box concentric with the density cup so that the bottom funnel lower opening is 19 mm (¾ in.) above the top of the density cup as shown in Fig. 1 when using the cubic inch cup. Fig. 2 shows some suggested modifications for use of the metric cup. Modifications A and C of Fig. 2 are suggested when the metric cup is to be used exclusively. Modification B of Fig. 2 is suggested when both cups are to be used interchangeably.

³ Apparatus may be purchased as the “Metal Powder Volumeter” (Catalog No. 66062-620) from VWR Scientific Co., 1310 Goshen Parkway, West Chester, PA 19380. The apparatus was formerly known as the “Scott, Schaeffer and White Paint Pigment Volumeter.”

4 Metric cup may be constructed or purchased from Alcan Powders and Pigments, Division of Alcan Aluminum Corp., 901 Lehigh Ave., Union, NJ 07083-7632.
5.2 Instrument Support—A stand or bench surface, level and vibration free.

5.3 Balance—A balance having a capacity of at least 200 g and an accuracy of ±0.05 g with full-range taring capability.

5.4 Brush—A good quality, 25.4-mm (1-in.) wide brush, preferably nylon.

5.5 Spatula—A standard 12.7-mm (½ -in.) wide stainless steel laboratory spatula.

6. Test Specimen

6.1 Obtain a test specimen in accordance with Practice B 215.
6.2 A minimum of 25 cm³ (1.5 in.³) of powder for the square cup and 35 cm³ (2.0 in.³) of powder for the cylindrical cup will be needed.

7. Calibration and Standardization

7.1 The density cups shall be calibrated in accordance with ASTM Test Method B 873.

8. Procedure

8.1 Tare or preweigh the density cup.

8.2 Pour the powder being tested carefully into the top receiving funnel and permit the powder to run into the density cup. Completely fill the density cup to overflowing or fill to overflowing and form a mound of powder above the top of the density cup. If the powder is not free-flowing, carefully brush the sample through the top receiving funnel screen without jarring the volumeter. Jarring of the volumeter could result in packing of the powder in the density cups, thus giving erroneously high values.

8.3 Remove the excess powder in the density cup by passing the edge of the spatula blade parallel to, and in contact with, the top of the cup. Move the spatula smoothly along the top surface of the cup and back again until all excess powder has been removed, special care being taken to direct the excess powder into the unfilled areas of the cup. It is important that the spatula be kept level at all times to prevent packing or pulling out of the powder. Perform the completed leveling operation to produce a uniform powder surface perfectly level with the top of the density cup.

8.4 After the leveling operation, lightly tap the side of the density cup to settle the powder to avoid spilling while transferring the cup to the balance for weighing.

8.5 Determine the mass of the powder to the nearest 0.1 g by weighing the cup plus powder and, if preweighed and not tared, subtracting the mass of the cup.

9. Calculation

9.1 Divide the mass of the powder by the volume of the cup to obtain the apparent density.

10. Report

10.1 Report test results as “Scott Density” or “Apparent Density, Scott” in grams per cubic centimetre to the nearest 0.1 g/cm³.

NOTE 3—If the square cup is used, multiply the result obtained in grams per cubic inch by 0.0610 for conversion to grams per cubic centimetre.

10.2 Report which density cup was used in the test.

10.3 Include a reference to this test method in the report.

11. Precision and Bias

11.1 Precision—Precision has been determined from round-robin testing performed prior to the approval of this test method. Those results, which have been re-verified, show a precision of from ±3 to 7 % of the value determined as the 2 s limits for powders with apparent densities of <1 to 5 g/cm³.

11.2 Bias—Bias cannot be stated since there is no universally accepted standard instrument, nor are there certified standards available.

12. Keywords

12.1 apparent density; bulk density; Paint Pigment Volume-meter; powder metallurgy; powders; refractory metals; Scott density; Scott Volumeter
Standard Test Method for Fisher Number of Metal Powders and Related Compounds

This standard is issued under the fixed designation B 330; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope *

1.1 This test method uses air permeability to determine an envelope-specific surface area and its associated average equivalent spherical diameter (from 1 to 50 µm) of metal powders and related compounds. The powders may be analyzed in their “as-supplied” (shipped, received, or processed) condition or after they have been de-agglomerated or milled by a laboratory procedure (“lab milled”) such as that specified in Practice B 859. The values obtained are not intended to be absolute but are generally useful on a relative basis for control purposes.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 243 Terminology of Powder Metallurgy
B 859 Practice for De-Agglomeration of Refractory Metal Powders and Their Compounds Prior to Particle Size Analysis
E 456 Terminology Relating to Quality and Statistics
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 ISO/DIS Document:

10070 Metallic Powders Determinations of Envelope-Specific Surface Area from Measurements of the Permeability to Air of a Powder Bed Under Steady-State Flow Conditions

3. Terminology

3.1 Definitions—Many terms used in this test method are defined in Terminology B 243.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 Fisher sub-sieve sizer, n—a commercially available permeability instrument for measuring envelope-specific surface area.

3.2.2 envelope-specific surface area, n—the specific surface area of a powder as determined by gas permeametry in accordance with ISO/DIS 10070.

3.2.3 air permeability, n—the measurement of air pressure drop across a packed bed of powder.

3.2.4 de-agglomeration, n—process used to break up agglomerates of particles.

3.2.5 Fisher Number, n—a calculated value equated to an average particle diameter, assuming all the particles are spherical and of uniform size.

3.2.6 Fisher calibrator tube, n—a jewel with a precision orifice mounted in a tube similar to a sample tube. The calibrator tube value is directly traceable to the master tube maintained by Fisher.

3.2.7 porosity of a bed of powder, n—the ratio of the volume of the void space in the powder bed to the that of the overall volume of the powder bed.

3.2.8 agglomerate, n—several particles adhering together.

4. Significance and Use

4.1 This test method provides a procedure for determining the envelope-specific surface area of powders, from which is calculated an “average” particle diameter, assuming the particles are monosize, smooth surface, nonporous, spherical particles. For this reason, values obtained by this test method will be defined as a Fisher Number. The degree of correlation between the results of this test method and the quality of powders in use will vary with each particular application and has not been fully determined.

4.2 This test method is generally applicable to all metal powders and related compounds, including carbides, nitrides, and oxides, for particles having diameters between 1 and 50 µm. It should not be used for powders composed of particles whose shape is too far from equiaxed, that is, flakes or fibers. In these cases, it is permissible to use the test method described only by agreement between the parties concerned. This test method shall not be used for mixtures of different powders nor for powders containing binders or lubricants. When the powder

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*A Summary of Changes section appears at the end of this standard.
contains agglomerates, the measured surface area may be affected by the degree of agglomeration. Methods of de-agglomeration such as those referenced in 1.1 may be used if agreed upon between the parties concerned.

4.3 When an "average" particle size of powders is determined using the Fisher sub-sieve sizer, it should be clearly kept in mind that this average size is derived from the determination of the specific surface area of the powder using a relationship that is true only for powders of uniform size and spherical shape.

5. Apparatus

5.1 The Fisher sub-sieve sizer consists of an air pump, an air-pressure regulating device, a precision-bore sample tube, a standardized double-range air flowmeter, and a calculator chart. Included is accessory equipment consisting of a plug manipulator, powder funnel, two porous plugs, a supply of paper disks, and a rubber tube support stand.

Note 1—Necessary replacement parts should be obtained from the manufacturer, especially in the case of the precision manometer which is a part of the air flowmeter.

5.2 The manufacturer also furnishes directions which should be followed except as amended as follows. Particular attention should be given to proper maintenance of the instrument with special reference to the instructions on (1) periodic checking of the water level in the pressure regulator standpipe, (2) manometer level before the sample tube is inserted, and (3) the sample packing assembly.

5.3 Jewel Calibrator Tube—a standard for average particle size measurement. It allows operators to relate their data to that of other analysts. Each calibrator is factory tested three times with the resulting readings and associated porosity recorded on the tube.

Note 2—Adjust the sample packing assembly (1) as described in the manufacturer’s directions with the exception that the plugs and paper disks are not inserted in the sample tube but are merely stacked together and placed between the brass support and the “flat” of the bottom of the rack of (2) as previously described except that a specially made baseline gage is used instead of the plugs and paper disks. This baseline gage shall have a height of 19.30 ± 0.10 mm. Check all plug heights when new plugs are purchased and periodically thereafter to make sure all are equal in height.

5.4 Balance—having a capacity of at least 50 g and a sensitivity of 0.001 g.

6. Standardization of Apparatus

6.1 Before proceeding with standardization of the Fisher sub-sieve sizer, the following items shall be checked:

6.1.1 The chart shall be properly aligned horizontally with the indicator pointer.

6.1.2 The rack and pinion shall be properly aligned vertically with the chart.

6.1.3 The sample tube or plugs shall not be worn.

6.1.4 The manometer and air resistors shall be free of visible contamination.

6.1.5 The rubber sample tube seals shall not be worn to the point where leakage occurs.

6.1.6 The sample packing post shall be properly adjusted.

6.1.7 The drying agent shall be in proper condition.

6.1.8 The manometer and standpipe levels shall be checked.

6.1.8.1 Adjust the manometer only when the machine is not operating and with the pressure released for minimum of 5 min to allow the manometer tube to drain completely.

6.2 The standardization of the Fisher sub-sieve sizer shall be made using the Fisher jewel calibrator tube (jewel orifice tube) as the primary standard. Specification shall be made at both ranges of the machine.

The Fisher jewel calibrator tube used for standardization shall be checked under a microscope at least once a month to determine the condition and cleanliness of the orifice.

If the orifice is not clean, clean as described in the Fisher sub-sieve sizer instruction manual.

6.3 With the sub-sieve sizer properly adjusted and set to the proper range, proceed as follows:

6.3.1 Mount the Fisher jewel calibrator tube between the rubber seal supports just to the right of the brass post. Clamp the upper cap down onto the tube so that an airtight seal is obtained at both ends.

6.3.2 Adjust the calculator chart so that the porosity reading corresponds to the value indicated on the jewel calibrator tube.

6.3.3 Switch on the machine and allow it to warm up for a minimum of 20 min. Adjust the pressure-control knob, located near the bubble observation window at the lower left of the panel, until the bubbles rise in the standpipe at the rate of two to three bubbles per second. This will cause the water line to rise above the calibration mark on the upper end of the standpipe. This is normal and does not mean the calibration is in error.

6.3.4 The liquid level in the manometer tube will rise slowly until it reaches a maximum. Allow at least 5 min for this to happen. At the end of this period, using care not to disturb the chart, turn the rack up until the upper edge of the crossbar coincides with the top of the liquid meniscus in the manometer. The Fisher Number is indicated by the location of the pointer tip in relation to the curves on the calculator chart. Record the ambient temperature to the nearest 1°C. Release the clamp on the upper end of the tube slowly so the manometer returns to its zero position slowly with very little overshoot. This limits the formation of liquid droplets on the inside of the manometer tube.

6.3.5 The value obtained in this manner must correspond to the Fisher Number indicated on the jewel calibrator tube within ±1 %.

6.3.6 If the Fisher Number value as indicated on the chart does not correspond to ±1 % of the value indicated on the jewel calibrator tube, calibrate the sub-sieve as follows: Adjust either the high needle valve or the low needle valve as required to bring the Fisher number indicated on the chart to the value indicated on the jewel calibrator tube. After adjustment is made, repeat 6.3.4.

Note 3—Because only one flowmeter is used for the low (1- to
20.0-µm) Fisher Number range while both flowmeters are used for the high (20.0- to 50.0-µm) Fisher Number range, the low range should be standardized first. After the low range is standardized, the high range is then standardized, making adjustments only to the one flowmeter opened up by the range-control knob.

6.3.7 Standardization with the jewel calibrator tube is recommended before and after any series of determinations or at least very 4 h of continued operation. Warm-up of the machine is required if it has been off for more than 30 min.

7. Procedure

7.1 Temperature of Test—Make Fisher Number determinations with ±2°C of the temperature at which standardization of the Fisher sub-sieve sizer was made. Restandardize if the temperature of the test varies more than ±2°C.

7.2 Size of Test Sample—The mass of sample used for tests should be equal in grams (within ±0.01 g) to the theoretical density of the powder (tungsten, 19.3 g; molybdenum, 10.22 g; tantalum, 16.6 g; and so forth).

7.3 Fisher Number Determination—The Fisher Number determination shall be made by the same operator who makes the standardizations and is started after standardization or the determination of another sample. Proceed as follows:

7.3.1 With the sub-sieve sizer properly adjusted, set the range control to the range desired.

7.3.2 Lay a paper disk over one end of the sample tube using one of the porous plugs with the perforated surface of the plug against the surface of the paper disk. This crimps the paper around the edges and the paper precedes the plug into the sample tube. Push the plug into the tube until it is even with the end of the sample tube. Place the sample tube in a vertical position in a support with the paper side of the plug up.

7.3.3 Determine the mass of the sample.

7.3.4 With the aid of the powder funnel, completely transfer the sample into the sample tube, tapping the side of the tube and funnel two or three times each to settle the powder. Lay a second paper disk over the top of the sample tube and, using another porous brass plug, force the plug and paper disk down into the sample tube until it is just inside the sample tube. Place the sample tube on the brass post beneath the rack and pinion with the lower plug in contact with the upper end of the brass post.

7.3.5 Lower the rack, guiding it until the flat-bottom end comes in contact with the upper plug. Pack the sample firmly by turning down the pinion knob with the torque wrench or torque screwdriver until a compressive force of 222 N (50 lbf) is applied to the sample. After this force is applied, the sample should not be touching the block in which the brass post is mounted. In cases in which the tube tends to move down and rest on the block during compression, the tube can be held temporarily by hand or a spacer can be used until most of the compressive force has been applied. The spacer is then removed when the maximum force is actually applied. Apply and release maximum force a total of three times. After the final maximum compression force has been applied, check the rack to make sure it has not been removed upward with the final release of pressure. Check torque wrench or torque screwdriver for standardization at least once every month using sample pressure calibrator or an equivalent device.

7.3.6 Shift the calculator chart laterally until the extreme tip of the pointer just coincides with the sample-height curve on the chart. The pointer should be midway between the top and bottom of the line. The chart must not be moved after this setting until the determination is finished. Record the porosity value indicated at the bottom of the chart.

7.3.7 Without disturbing the sample in any way, mount the sample tube between the rubber-cushioned supports just to the right of the brass post. Clamp the upper cap down onto the sample tube so that an airtight seal is obtained at both ends.

Note 4—The sample may eventually wear and cause faulty values. When this condition is suspected, replace the tube. Sample tubes with obvious wear or scratches, or both, should be discarded.

7.3.8 Determine the Fisher Number, switching on the machine and allowing the liquid level in the manometer tube to rise until it reaches a maximum. Allow a minimum of 5 min for this to happen. The Fisher Number is indicated by the location of the tip of the pointer in relation to the curves on the calculator chart. Record this value along with the porosity for the sample and the ambient temperature at which the measurement was made.

Note 5—Formulas for calculating the Fisher Number or the equivalent spherical diameter and porosity values from sample and manometer heights are as follows:

Where sample mass is equal to the theoretical density of the material being tested:

\[
\text{Porosity} = \frac{LA - 1}{LA} \quad \text{and} \quad \text{Fisher Number (µm)} = CL \sqrt{\frac{F}{(P - F)(AL - 1)^2}}
\]

Where:

- \(L\) = sample height after compaction, cm;
- \(A\) = cross-sectional area of the sample tube, cm² (≈1.267 cm³);
- \(M\) = mass of sample for 1 cm³;
- \(D\) = theoretical density of material being tested, g/cm³;
- \(C\) = cross-sectional constant = 3.80;
- \(F\) = pressure difference of water, cm (Note: \(F = 2H\), where \(H\) = height of water column above base line, cm); and
- \(P\) = overall air pressure (determined by standpipe) = 50 cm of water.

Where sample mass is not equal to the theoretical density of material being tested, as in the case when the sample size is less than the true density:

\[
\text{Porosity} = \frac{LA - \left(\frac{M}{D}\right)}{LA} \quad \text{and} \quad \text{Fisher Number (µm)} = \frac{CLM}{D} \sqrt{\frac{F}{(P - F)(AL - \frac{M}{D})^2}}
\]

Where:

- \(M\) = mass of sample, g, and
- \(D\) = true density of material being tested, g/cm³.

For a powder in which all particles are spherical and of uniform size, the particle size \(d\) in micrometres may be
calculated from the volume-specific surface area $S_v$: 

$$d = \frac{6 \times 10^6}{S_v}$$  

(5)

A calculation of an equivalent spherical diameter based on this equation is performed automatically by the calculator chart of the Fisher sub-sieve sizer from the values related to the porosity and to the permeability of the powder bed measured by the instrument. In other words, what is determined with the instrument is the specific surface area of the powder. When an equivalent spherical diameter is determined using the Fisher sub-sieve sizer, it should be clearly kept in mind that this equivalent spherical diameter is derived from the determination of the specific surface area of the powder using a relationship that is true only for powders of uniform size and spherical shape. Hence, the term “Fisher Number” is preferred to describe the result of this test, rather than “particle size” or “equivalent spherical diameter.”

8. Report

8.1 Report the Fisher Numbers and porosities of “as-supplied” powders as the results of two determinations, each made on separate portions of the sample. Report both measured porosities of the packed samples along with both Fisher Numbers and identify the results as being determined on “as supplied” powder.

8.2 If the powder is de-agglomerated or milled in the laboratory before analysis in accordance with Practice B 859, only one determination of the Fisher Number need be made and reported along with the porosity value determined and identified as “lab milled.” If another laboratory method is used to de-agglomerate or mill the powder, sufficient information to describe the procedure completely must also be included with the results.

8.3 Table 1 provides limitations of Fisher Number.

9. Precision and Bias

9.1 Precision—The results of an interlaboratory study to determine the precision of this test method are available in ASTM Research Report No. B09–1010, a report on a study done in five laboratories on tungsten carbide powders in both the as-supplied and laboratory-milled conditions. Although this is not in conformance with the requirements of Practice E 691 (six laboratories are required), the user of this test method may infer its precision from this interlaboratory study. The pertinent conclusions are presented in 9.1.1 and 9.1.2.

9.1.1 The within-laboratory repeatability limit, $r$, as defined by Terminology E 456, was estimated to be 2 to 6 % of the measured Fisher Number. Duplicate results from the same laboratory should not be considered suspect unless they differ by more than $r$.

9.1.2 The between-laboratory reproducibility limit, $R$, as defined by Terminology E 456, was found to be estimated by the following equation:

$$R = 0.173F - 0.042$$  

(6)

where:

$R$ = the reproducibility limit and

$F$ = the measured Fisher Number.

Results from two different laboratories should not be considered suspect unless they differ by more than $R$.

9.2 Bias—The Fisher Number is a calculated estimate of average particle diameter in a powder. No absolute method of determining powder particle size exists, nor are there any universally recognized standard or reference powders for this measurement; therefore, it is not possible to discuss the bias of results by this test method.

10. Keywords

10.1 envelope-specific surface area; Fisher Number; metal powder; particle size; porosity; powder; specific surface

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6 Supporting data are available from ASTM Headquarters. Request RR: B09–1010.
Committee B09 has identified the location of selected changes to this standard since the last issue (B 330 – 00) that may impact the use of this standard.

(1) Paragraph 8.1 was modified to remove the 3 % duplication requirement and to report both results. Rationale: It has been found that sampling and precision differences with fine metal powders preclude meeting the former 3 % duplication criterion for “as-supplied” materials. The decision was made at a meeting of Subcommittee B09.03 on April 30, 2002, to remove the 3 % requirement, retaining the duplicate test requirement, and reporting both results.

(2) Paragraphs 1.1 and 4.2 and Note 3 were changed to limit applicability to >1 µm. Rationale: Discussions at the same subcommittee meeting mentioned above indicated that the range of applicability of this method does not extend to the submicrometer particle size regime.

(3) Paragraph 4.1 was editorially modified.

(4) Equation 3 in Note 5 was changed to correct a typographical error.

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Standard Test Method for Compressibility of Metal Powders in Uniaxial Compaction

This standard is issued under the fixed designation B 331; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers determination of compressibility of metal powders as measured by the extent to which they can be densified in a specified die under controlled conditions.

1.2 The values stated in inch-pound units are to be regarded as the standard. The SI equivalents are in parentheses and may be approximate.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 215 Practices for Sampling Finished Lots of Metal Powders

B 243 Terminology of Powder Metallurgy

B 328 Test Method for Density, Oil Content, and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings

3. Terminology

3.1 Definitions—For definitions of terms used in this test method refer to Terminology B 243.

4. Summary of Test Method

4.1 The test method consists of compacting a sample of metal powder in a confining die, ejecting it from the die, and measuring its green density. The powder is subjected to uniaxial loading in a standardized die of rectangular or of round cross section.

5. Significance and Use

5.1 The compressibility obtained is a measure of a material characteristic inherent in the powder. The test method is useful as a quality control test in the evaluation and manufacturing control of metal powder production, and as an acceptance test for shipment of metal powder lots.

5.2 Results may be affected by test conditions such as the type amount, and method of lubrication, dwell time, and die material. They may not necessarily agree with results obtained under production conditions.

6. Apparatus

6.1 Die and Two Steel Punches—The die should be made of cemented carbide, or alternatively of tool steel. The set may be designed either for rectangular compacts or round compacts. Fig. 1 illustrates typical rectangular tooling and Fig. 2 illustrates typical round tooling.

6.2 Compression Testing Machine or Hydraulic Powder Compacting Press, capable of applying an adequate load with an accuracy of at least ±1.0 %.

6.3 Balance, suitable for weighing at least 100 g to the nearest 0.01 g.

6.4 Micrometer, or other suitable measuring device for measuring the dimensions of the compacts to the nearest 0.0002 in. (0.005 mm).

7. Sampling

7.1 A quantity of powder capable of producing the required number of test specimens (see 9.1 and Section 11) shall be obtained in accordance with Practices B 215.

8. Preparation of Apparatus

8.1 Lubrication is necessary to assist the ejection of the compacted test specimen from the die. Either die wall lubrication or powder lubrication may be used.

8.1.1 Unlubricated Powder may be tested in a die with lubricated walls. Apply to the die walls a mixture of a lubricant in a volatile organic liquid. After any excess liquid has drained away, allow the solution adhering to the walls to evaporate leaving a thin layer of lubricant.

Note 1—An example of such a mixture is 100 g of zinc stearate in 1 L of methyl alcohol.

8.1.2 An alternative way of testing powder not containing a lubricant is to use an otherwise unlubricated die after thoroughly mixing into the powder a sufficient amount of a suitable lubricant.
8.1.3 Lubricated Powder, received already mixed with sufficient lubricant should be tested in an otherwise unlubricated die.

8.2 Compressibility may vary according to the method of lubrication, type, and amount of lubricant.

8.3 The parties shall agree on the method, amount, and type of lubricant.

9. Procedure

9.1 Powder Sample Mass—The powder sample mass shall be such as to result in a rectangular compact 0.240 to 0.260 in. thick (6.1 to 6.6 mm) or a round compact 0.27 to 0.29 in. (6.9 to 7.4 mm) thick. For a powder whose compressibility is not known, it may be necessary to adjust the powder mass based on the specimen thickness obtained in an initial test.

9.2 Compaction—The specimen shall be made using a double action pressing process. One example of this type of compaction is as follows:

9.2.1 Insert the lower punch into the die cavity. Position the die cavity to the desired filling height using supporting spacers between the die and the lower press platen. Pour the powder sample into the die cavity taking care to ensure that the powder is uniformly distributed. Insert the upper punch and then apply and release a preliminary pressure of approximately 5000 psi (35 MPa). Remove the spacers supporting the die. If the die is supported by springs, or in some similar way, it is unnecessary to apply the preliminary pressure. Apply the final pressure. In
special cases where the results may be affected by the rate of pressure application, a rate not exceeding 60,000 psi/min (415 MPa/min) is recommended.

9.2.2 Release the pressure as soon as the maximum pressure is attained, because pressure dwells of as little as 10 s can increase iron powder compressibility by 0.3%.

9.3 Ejection—the compact is then either ejected from the die or exposed via a withdrawal process, in accordance with the type of apparatus used. For the commonly used punch and die systems described in 9.2, the part is ejected by pushing back the die with the aid of two spacer blocks, or the like. The blocks should be longer than the combined length of the upper punch and the formed part. If possible, remove the upper punch by hand. If not possible, perform ejection with blocks until the punch clears the die, remove the punch, and continue to eject the test piece until it clears the die. Carefully deburr test pieces with fine emery paper.

9.4 Measurements—Weigh the compact to the nearest 0.01 g. Measure its dimensions to the nearest 0.0002 in. (0.005 mm). Compacts may not have top and bottom surfaces that are exactly parallel and care must be taken in determining an average thickness.

9.5 Compacting Pressures—Samples of powder may be pressed either at a single specified pressure or at a series of specified pressures. In the latter case, the densities obtained can be utilized for drawing the compressibility curve of the powder, such as, a graph of density as a function of the compacting pressure. Alternatively, by trial and error, the powder may be compacted to a specified green density and the compacting pressure taken as a measure of its compressibility.

9.6 For the method selected in 9.5, repeat 9.2-9.4 twice more.
10. Calculations

10.1 Calculate the density of the green compact as follows:

10.1.1 For round compacts:

\[ \rho_g = \text{Green density, g/cm}^3 = \frac{4}{(4/\pi) \times 1000 \times M (d^2 \times l)} \]
\[ = 0.0777 (M/d^2 \times l) \]  

(1)

where:

\( M \) = mass of compact, g,
\( d \) = diameter of compact, in., and
\( t \) = thickness of compact, in.

Where the test specimen is measured in mm, the equation is:

\[ \rho_g = \text{Green density, g/cm}^3 = \frac{1273 (M/d^2 \times h)}{1000} \]
\[ = 0.0610 (M/L \times W \times T) \]  

(2)

10.1.2 For rectangular compacts:

\[ \rho_g = \text{Green density, g/cm}^3 = \frac{1}{(1/2.54)^3 \times M(L \times W \times l)} \]
\[ = 0.025 \text{ (M/L \times W \times T)} \]  

(3)

where:

\( M \) = mass of compact, g,
\( L \) = length of compact, in.,
\( W \) = width of compact in., and
\( t \) = thickness of compact, in.

Where the test specimen is measured in mm, the equation is:

\[ \rho_g = \text{Green density, g/cm}^3 = 1000 (M/L \times W \times t) \]  

(4)

10.2 Alternatively, the density of the green compact may be calculated by the water immersion method of Test Method B 328.

11. Report

11.1 Report the compressibility as the average of three density measurements, calculated to the nearest 0.01 g/cm³. Report also the individual density measurements and the compaction pressure rounded to the nearest 0.5 tsi.

11.2 Alternatively, report the compressibility curve of the powder as a graph drawn through points representing a single determination, at not less than four compacting pressures or, draw the line through points representing the average of three determinations at three compacting pressures.

11.3 If the alternative method of compacting to a specified green density is used, report the compressibility as the compacting pressure, with its precision, and the individual pressures. Report also the specified green density used.

11.4 Report the compact thickness, and type, amount, and method of lubrication. (For die wall lubrication, specify the amount of lubrication as the weight percent dissolved in the solvent. Where admixed lubricant is used, the type and amount used shall be indicated).

11.5 Report whether the method used for determination of density was as prescribed in 10.1 or 10.2.

12. Precision and Bias

12.1 For the density determination method of 10.1, ferrous and nonferrous powders, the repeatability interval, \( r \), is 0.025 g/cm³. On the basis of test error alone, the difference in absolute value of individual test results obtained in the same laboratory on the same material will be expected to exceed 0.025 g/cm³ only about 5 % of the time.

12.2 For the density determination method of 10.1, the reproducibility interval, \( R \), for ferrous and nonferrous powders is 0.07 g/cm³. On the basis of test error alone, the difference in absolute value between individual test results obtained in two different laboratories on the same material will be expected to exceed \( R \) only about 5 % of the time. Thus, if a larger difference is found, there is reason to question one or both test results.

12.3 No precision data for this test method are available, based on 10.2, the use of Test Method B 328 for density determination.

12.4 No statement can be made about bias, because there are no accepted standard or reference powders for compressibility testing.

13. Keywords

13.1 compressibility; metal powder

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NOTE: Supporting data are available from ASTM Headquarters. Request RR:B09-1002.
Standard Practice for Preparation of Nickel for Electroplating with Nickel

This standard is issued under the fixed designation B 343; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice summarizes well-known, generally practical methods for producing adherent electrodeposits of nickel on nickel.

1.2 Electrodeposits of nickel on nickel are produced, for example, to improve the performance of decorative coatings, to reclaim electroplated parts that are defective, and to resume nickel electroplating after interruptions in processing. Interruptions may be deliberate, for example, to machine the electrodeposit at an intermediate stage in the electrodeposition of thick nickel coatings. The interruptions may be unintentional, for example, resulting from equipment and power failures.

1.3 To ensure good adhesion of nickel to nickel, precautions should be taken to avoid biopolar effects during nickel electroplating. This is of particular importance in return-type automatic plating machines where one rack follows another rack closely. Bipolar effects can be avoided by making the racks cathodic while they are entering or leaving the nickel tank. Separate current control on entry and exit stations is desirable.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Types of Nickel

2.1 The types of nickel for which an overplate of nickel may be desired are dull nickel, semi-bright nickel, bright nickel, and nickel strike. Variations in these types may possibly require special handling.

2.2 Surface conditions of the nickel may vary as follows:

2.2.1 freshly electroplated surfaces that are still wet with electroplating solution or rinse water (see 5.1),

2.2.2 Freshly electroplated surfaces that have been allowed to dry (see 5.2),

2.2.3 Buffed, polished, or machine-ground surfaces (see 5.3), and

2.2.4 Surfaces that have been given a reverse-current treatment in an alkaline solution for cleaning or possibly stripping an overplate of chromium (see 5.4).

3. Cleaning

3.1 The following cleaning treatments may be used for all conditions and types of electrodeposited nickel. The choice of the procedure will be governed largely by the condition of the surface.

3.1.1 Degreasing—Degreasing is used to remove the bulk of grease, oil, and buffing compounds that may be present on the surface. The cleaning may be effected with vapor degreasing, organic solvents, emulsion cleaners, or soak cleaner.

3.1.2 Electrolytic Alkaline Cleaning—Removal of final traces of dirt, grease, and oil is accomplished best with electrolytic alkaline cleaning. The solution may be either a proprietary cleaner or a formulated one. Since a nickel surface forms an oxide coating if treated anodically in an alkaline solution, this condition must be altered in subsequent steps if it cannot be avoided.

4. Activating

4.1 The procedure used for etching or activating the nickel surface usually determines the soundness of the adhesion. The choice of the procedure may be governed by the condition of the surface and possibly the type of nickel. The milder etching treatment should be used in the case of highly finished surfaces, but it may result in sacrificing maximum adhesion. The thickness of the nickel may mitigate against the use of certain etching procedures, and therefore the thickness removed is indicated for each procedure described in 4.2 to 4.8.

4.2 Anodic Treatment in Concentrated Sulfuric Acid—(Nickel removed nil). A 70 mass % sulfuric acid solution containing 661 mL of concentrated, 96 mass % sulfuric acid (density 1.83 mL) diluted to 1 L may be used for activating the nickel surface provided the temperature of the solution is not over 30°C (see Warning). When the initial mixture cools,
dilute to exact volume. The time of treatment should be about 1 min at a current density of 10 A/dm². At this current density the nickel normally goes passive and a bright surface becomes only slightly dull. This type of passivity is removed by subsequent rinsing in water. (Warning—Slowly add the sulfuric acid with rapid stirring to the approximate amount of water required.)

4.3 Anodic Etching in Sulfuric Acid—(Nickel removed approximately 1.3 µm.) A 25 mass % sulfuric acid solution, containing 166 mL of concentrated, 96 mass % sulfuric acid (density 1.83 g/mL), diluted to 1 L is used for this anodic etching treatment in which the nickel surface is first etched at a low current density of 2 A/dm² for 10 min and then made passive at 20 A/dm² for 2 min and finally cathodic for 2 or 3 s at 20 A/dm². See Warning in 4.2. The temperature of the solution should be kept below 25°C. This treatment results in excellent adhesion, but the amount of etching makes it less desirable for a highly finished surface.

4.4 Anodic Etching in Watts-Type Bath—(Nickel removed approximately 4 µm.) This procedure employs an anodic treatment in a low-pH Watts bath for 3 min at 1 A/dm²(10 A/ft²), followed by cathodic treatment for 3 to 6 min at 3 A/dm²(30 A/ft²). The composition of the solution is 240 to 300 g/L nickel sulfate (NiSO₄ · 7H₂O), 40 to 60 g/L nickel chloride (NiCl₂ · 6H₂O), and 25 to 40 g/L boric acid. It is operated at a temperature between 25 and 50°C and at a pH between 1.5 and 2.0. An additional bath is not required if a means of reversing the current is available. The amount of etching obtained is sufficient to dull a bright surface, and there is danger of bath contamination from bare areas.

4.5 Acid-Nickel Chloride Treatment—(Nickel removed approximately 1.3 µm.) This procedure employs an anodic treatment followed by a cathodic treatment in a low-pH nickel chloride solution. The composition of the solution is 240 g/L of nickel chloride (NiCl₂ · 6H₂O) and 31 mL of concentrated, 37 mass % hydrochloric acid (density 1.16 g/mL). The normal procedure is to make the work anodic for 2 min at 3 A/dm² and then cathodic for 6 min at the same current density. Where it is not possible to reverse the current in the same tank, the two steps may be carried out in separate tanks. The latter arrangement may be more practical commercially. This treatment produces a dull finish on a bright surface, but the etching is not sufficiently drastic to preclude finishing with bright nickel deposit.

Note 1—Nickel anode materials containing greater than 0.01 mass % sulfur are not recommended for use in acid nickel strike baths operating at pH 0.5, or lower, to avoid oxidation of sulfides by hydrochloric acid.

4.6 Etching by Acid Immersion—(Nickel removed approximately 1.3 µm.) Adequate etching may be obtained on some nickel surfaces by a short dip at room temperature in a solution of either 500 mL of concentrated 37 mass % hydrochloric acid (density 1.18 g/mL) diluted to 1 L, or 150 mL of concentrated 96 mass % sulfuric acid (density 1.83 g/mL) diluted to 1 L. See 4.2. The length of the immersion required may vary from 10 s to 1 min.

4.7 Electropolishing Treatment—(Nickel removed approximately 1.3 µm.) This procedure is commonly employed on rejects that have been repolished to remove the defective area.

The electropolishing solution commonly used consists of a mixture of 150 mL of 96 mass % sulfuric acid (density 1.83 g/mL) and 630 mL of 85 mass % phosphoric acid (density 1.69 g/mL) diluted to 1 L. See Warning in 4.2. Temperature of solution ranges from 45 to 55°C. The work is made anodic at current densities from 15 to 20 A/dm². The electropolishing treatment is usually applied for 2 to 15 min. The cathodes may be electrolytic nickel strip. Subsequent alkaline cleaning and an acid dip are normally used before electroplating.

4.8 Cathodic Treatment—(Nickel removed nil.) These procedures are recommended where the nickel surface has not been severely passivated. Prior cleaning may be required, such as alkaline soak cleaning or electrocleaning, or both. If electrocleaning is employed, only cathodic current should be used. In the following formulations, 96 mass % sulfuric acid with a density of 1.83 g/mL, and 37 mass % hydrochloric acid with a density of 1.16 g/mL, are used.

4.8.1 Cathodic Treatment in Sulfuric Acid:

<table>
<thead>
<tr>
<th>Component</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sulfuric acid</td>
<td>30 to 100 mL</td>
</tr>
<tr>
<td>Water</td>
<td>to 1 L</td>
</tr>
<tr>
<td>Temperature</td>
<td>ambient to 45°C</td>
</tr>
<tr>
<td>Current density</td>
<td>1 A/dm²</td>
</tr>
<tr>
<td>Anodes</td>
<td>pure lead</td>
</tr>
<tr>
<td>Time</td>
<td>30 s to 5 min</td>
</tr>
</tbody>
</table>

4.8.2 Cathodic Treatment in Hydrochloric Acid:

<table>
<thead>
<tr>
<th>Component</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrochloric acid</td>
<td>100 to 300 mL</td>
</tr>
<tr>
<td>Water</td>
<td>to 1 L</td>
</tr>
<tr>
<td>Temperature</td>
<td>ambient</td>
</tr>
<tr>
<td>Current density</td>
<td>1 A/dm²</td>
</tr>
<tr>
<td>Anodes</td>
<td>electrolytic nickel strip or nickel bar anodes</td>
</tr>
<tr>
<td>Time</td>
<td>30 s to 5 min</td>
</tr>
</tbody>
</table>

Note 2—In some instances, the use of 2 g/L of ammonium bifluoride (NH₄ · HF) has been found to be beneficial in either of the above formulations.

Note 3—Proprietary acid salt formulations may be used with the supplier’s recommendations.

5. Electroplating Procedures

5.1 Wet Freshly Electroplated Surfaces—Nickel surfaces that are still wet with electroplating solution can sometimes be placed directly in the subsequent nickel electroplating bath without any special treatment. If the solutions are not compatible, a water rinse may be used between nickel baths. This procedure will normally give good adhesion with Watts-type nickel and certain semi-bright nickels but may give poor adhesion with bright nickels. A mild etch, such as a short immersion in a dilute acid solution, may be required for some semi-bright and bright nickel surfaces.

5.2 Dry Freshly Electroplated Surfaces—Nickel surfaces that have been allowed to dry following the rinse from the electroplating bath may only require an activation treatment. However, if there is a possibility of a trace of oil or grease on the surface, a cathodic alkaline cleaner should be used first. A short immersion in a dilute acid solution should be adequate to activate most nickel surfaces, but certain bright nickels may require more drastic activation.
5.3 Polished, Buffed, or Machine Ground Surface—These nickel surfaces can usually be treated alike for nickel electroplating, although the buffed surface will require more drastic degreasing treatment and alkaline cleaning. For decorative surfaces, a mild etch, such as a short immersion in a dilute acid or a short anodic treatment in sulfuric acid, should be adequate to assure good adhesion.

5.4 Anodic Alkaline Treated Surface—The oxide film on a nickel surface from anodic treatment in an alkaline solution must be removed by a suitable activation treatment before the nickel surface can be electroplated adherently with nickel. An acid dip or a mild anodic etch in sulfuric acid is usually not adequate. A heavy anodic etch in sulfuric acid, an electropolishing treatment, a low-pH nickel bath, or the acid-nickel chloride treatment will normally be required to provide a sound deposit. A wipe on a buffing wheel will also serve to remove the oxide film.

5.5 Rejects with Chromium Electroplate—On parts requiring the removal of chromium for reprocessing, use of an anodic alkaline chromium strip should preferably be avoided. Acid stripping should be used. If an anodic alkaline strip is used, then an anodic acid etch is absolutely necessary.
Standard Test Method for
Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing
(CASS Test) ¹

This standard is issued under the fixed designation B 368; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

¹ NOTE — Sections 8.1 and 10.1.12 were editorially updated in July 2003.

1. Scope

1.1 This method prescribes the conditions required in copper-accelerated acetic acid-salt spray (CASS) testing for specification purposes. The standard does not specify the type of test specimen or exposure periods to be used for a specific product, nor the interpretation to be given to the results.

1.2 This method is applicable to evaluating the corrosive performance of decorative copper/nickel/chromium or nickel/chromium coatings on steel, zinc alloys, aluminum alloys, and plastics designed for severe service. It is also applicable to the testing of anodized aluminum. The suitability of this test and correlation of results with service experience should be determined before it is specified for coating systems or materials other than those mentioned in this paragraph.

NOTE 1—The following standards are not requirements. They are referenced for information only: Practices B 537 and E 50, Specifications B 456 and B 604, and Test Method B 602.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For more specific safety precautionary information see 8.1.

2. Referenced Documents

2.1 ASTM Standards:
B 117 Practice for Operating Salt Spray (Fog) Apparatus²
B 162 Specification for Nickel Plate, Sheet, and Strip³
B 456 Specification for Electrodeposited Coatings of Copper Plus Nickel Plus Chromium and Nickel Plus Chromium⁴
B 537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure⁴
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings⁴
B 604 Specification for Decorative Electroplated Coatings of Copper Plus Nickel Plus Chromium on Plastics⁴
D 1193 Specification for Reagent Water⁵

3. Significance and Use

3.1 The CASS test is widely employed and is useful for specification acceptance, simulated service evaluation, manufacturing control, and research and development. It was developed specifically for use with decorative, electrodeposited nickel/chromium and copper/nickel/chromium coatings. Use of the test has improved the quality of electroplated parts and led to the development of new and superior electroplating processes.

4. Apparatus

4.1 The apparatus required for the CASS test consists of a fog chamber, a salt-solution reservoir, a supply of compressed air, one or more atomizing nozzles, specimen supports, provision for heating the chamber, and necessary means of control.

4.2 The size and detailed construction of the apparatus are optional, provided the conditions meet the requirements of this method. The construction of the apparatus is described in the appendix of Test Method B 117. For the CASS test, however,
the requirements for air pressure and temperature are typically 0.08 to 0.12 MPa and 60 to 65°C, respectively. The actual air pressure will be that required to produce the proper collection rate (see 8.3.1).

4.3 The apparatus shall be constructed so that drops of solution that accumulate on the ceiling or cover of the chamber do not fall on the specimens being tested. Drops of solution that fall from the specimens shall not be returned to the solution reservoir for respraying.

4.4 Materials of construction shall not affect the corrosiveness of the fog, nor be themselves corroded by the fog.

5. Test Solution

5.1 Prepare the salt solution by dissolving 5 parts by weight of salt in 95 parts of water conforming to Specification D 1193, Type IV. The salt shall be sodium chloride (NaCl), ACS reagent grade, or equivalent. The pH of this solution shall be between 6.0 and 7.0. Impurities or contamination of either the salt or the water, or both, should be suspected if the pH is outside of this range (Note 2).

5.2 Add 0.25 g of reagent grade copper chloride (CuCl₂·2H₂O) to each litre of the salt solution; dissolve and mix thoroughly.

5.3 The pH of the salt-copper solution shall be adjusted to the range of 3.1 to 3.3, as measured on a sample of the collected spray, by the addition of glacial acetic acid, ACS reagent grade, or equivalent (Note 3). The pH measurement shall be made electrometrically at 25°C. Before the solution is atomized, it shall be free of suspended solids (Note 4).

NOTE 2—A solution having a specific gravity of 1.030 to 1.040, when measured at a temperature of 25°C, will meet the concentration requirements. It is suggested that a daily check be made.

NOTE 3—The initial solution may be adjusted to a pH of 3.0 to 3.1, with the exception that the pH of the collected fog will be within the specified limits. Adjustment of the initial pH for makeup solution is based upon the requirements to maintain the required pH of the collected samples. If less than 1.3 or more than 1.6 mL of the glacial acetic acid are required per litre of sodium chloride and copper solution to attain the specified pH, some discrepancy in the system may be suspected (the purity of the water or salt, or both; the accuracy of the pH meter; the general cleanliness of the system; etc.).

NOTE 4—The freshly prepared salt solution may be filtered or decanted before it is placed in the reservoir, or the end of the tube leading from the solution to the atomizer may be covered with a double layer of cheesecloth to prevent plugging of the nozzle.

6. Air Supply

6.1 The compressed air supply to the nozzle or nozzles for atomizing the test solution shall be free of oil and dirt (Note 5). Pressure shall be adequate to provide a specified condensate rate. Pressure of 0.10 ± 0.02 MPa has been found satisfactory (Note 6).

NOTE 5—The air supply can be freed of oil and dirt by passing it through a water scrubber or at least 60 cm of suitable cleaning material, such as sheep’s wool, excelsior, slag wool, or activated alumina. Commercial filters for compressed air may be used.

NOTE 6—Atomizing nozzles may have a critical pressure, at which an abnormal increase in the corrosiveness of the salt fog occurs. If the critical pressure of a nozzle has not been determined with certainty, control of fluctuation in the air pressure within ±0.0007 MPa by installing a pressure regulator valve minimizes the possibility that the nozzle will be operated at its critical pressure.

7. Test Specimens

7.1 The type and number of test specimens to be used, as well as the criteria for the evaluation of the test results, shall be defined in the specifications covering the material or product being tested or shall be mutually agreed upon between the purchaser and the supplier.

8. Procedure

8.1 Preparation of Test Specimens—Clean metallic and metallic coated specimens. Unless otherwise agreed upon, clean decorative copper/nickel/chromium or nickel/chromium coatings immediately before testing by wiping significant surfaces with a cotton pad saturated with a slurry containing 10 g of pure magnesium oxide powder (ACS reagent grade) in 100 mL of distilled water. Upon rinsing in warm running water, be sure that the clean surface is free of water break. Anodized aluminum parts may be cleaned with inhibited 1,1,1-trichloroethane or other suitable organic solvent (see Warning). Do not clean organic and other nonmetallic coated specimens. Other methods of cleaning, such as the use of a nitric-acid solution for the chemical cleaning or passivation of stainless steel specimens, are permissible when agreed upon between the purchaser and the supplier. Take care that the specimens after cleaning are not recontaminated by excessive or careless handling. Protect the cut edges of plated, coated, or multilayered materials and areas containing identification marks or in contact with the racks or supports with a coating that is stable under the conditions of the test, such as wax, stop-off lacquer, or pressure-sensitive tape. (Warning—1,1,1-Trichloroethane should be used in a well-ventilated area away from open flames.)

8.2 Positioning of Specimens—Position the specimens in the CASS test chamber during the test so that the following conditions are met:

8.2.1 Support or suspend the specimens 15 ± 2° from the vertical and preferably parallel to the principal direction of horizontal flow of fog through the chamber, based upon the dominant surface being tested. Support or suspend automobile parts, however, so as to expose all significant surfaces at the general level of the condensate collectors. If the position on the automobile is vertical, place the part in an incline position 15° from vertical to allow surface wetting by the condensate. If the position on the automobile is facing down, rotate the part approximately 180° to test the significant surface. If there are several significant surfaces at different angles, expose each surface of one or more specimens.

8.2.2 Make sure the specimens do not come in contact with each other or any other metallic material or any material capable of acting as a wick.

8.2.3 Place each specimen so as to permit free settling of fog on all specimens.

8.2.4 Make sure the salt solution from one specimen does not drip on any other specimen.

8.2.5 Place the specimens in the chamber just prior to bringing the test chamber to the required temperature and
turning on the air, since storage in an idle chamber overnight, or for other significant length of time, can affect test results.

NOTE 7—Suitable materials for the construction or coating of racks and supports are glass, rubber, plastic, or suitably coated wood. Bare metal should not be used. Specimens are preferably supported from the bottom or the side. Slotted wooden strips are suitable for the support of flat panels. Suspension from glass hooks or waxed string may be used as long as the specified position of the specimens is obtained. If necessary, such suspension may be made by means of secondary support at the bottom of the specimens.

8.3 Conditions in the Salt-Spray Chamber—Maintain the exposure zone of the CASS test chamber at a temperature of 49 ± 1°C during the exposure period (Note 8). After closing the test chamber, bring the temperature to 49°C before the fog is turned on. The test duration shall commence when the temperature is 49 ± 1°C and fog is present in the chamber. Heat the air supply by passing fine bubbles through heated distilled or deionized water (see Specification D 1193, Type IV) so that the temperature of the air after expansion at the nozzle is 49 ± 1°C. Record the temperature within the exposure zone of the closed cabinet (Note 9) twice a day at least 7 h apart (except Saturdays, Sundays, and holidays, when the salt-spray test is not interrupted for exposing, rearranging, or removing test specimens or to check and replenish the solution in the reservoir).

NOTE 8—This can be best accomplished by preheating the chamber to 49°C before starting solution atomization.

NOTE 9—A suitable method to record the temperature is by a thermometer that can be read from outside the closed cabinet. The recorded temperature must be obtained with the salt-spray chamber closed to avoid a false low reading, because of wet-bulb effect when the chamber is open. Automatic control of temperature in the chamber and a continuous record of temperature are desirable.

8.3.1 Place at least two clean fog collectors within the exposure zone so that no drops of solution will be collected from the test specimens or any other source. Position the collectors in the proximity of the test specimens, one nearest to any nozzle and the other farthest from all nozzles. A preferred arrangement is shown in Fig. 1. Make sure that the fog is such that for each 80 cm² of horizontal collecting area each collector collects from 1.0 to 2.0 mL/h of solution, based on a typical run of at least 22 h (Note 10). Maintain the sodium chloride concentration of the collected solution between 4.5 and 5.5 mass % (Note 11 and Note 12). Dilution and evaporation of condensate should be avoided (Note 13).

NOTE 10—Suitable collecting devices are glass funnels with the stems inserted through stoppers into graduated cylinders. Funnels with a diameter of 10 cm (area of about 80 cm²) and 50-mL cylinders are preferred.

NOTE 11—A solution having a specific gravity of from 1.030 to 1.040, when measured at 25°C, will meet the concentration requirement. The concentration may also be determined as follows: Dilute 5 mL of the collected solution to 100 mL with distilled water and mix thoroughly; pipet a 10-mL aliquot into an evaporating dish or casserole; add 40 mL of distilled water and 1 mL of 1% potassium chromate (K₂CrO₄) (ACS reagent grade with less than 0.005% chloride) and titrate with 0.1 N silver nitrate (AgNO₃) (ACS reagent grade) solution to the first appearance of a persistent red coloration. A test solution that requires between 3.9 and 4.9 mL of 0.1 N AgNO₃ solution will meet the concentration requirements.

NOTE 12—It has not been found necessary to check copper concentration of the collected solution.

NOTE 13—Factors that may contribute to dilution and evaporation of condensate and make it difficult to control the concentration are lower temperature, inadequate cover insulation, and prolonged storage of test solution at above room temperature.

8.3.2 Direct or baffle the nozzle or nozzles so that none of the spray can impinge directly on the test specimens.

8.4 Continuity of Test—Unless otherwise specified in the specification covering the material or product being tested, the test shall be continuous for the duration of the entire test period. Continuous operation implies that the chamber be closed and the spray operating continuously, except for the short daily interruptions necessary to inspect, rearrange, or remove test specimens, to check and replenish the solution in the reservoir, and to make necessary recordings as described in 8.3. Schedule operations so that these interruptions are held to a minimum.

8.5 Period of Test—Designate the period of test by the specifications covering the material or product being tested or as mutually agreed upon between the purchaser and the supplier.

NOTE 14—Typical exposure periods are: 6, 16, 22, 48, 96, 192, 240, 504, and 720 h.

8.6 Cleaning Tested Specimens—Unless specified in the specifications covering the material or product being tested, carefully remove specimens at the end of the test, rinse gently in clean, running, warm water (38°C, max) to remove salt deposits from their surfaces, and then dry immediately. To dry the specimens use a stream of clean, compressed air at 0.25 to 0.30 MPa gage pressure.

NOTE 15—If steel parts are rinsed immediately after removal from the test chamber, small rust spots or flakes of rust may lift off and be rinsed away. It is then difficult to tell where the spots had been. If, after removal from the test chamber, the part is allowed to dry 30 to 60 min before rinsing, all corrosion spots are easily visible. This not does not apply to zinc-die castings.

8.7 Calibration of Corrosive Conditions—This procedure for calibration and standardization of the corrosive conditions within the cabinet involves exposure of nickel test panels and determination of their mass loss in a specified period of time. This may be done monthly or more frequently to make sure the cabinet is operating within the limits specified in 8.7.4.

8.7.1 Use nickel panels that are 7.5 by 10 cm, approximately 0.09 cm thick, and made from the higher carbon grade (0.15% max) given in Specification B 162.

8.7.2 Place one weighed nickel test panel in each corner of the cabinet with the 10-cm length inclined 30° to the vertical using plastic supports that are about 1.5 by 4 by 7.5 cm. The four test panels should be on the inboard side of the condensate.
collectors parallel to the cabinet length. Make sure the top of the panels are at the same level as the top of the condensate collectors.

8.7.3 Expose the test panels to 22 h of CASS testing, recording test operating conditions on the data sheet as usual. Remove the panels from the cabinet and rinse each in reagent grade water (see Specification D 1193, Type IV) to remove salt. Immerse each panel in 20% by volume hydrochloric acid prepared with distilled water for 2 min at 20 to 25°C using nickel-copper alloy 400 (UNS N04400) tongs. The tongs may be nonmetallic or another material that will not affect the mass loss of the nickel by galvanic action. Rinse each panel in water at room temperature and then in warm (38°C) flowing water. Dry panels in an oven at 100°C for 10 min. Allow the panels to reach room temperature and weigh immediately on an analytical balance.

8.7.4 Determine the mass loss of each panel by subtracting its weight after exposure from its initial weight. Determine the corrosive loss by dividing the mass loss by the area of one face of the test panel (77.5 cm²). The corrosive rate shall be 0.45 to 0.85 mg/cm² to satisfy the requirements of this method.

9. Evaluation of Results

9.1 A careful and immediate examination shall be made for the extent of corrosion of the dry-test specimens; or for other failure, as required by the specifications covering the material or product being tested; or by agreement between the purchaser and the supplier.

10. Records and Reports

10.1 The following information shall be recorded, unless otherwise prescribed in the specifications covering the material or product being tested:

10.1.1 The title, designation and year of issue of this method.

10.1.2 The salt, water, copper chloride, and acetic acid used in preparing the salt solution,

10.1.3 All readings of temperature within the exposure zone of the chamber,

10.1.4 Daily records of data obtained from each fog collecting device, including the following:

10.1.4.1 Volume of salt solution collected in (mL/h)/8 cm²,

10.1.4.2 Concentration or specific gravity at 25°C of solution collected, and

10.1.4.3 pH of solution collected,

10.1.5 Nickel panel corrosion rate calibration,

10.1.6 Type of specimen and its dimensions, or number, or description of part,

10.1.7 Method of cleaning specimens,

10.1.8 Method of supporting or suspending the article in the salt-spray chamber,

10.1.9 Description of protection used as required in 8.1,

10.1.10 Exposure period,

10.1.11 Interruptions in the test, cause and length of time, and

10.1.12 Results of inspections.

Note 16—If any of the atomized salt solution that has not come in contact with the test specimens is returned to the reservoir, it is advisable to record the concentration or specific gravity and the pH of this solution also.

Note 17—Round robin testing revealed the inability to exhibit repeatability of the mass loss requirements of 8.7.4. Further testing is ongoing to clarify the root causes.

11. Precision and Bias

11.1 The reliability, reproducibility, and accuracy of the CASS test depends on proper and consistent control of a number of factors, including:

11.1.1 Cleaning of the test samples,

11.1.2 Positioning of the specimens and samples in the test cabinet,

11.1.3 Rate of condensation within the test cabinet,

11.1.4 CASS solution makeup,

11.1.5 Cabinet maintenance, and

11.1.6 Calibration of corrosive conditions.

11.2 The precision and bias of the CASS test is being determined and will include study of the reproducibility of the calibration procedure described in 8.7.
INTRODUCTION

These definitions correspond to interpretations as applied to electroplating and do not necessarily correspond to the definitions used in other fields.

abrasive blasting—a process for cleaning or finishing by means of an abrasive directed at high velocity against the work piece.
activator—in diffusion coatings, a chemical, usually a halide salt, that enters into a reaction with the source or master alloy, depositing the source on the substrate.
activation—elimination of a passive condition on a surface.
activity (ion)—the ion concentration corrected for deviations from ideal behavior. Concentration multiplied by activity coefficient.
addition agent—a material added in small quantities to a solution to modify its characteristics. It is usually added to a plating solution for the purpose of modifying the character of a deposit.
adhesion—the attractive force that exists between an electrodeposit and its substrate that can be measured as the force required to separate an electrodeposit and its substrate.
adhesion, practical, n—the force or work required to detach or remove a coating from the underlayer or substrate; it can be measured in terms of peel, pull, or shear strength as an experimentally determined quantity.
aluminizing—forming of an aluminum or aluminum alloy coating on a metal by hot dipping, hot spraying, or diffusion.
amphorous—noncrystalline, or devoid of regular structure.
amper—current that will deposit silver at the rate of 0.0011180 g/s. Current flowing at the rate of 1 C/s.
angstrom unit (Å)—10^-8 cm.
anion—a negatively-charged ion.
anode—the electrode in electrolysis, at which negative ions are discharged, positive ions are formed, or other oxidizing reactions occur.
anode corrosion—dissolution of anode metal by the electrochemical action in an electrolytic cell.
anode efficiency—current efficiency of a specified anodic process.
anode film—(1) the layer of solution in contact with the anode that differs in composition from that of the bulk of the solution. (2) The outer layer of the anode itself consisting of oxidation or reaction products of the anode metal.
anode polarization—See polarization.
anodic coating—a protective, decorative, or functional coating, formed by conversion of the surface of a metal in an electrolytic oxidation process.
anodizing—an electrolytic oxidation process in which the surface of a metal, when anodic, is converted to a coating having desirable protective, decorative, or functional properties.
anolyte—the portion of electrolyte in the vicinity of the anode; in a divided cell, the portion of electrolyte on the anode side of the diaphragm.
audi-pitting agent—an addition agent for the specific purpose of preventing gas pits in a deposit.
autocatalytic plating—deposition of a metal coating by a controlled chemical reduction, catalyzed by the metal or alloy being deposited.
amtomatic machine (or conveyor)—a machine for mechanically processing parts through treatment cycles, such as cleaning, anodizing, or plating.
amtomatic plating: (1) full—plating in which the cathodes are automatically conveyed through successive cleaning and plating tanks. (2) semi—plating in which the cathodes are conveyed automatically through only one plating tank.
auxiliary anode—a supplementary anode employed during electrodeposition to achieve a desired thickness distribution of the deposit.
auxiliary cathode—See thief.
back emf (electromotive force)—the potential set up in an electrolytic cell that opposes the flow of current, caused by such factors as concentration polarization and electrode films. See emf (electromotive force).
boll burnishing—See barrel burnishing.
barrel burnishing—the smoothing of surfaces by means of tumbling the work in rotating barrels in the presence of metallic or ceramic shot, and in the absence of abrasive. In ball burnishing, the shot consists of hardened steel balls.

barrel electroplating—an electroplating process in which electrodeposits are applied to articles in bulk in a rotating, oscillating, or otherwise moving container.

barrel finishing (or tumbling)—bulk processing in barrels, in either the presence or absence of abrasives or burnishing shot, for the purpose of improving the surface finish.

barrel plating (or cleaning)—plating or cleaning in which the work is processed in bulk in a rotating container.

barrel processing—mechanical, chemical, cleaning, or electrolytic treatment of articles in bulk or in a rotating, oscillating, or otherwise moving container.

barrier layer—in anodizing aluminum, the thin, pore-free, semiconducting aluminum oxide region nearest the metal surface and distinct from the main anodic oxide coating which has a pore structure.

base metal—(1) See basis metal; (2) in diffusion coatings, the metal present in the largest proportion in an alloy.

basis metal (or material)—material upon which coatings are deposited.

bipolar electrode—an electrode that is not directly connected to the power supply but is so placed in the solution between the anode and the cathode that the part nearest the anode becomes cathodic and the part nearest the cathode becomes anodic.

black oxide—a finish on metal produced by immersing a metal in hot oxidizing salts or salt solutions.

blister—See sand blasting; grit blasting; wet blasting.

blister—a dome-shaped imperfection or defect, resulting from loss of adhesion between a metallic deposit and the substrate.

bloom—a visible exudation or efflorescence on a surface.

blue dip—a solution, once widely used, containing a mercury compound used to deposit mercury upon a metal by immersion, usually prior to silver plating.

blueing—the formation of a thin oxide film on steel, either by heating in air, or by immersion in oxidizing solutions.

bright dip (nonelectrolytic)—a solution used to produce a bright surface on a metal.

bright electroplating—a process that produces an electrodeposited having a high degree of specular reflectance in the as-plated condition.

bright electroplating range—the range of current densities within which an electroplating solution produces a bright deposit under a given set of operating conditions.

bright plating—a process that produces an electrodeposited having a high degree of specular reflectance in the as-plated condition.

bright plating range—the range of current densities within which a given plating solution produces a bright plate.

brightener—an addition agent that leads to the formation of a bright plate, or that improves the brightness of the deposit.

bright-throwing power—the measure of the ability of a plating solution or a specified set of plating conditions to deposit uniformly bright electroplate upon an irregularly shaped cathode.

bronzing—the application of a chemical finish to copper or copper alloy surfaces to alter the color.

brush plating—a method of plating in which the plating solution is applied with a pad or brush, within which is an anode and which is moved over the cathode to be plated.

brush polishing (electrolytic)—a method of electropolishing (q.v.) in which the electrolyte is applied with a pad or brush in contact with the part to be polished.

buffer—a compound or mixture that, when contained in solution, causes the solution to resist change in pH over which it is effective.

buffing—the smoothing of a surface by means of a rotating flexible wheel to the surface of which fine, abrasive particles are applied in liquid suspension, paste, or grease stick form.

building up—electroplating for the purpose of increasing the dimensions of an article.

burn off—the unintentional removal of an autocatalytic deposit from a nonconductive substrate, during subsequent electroplating operations, owing to the application of excess current or a poor contact area.

burnishing—the smoothing of surfaces by rubbing, accomplished chiefly by the movement rather than the removal of the surface layer.

burnt deposit—a rough, noncoherent or otherwise unsatisfactory deposit produced by the application of an excessive current density and usually containing oxides or other inclusions.

bus (bus bar)—a rigid conducting section, for carrying current to the anode and cathode bars.

butler finish—a finish composed of fine, uniformly distributed parallel lines, having a characteristic luster usually produced with rotating wire brushes or cloth wheels with applied abrasives.

calomel half cell (calomel electrode)—a half cell containing a mercury electrode in contact with a solution of potassium chloride of specified concentration that is saturated with mercurous chloride (calomel).

calorizing—imparting resistance to oxidation to an iron or steel surface by heating in aluminum powder at 800 to 1000°C (1470 to 1830°F).

CASS test (copper accelerated salt spray)—an accelerated corrosion test for some electrodeposits and for anodic coatings on aluminum (see Test Method B 368\textsuperscript{2}).

cataphoresis—See electrophoresis.

cathode—the electrode in electrolysis at which positive ions are discharged, negative ions are formed, or other reducing actions occur.

\textsuperscript{2} Annual Book of ASTM Standards, Vol 02.05.
cathode efficiency—the current efficiency of a specified cathodic process.
cathode film—the layer of solution in contact with the cathode that differs in composition from that of the bulk of the solution.
cathode polarization—See polarization.
catholyte—the portion of the electrolyte in the vicinity of the cathode; in a divided cell the portion of the cathode side of the diaphragm.
cation—a positively-charged ion.
caustic dip—in diffusion coatings, a strongly alkaline treatment applied by dip or spray for neutralizing acid residues.
cementation—See pack cementation.
chelate compound—a compound in which the metal is contained as an integral part of a ring structure and is not readily ionized.
chelating agent—a compound capable of forming a chelate compound with a metal ion. See chelate compound.
chemical milling—the shaping of a work piece by immersion in an etchant employing a resist for selective removal of material.
chemical plating—deposition of a metal coating by chemical, non-electrolytic methods. See also immersion plate, contact plating.
chemical polishing—the improvement in surface smoothing of a metal by simple immersion in a suitable solution. See bright dip (nonelectrolytic).
chromating—the process for producing a conversion coating (q.v.) containing chromium compounds.
chromizing—a surface treatment at elevated temperatures, generally carried out in pack, vapor, or salt baths, in which an alloy is formed by the inward diffusion of chromium into the base metal.
cleaning—the removal of grease, oxides, or other foreign material from a surface.
alkaline cleaning—cleaning by means of alkaline solutions.
anodic or reverse cleaning—electrolytic cleaning in which the work is the anode.
cathodic or direct cleaning—electrolytic cleaning in which the work is the cathode.
diphase cleaning—cleaning by means of solutions that contain a solvent layer and an aqueous layer. Cleaning is effected both by solvent and emulsifying action.
direct current cleaning—See cathodic or direct cleaning.
extrolytic cleaning—alkaline cleaning in which a current is passed through the solution, the work being one of the electrodes.
emulsion cleaning—cleaning by means of solutions containing organic solvents, water, and emulsifying agents.
immersion—See soak cleaning.
reverse current cleaning—See anodic or reverse cleaning.
spray cleaning—cleaning by immersion without the use of current, usually in alkaline solution.
solvent cleaning—cleaning by means of organic solvents.
spraying—cleaning by means of spraying.
solution removal of grease, oxides, or other foreign material.
ultrasonic cleaning—cleaning by any chemical means aided by ultrasonic energy.
cleaning-emulsifiable solvent—two-stage cleaning system wherein a concentrate containing organic solvents and surface-active agents is applied to a surface, subsequently emulsified, and removed along with the soil, by water rinsing.
cleaning cycle—specific time and temperature to achieve desired depth of diffusion.
cleaning, diffusion—an alloy coating produced by applying heat to one or more coatings deposited on a metal substrate.
colloidal particle—an electrically-charged particle, generally smaller in size than 200 mµ, dispersed in a second continuous phase.
color anodizing—in anodizing aluminum, formation of a colored coating on aluminum where the colored compound, pigment, or dye is incorporated after the coating has been formed.
coloring—(1) the production of desired colors on metal surfaces by appropriate chemical or electrochemical action.
(2) light buffing of metal surfaces for the purpose of producing a high luster. Called “color buffing.”
complex ion—an ion composed of two or more ions or radicals, both of which are capable of independent existence, for example, cuprocyanide (Cu(CN)₃)₂⁻.
complexing agent—a compound that will combine with metallic ions to form complex ions. See complex ion.
composite coating—a coating consisting of deposits incorporating particles of another material. See also dispersion coating.
composite plate—an electrodeposit consisting of two or more layers of metal deposited successively.
concentration polarization—that part of the total polarization that is caused by changes in the activity of the potential-determining components of the electrolyte.
conductance—the capacity of a medium, usually expressed in mhos, for transmitting electric current. The reciprocal of resistance.
conducting salt—a salt added to the solution in order to increase its conductivity.
conductivity—specific conductance—the current transferred across unit area per unit potential gradient. In the metric system, K = amperes per square centimetre divided by volts per centimetre. The reciprocal of resistivity.
contact plating—deposition of a metal by the use of an internal source of current by immersion of the work in solution in contact with another metal.
contact potential—the potential difference at the junction of two dissimilar substances.
conversion coating—a coating produced by chemical or electrochemical treatment of a metallic surface that gives a superficial layer containing a compound of the metal, for example, chromate coatings on zinc and cadmium, oxide coating on steel.
conversion treatment—a chemical or electrochemical process producing a superficial layer containing a compound of the metal.

Corrodikote test—an accelerated corrosion test for electrodeposits (see Method B 380).

corrosion—(1) gradual solution or oxidation of a metal.  
(2) solution of anode metal by the electrochemical action in the plating cell.

coulomb—the quantity of electricity that is transmitted through an electric circuit in 1 s when the current in the circuit is 1 A. The quantity of electricity that will deposit 0.0011180 g of silver.

coulometer—an electrolytic cell arranged to measure the quantity of electricity by the chemical action produced in accordance with Faraday’s law.

covering power—the ability of a plating solution under a specified set of plating conditions to deposit metal on the surfaces of recesses or deep holes. (To be distinguished from throwing power.)

crazing—a network of fine hairline cracks in a coating.

critical current density—a current density above which a new and sometimes undesirable reaction occurs.

current density (cd)—current per unit area.

current efficiency—the proportion, usually expressed as a percentage, of the current that is effective in carrying out a specified process in accordance with Faraday’s law.

cut wire blasting—blasting with short, cut lengths of metal wire. See abrasive blasting.

cutting down—polishing or buffing for the purpose of removing roughness or irregularities.

deburring—the removal of burrs, sharp edges, or fins by mechanical, chemical, or electrochemical means.

decarburization—loss of carbon from the surface layer of a carbon containing alloy due to reaction with one or more chemical substances in a medium that contacts the surface.

decomposition potential—the minimum potential, exclusive of IR drop, at which an electrochemical process can take place at an appreciable rate.

degreasing—the removal of grease and oils from a surface. 
  solvent degreasing—degreasing by immersion in liquid organic solvent.
  vapor degreasing—degreasing by solvent vapors condensing on the parts being cleaned.

deionization—the removal of ions from a solution by ion exchange.

depolarization—a decrease in the polarization of an electrode at a specified current density.

depolarizer—a substance or a means that produces depolarization.

detergent—a surface-active agent that possesses the ability to clean soiled surfaces.  
  anionic detergent—a detergent that produces aggregates of negatively-charged ions with colloidal properties.  
  cationic detergent—a detergent that produces aggregates of positively-charged ions with colloidal properties.  
  nonionic detergent—a detergent that produces aggregates of electrically-neutral molecules with colloidal properties.

diaphragm—a porous or permeable membrane separating anode and cathode compartments of an electrolytic cell from each other or from an intermediate compartment.

diffusion—(1) spreading of a constituent in a gas, liquid, or solid tending to make the composition of all parts uniform;  
(2) the spontaneous movement of atoms or molecules to new sites within a material.

diffusion coating—an alloy coating produced by applying heat to one or more coatings deposited on a basis metal.

diffusion cycle—specific time and temperature to acquire a depth of diffusion and composition.

diffusion treatment (or coating)—(1) process of producing a surface layer (diffusion layer) by diffusion of another metal or non-metal into the surface of the basis material.  
(2) in electroplating, heat treatment applied to a work piece to achieve alloying or intermetallic compound formation between two or more coatings on a basis material.

dispersing agent—a material that increases the stability of a suspension of particles in a liquid medium.

dispersion coating—a coating consisting of particles of one material contained in a matrix of another metal or nonmetal.

divided cell—a cell containing a diaphragm or other means for physically separating the anolyte from the catholyte.

double salt—a compound of two salts that crystallize together in a definite proportion.

drag-in—the water or solution that adheres to the objects introduced into a bath.

drag-out—the solution that adheres to the objects removed from a bath.

ductility—the ability of a material to deform plastically without fracturing.

dummy (or dummy cathode)—a cathode in a plating solution that is not to be made use of after plating. Often used for removal or decomposition of impurities.

duplex coating—See composite plate.

electrochemical equivalent—the weight of an element, compound, radical, or ion involved in a specified electrochemical reaction during the passage of unit quantity of electricity, such as a Faraday, ampere-hour, or coulomb.

electrochemistry—the branch of science and technology which deals with transformations between chemical and electrical energy.

electrode—a conductor through which current enters or leaves an electrolytic cell, at which there is a change from conduction by electrons to conduction by charged particles of matter, or vice versa.

electrode potential—the difference in potential between an electrode and the immediately adjacent electrolyte referred to some standard electrode potential as zero.

dynamic E.P.—the electrode potential measured when current is passing between the electrode and the electrolyte.

equilibrium E.P.—a static electrode potential when the electrode and the electrolyte are in equilibrium with respect to a specified electrochemical reaction.

standard E.P.—an equilibrium electrode potential for an electrode in contact with an electrolyte in which all of the
components of a specified chemical reaction are in their standard states. The standard state for an ionic constituent is unit ion activity.

**Static E.P.**—the electrode potential measured when no net current is flowing between the electrode and the electrolyte.

**Electrodeposition**—the process of depositing a substance upon an electrode by electrolysis. See electroforming, electrolyplating, electrorefining, and electrowinning.

**Electroforming**—the production or reproduction of articles by electrodeposition upon a mandrel or mold that is subsequently separated from the deposit.

**Electrogalvanizing**—electrodeposition of zinc coatings.

**Electroless plating**—term in use but not recommended. See autocatalytic plating.

**Electrolysis**—production of chemical changes by the passage of current through an electrolyte.

**Electrolyte**—(1) a conducting medium in which the flow of current is accompanied by movement of matter. Most often an aqueous solution of acids, bases, or salts, but includes many other media, such as fused salts, ionized gases, some solids, etc. (2) a substance that is capable of forming a conducting liquid medium when dissolved or melted.

**Electrolytic cell**—an apparatus in which electrochemical reactions are produced by applying electrical energy, or which supplies electrical energy as a result of chemical reactions and which includes two or more electrodes and one or more electrolytes contained in a suitable vessel.

**Electromotive series**—a table that lists in order the standard electrode potentials of specified electrochemical reactions.

**Electrophoresis**—the movement of colloidal particles produced by the application of an electric potential.

**Electroplating**—the electrodeposition of an adherent metallic coating upon an electrode for the purpose of securing a surface with properties or dimensions different from those of the basis metal.

**Electropolishing**—the improvement in surface finish of a metal effected by making it anodic in an appropriate solution.

**Electrorefining**—the process of anodically dissolving a metal from an impure anode and depositing it cathodically in a purer form.

**Electrotyping**—the production of printing plates by electroforming.

**Electrowinning**—the production of metals by electrolysis with insoluble anodes in solutions derived from ores or other materials.

**Embrittlement, hydrogen**—See hydrogen embrittlement.

**Emf (electromotive force)**—an electrical potential.

**Emulsifying agent**—a substance that increases the stability of an emulsion.

**Emulsion**—a suspension of fine particles or globules of one or more liquids in another liquid.

**Energy efficiency**—the product of the current efficiency and the voltage efficiency for a specified electrochemical process.

**Equivalent conductivity**—in an electrolyte, the conductivity of the solution divided by the number of equivalents of conducting solute per unit volume, that is, the conductivity divided by the normality of the solution.

**Etch, n**—a roughened surface produced by a chemical or electrochemical means.

**Etch, v**—to dissolve unevenly a part of the surface of a metal.

**Faraday**—the number of coulombs (96,490) required for an electrochemical reaction involving one chemical equivalent.

**Filler**—a material used to increase the bulk of a product without adding to its effectiveness in functional performance.

**Filter aid**—an inert, insoluble material, more or less finely divided, used as a filter medium or to assist in filtration by preventing excessive packing of the filter cake.

**Flash (or flash plate)**—a very thin electrodeposited used for a final coating; for intermediate coatings of the same nature use strike.

**Floculate**—to aggregate into larger particles, to increase in size to the point where precipitation occurs.

**Flow brightening**—the melting of an electrodeposited, followed by solidification, especially of tin plate.

**Formula weight**—the weight, in grams, pounds, or other units, obtained by adding the atomic weights of all elemental constituents in a chemical formula.

**Free cyanide**—(1) true—the actual concentration of cyanide radical, or equivalent alkali cyanide, not combined in complex ions with metals in solution. (2) calculated—the concentration of cyanide, or alkali cyanide, present in solution in excess of that calculated as necessary to form a specified complex ion with a metal or metals present in solution. (3) analytical—the free cyanide content of a solution, as determined by a specified analytical method.

**Galvanic cell**—an electrolytic cell capable of producing electrical energy by electrochemical action.

**Galvanic series**—a list of metals and alloys arranged according to their relative potentials in a given environment. See electromotive series.

**Galvanizing**—application of a coating of zinc.

**Gassing**—the evolution of gases from one or more of the electrodes during electrolysis.

**Glass electrode**—a half cell in which the potential measurements are made through a glass membrane.

**Grinding**—the removal of metal by means of rotating rigid wheels containing abrasive.

**Grit blasting**—abrasive blasting with small irregular pieces of steel or malleable cast iron.

**Half cell**—an electrode immersed in a suitable electrolyte. It may be designed to yield a known constant potential, in which case unknown potentials may be measured against it; for example, the calomel half cell.

**Hard chromium**—chromium plate for engineering rather than decorative applications. Not necessarily harder than the latter.

**Hard-coating**—in anodizing aluminum, an anodic oxide coating on aluminum with a higher apparent density and thickness and a greater resistance to wear than conventional coatings.
Haring cell—A rectangular box of non-conducting material, with principal and auxiliary electrodes so arranged as to permit estimation of throwing power or electrode polarizations and potentials between them.

high lights—those portions of a metal article most exposed to buffing or polishing operations, and, hence, having the highest luster.

hot dip coating—a metallic coating obtained by dipping the basis metal into a molten metal.

Hull cell—a trapezoidal box of non-conducting material with electrodes arranged to permit observation of cathodic or anodic effects over a wide range of current densities.\(^3\)

hydrogen embrittlement—embrittlement of a metal or alloy caused by absorption of hydrogen which may occur, for example, during pickling, cathodic cleaning, electroplating, and autocatalytic plating processes.

hydrogen overvoltage—overvoltage associated with the liberation of hydrogen.

hydrophilic—(1) tending to absorb water. (2) tending to concentrate in the aqueous phase.

hydrophobic—(1) tending to repel water. (2) lacking affinity for water.

immersion plate—a metallic deposit produced by a displacement reaction in which one metal displaces another from solution, for example:

\[
\text{Fe} + \text{Cu}^{2+} \rightarrow \text{Cu} + \text{Fe}^{2+}
\]

indicator (pH)—a substance that changes color when the pH of the medium is changed. In the case of most useful indicators, the pH range within which the color changes is narrow.

inert anode—an anode that is insoluble in the electrolyte under the conditions prevailing in the electrolysis.

inhibitor—a substance used to reduce the rate of a chemical or electrochemical reaction, commonly corrosion or pickling.

interfacial tension—the contractile force of an interface between two phases. See surface tension.

ion—an electrified portion of matter of atomic or molecular dimensions.

ion exchange—a reversible process by which ions are interchanged between a solid and a liquid with no substantial structural changes of the solid.

IR drop—the voltage across a resistance in accordance with Ohm’s law:

\[
E = IR
\]

where:

- \(E\) = potential (voltage),
- \(I\) = current, and
- \(R\) = resistance.

karat—a twenty-fourth part by weight; thus 18-karat gold is 18/24 pure.

lapping—rubbing two surfaces together, with or without abrasives, for the purpose of obtaining extreme dimensional accuracy or superior surface finish.

leveling action—the ability of a plating solution to produce a surface smoother than that of the substrate.

limiting current density:(1) cathodic—the maximum current density at which satisfactory deposits can be obtained.

(2) anodic—the maximum current density at which the anode behaves normally, without excessive polarization.

mandrel—a form used as a cathode in electroforming; a mold or matrix.

masking—various materials applied to specific areas of parts to prevent coating from being deposited.

master alloy—an alloy rich in one or more desired addition elements that is added to a melt to raise the percentage of a desired constituent.

mat finish (matte finish)—a dull finish.

matrix—See mandrel.

measurement area—the surface area that is examined for conformance to one or more specified requirements.

mechanical cleaning—process for removing dirt, scale, or other deposits from surfaces through the use of mechanical means such as abrasive blasting.

mechanical plating—the application of a metallic layer by impacting spherical objects (for example glass beads) against the work surface in the presence of powdered coating metal and appropriate chemicals.

mechanical testing—determination of mechanical properties.

metal (or metallic) coating—generally denotes all metal coatings obtained by any method applied to a basis material.

metal distribution ratio—the ratio of the thickness of metal upon two specified areas of a cathode. See throwing power.

metal spraying—application of a metallic coating by projecting molten or heat-softened metal from a source (gun) onto the workpiece.

metallizing—(1) the application of a metal layer to the surface of non-conducting or non-metallic materials.

(2) the application of metallic coatings by nonelectrolytic procedures such as spraying of molten or heat-softened metal or deposition from the vapor phase.

microinch—one millionth of an inch, 0.000001 in. = 0.001 mil.

micrometer (µm)—one millionth of a meter, 0.001 mm.

micron—obsolete (see micrometer).

microthrowing power—the ability of a plating solution or a specified set of plating conditions to deposit metal in pores or scratches.

mil—one thousandth of an inch, 0.001 in. = 25.4 µm.

mill scale—the heavy oxide layer that forms during hot fabrication or heat treatment of metals.

modulated current plating—a method of electroplating in which the cathode current density is changed periodically. See pulse plating, ripple plating, periodic reverse plating.

motor-generator (MG set)—a machine that consists of one or more motors mechanically coupled to one or more generators. In plating, such a machine in which the generator delivers dc of appropriate amperage and voltage.

multilayer deposit—a deposit consisting of two or more layers of metal deposited successively, consisting of either different metals or layers of the same metal with different characteristics.

\(^3\) U. S. Patents 2,149,344; 2,760,928; and 2,801,963.
noble metal—A metal that does not readily tend to furnish ions, and therefore does not dissolve readily, nor easily enter into such reactions as oxidations, etc. The opposite of base metal.

Note 2—Since there is no agreement over the sign of electrode potentials, the words noble and base are often preferred because they are unambiguous.

nodule—a rounded projection formed on a cathode during electrodeposition.

orange peel—a finish resembling the dimpled appearance of an orange peel.

overvoltage—the irreversible excess of potential required for an electrochemical reaction to proceed actively at a specified electrode, over and above the reversible potential characteristics of that reaction.

oxidation—a reaction in which electrons are removed from a reactant. Sometimes, more specifically the combination of a reactant with oxygen.

oxidizing agent—a compound that causes oxidation, thereby itself becoming reduced.

pH—the cologarithm (negative logarithm) of the hydrogen ion activity, less precisely, concentration, of a medium, as determined by inhibitors or electrometric means.

pack cementation—in diffusion coatings, a coating process similar to pack carburizing that involves packing the parts to be coated into a retort with a carefully blended mixture of powders and then exposing the pack under hermetically sealed conditions or in an inert atmosphere to elevated temperatures.

passivating—a process for creating passivity (q.v.).

passivity—the condition of a metal that retards its normal reaction in a specified environment and associated with the assumption of a potential more noble than its normal potential.

peeling—the detachment or partial detachment of an electrodeposited coating from a basis metal or undercoat.

peen plating—See mechanical plating.

peening—See shot peening.

periodic reverse plating—a method of plating in which the current is reversed periodically. The cycles are usually no longer than a few minutes and may be much less.

pickle—an acid solution used to remove oxides or other compounds from the surface of a metal by chemical or electrochemical action.

pickling—the removal of oxides or other compounds from a metal surface by means of a pickle.

pit—a small depression or cavity produced in a metal surface during electrodeposition or by corrosion.

plastisol—a suspension of a finely divided resin in a plasticizer, that can be converted to a continuous film by the application of heat. Distinct from baking enamels etc., in that substantially all the original mixture becomes a part of the film; there is no significant evaporation of solvent. The films are usually much thicker than obtainable from coatings which depend on the evaporation of a volatile solvent.

plating range—the current density range over which a satisfactory electroplate can be deposited.

polarization—the change in the potential of an electrode during electrolysis, such that the potential of an anode always becomes more noble and that of a cathode less noble than their respective static potentials. Equal to the difference between the static potential and the dynamic potential.

polarizer—a substance or a means that produces or increases polarization.

polishing—the smoothing of a metal surface by means of the action of abrasive particles attached by adhesive to the surface of wheels or endless belts usually driven at a high speed.

pore—a discontinuity, essentially circular in cross section, in a coating extending through to the underlying coating or basis material.

primary current distribution—the distribution of the current over the surface of an electrode in the absence of polarization.

protective atmosphere—(1) a gas envelope surrounding the part to be brazed, welded, or thermal sprayed, with the gas composition controlled with respect to chemical composition, dew point, pressure, flow rate; (2) the atmosphere in a heat treating or sintering furnace formulated to protect the parts or compacts from oxidation, nitridation, or other contamination from the environment.

pulse plating—a method of electroplating in which the current is frequently interrupted or periodically decreased.

rack, plating—a frame for suspending and carrying current to articles during plating and related operations.

rectification—the conversion of alternating into direct current.

rectifier—a device that converts alternating into direct current by virtue of a characteristic permitting appreciable flow of current in one only direction.

reducing agent—a compound that causes reduction, thereby itself becoming oxidized.

reduction—a reaction in which electrons are added to a reactant. More specifically, the addition of hydrogen or the abstraction of oxygen. Such a reaction takes place, for example, at the cathode in electrolysis.

reflowing—See flow brightening.

relieving—the removal of material from selected portions of a colored metal surface by mechanical means, to achieve a multicolored effect.

resist, n—(1) a material applied to a part of a cathode or plating rack to render the surface nonconductive. (2) A material applied to a part of the surface of an article to prevent reaction of metal from that area during chemical or electrochemical processes.

retorts—containers fabricated from various metals in which parts are loaded to be diffusion coated or diffusion heat treated.

ripple (dc)—regular modulations in the dc output wave of a rectifier unit, or a motor-generator set, originating from the harmonics of the ac input system in the case of a rectifier, or from the harmonics of the induced voltage of a motor generator set.

ripple plating—a method of electroplating in which the current is changed periodically by superimposing surges,
ripples, pulses, or alternating current to the d-c electroplating current.

robber—See thief.

roll straightening—in diffusion coatings, straightening of metal stock of various shapes by passing it through a series of staggered rolls, the rolls usually being in horizontal and vertical planes.

sacrificial protection—the form of corrosion protection wherein one metal corrodes in preference to another, thereby protecting the latter from corrosion.

sand blasting—abrasive blasting with sand.

saponification—the alkaline hydrolysis of fats whereby a soap is formed; more generally, the hydrolysis of an ester by an alkali with the formation of an alcohol and a salt of the acid portion.

satin finish—a surface finish that behaves as a diffuse reflector and which is lustrous but not mirrorlike.

scale—an adherent oxide coating that is thicker than the superfluous film referred to as tarnish.

sealed (anodic) coating—in anodizing aluminum, an anodic oxide coating on aluminum that has been treated in an aqueous or steam medium resulting in reduced porosity of the coating.

sealing of anodic coating—a process which, by absorption, chemical reaction, or other mechanism, increases the resistance of an anodic coating to staining and corrosion, improves the durability of colors produced in the coating, or imparts other desirable properties.

sequestering agent—an agent that forms soluble complex compounds with, or sequesters, a simple ion, thereby suppressing the activity of that ion. Thus, in water treatment the effects of hardness can be suppressed by adding agents to sequester calcium and magnesium. See chelating agent.

shelf roughness—roughness on upward facing surfaces where undissolved solids have settled on parts during a plating operation.

shield, n—a nonconducting medium for altering the current distribution on an anode or cathode.

shield, v—to alter the normal current distribution on an anode or cathode by the interposition of a nonconductor.

shot blasting—blasting with small spherical objects, such as metallic shot, propelled against a metallic surface. See abrasive blasting.

shot peening—a process whereby hard, small spherical objects (such as metallic shot) are propelled against a metallic surface for the purpose of introducing compressive stresses into that surface, hardening it or obtaining decorative effects.

siliconizing—in diffusion coatings, diffusion of silicon into solid metal at elevated temperatures.

slurry—a suspension of solids in water.

spotting out—the delayed appearance of spots and blemishes on plated or finished surfaces.

stalagmometer—an apparatus for determining surface tension. The mass of a drop of a liquid is measured by weighing a known number of drops or by counting the number of drops obtained from a given volume of the liquid.

standoff—in abrasive blasting, distance from blast nozzle to part when abrasive blasting.

stardusting—an extremely fine form of roughness on the surface of a metal deposit.

stop-off—See resist.

stopping off—the application of a resist to any part of an electrode—cathode, anode, or rack.

stray current—current through paths other than the intended circuit, such as through heating coils or the tank.

strike: (1) n—a thin film of metal to be followed by other coatings.
(2) n—a solution used to deposit a strike.
(3) v—to plate for a short time, usually at a high initial current density.

strip, n—a process or solution used for the removal of a coating from a basis metal or an undercoat.

strip, v—to remove a coating from the basis metal or undercoat.

substrate—in diffusion coatings, material on which coating is deposited.

superimposed ac—a form of current in which an alternating current component is superimposed on the direct plating current.

surface active agent—a substance that affects markedly the interfacial or surface tension of solutions even when present in very low concentrations.

surface tension—that property, due to molecular forces, that exists in the surface film of all liquids and tends to prevent the liquid from spreading.

tank voltage—the total voltage between the anode and cathode of a plating bath or electrolytic cell during electrolysis. It is equal to the sum of: (1) the equilibrium reaction potential, (2) the IR drop, and (3) the electrode potentials.

tarnish—corrosion products in the form of thin films or spots that do not protrude significantly from the surface of the metallic coating; for example, reaction products of copper from oxygen or reduced sulfur.

thief—an auxiliary cathode so placed as to divert to itself some current from portions of the work which would otherwise receive too high a current density.

throwing power—the improvement of the coating (usually metal) distribution over the primary current distribution on an electrode (usually cathode) in a given solution, under specified conditions. The term may also be used for anodic processes for which the definition is analogous.

total cyanide—the total content of cyanide expressed as the radical CN\(^{-}\), or alkali cyanide whether present as simple or complex ions. The sum of both the combined and free cyanide content of a solution.

transference (or transport, or migration)—the movement of ions through the electrolyte associated with the passage of the electric current.

transference number (transport number)—the proportion of the total current carried by the ions of a given kind.

trees—branched or irregular projections formed on a cathode during electrodeposition especially at edges and other high current density areas.

tripoli—fragile and dustlike silica used as an abrasive.

tumbling—See barrel finishing.

vapor deposition:
chemical—ion, induced by heat or gaseous reduction of a vapor condensing on the substrate.

physical—a process for depositing a coating by evaporating and subsequently condensing an element or compound, usually in a high vacuum.

vibratory finishing—a process for deburring and surface finishing in which the product and an abrasive mixture are placed in a container and vibrated.

void—a defective area in which a part of the basis material or underlayer is visible after final coating.

voltage efficiency—the ratio, usually expressed as a percentage, of the equilibrium reaction potential in a given electrochemical process to the bath voltage.

water break—the appearance of a discontinuous film of water on a surface signifying nonuniform wetting and usually associated with a surface contamination.

wet blasting—a process for cleaning or finishing by means of a slurry of abrasive in water directed at high velocity against the work pieces.

wetting agent—a substance that reduces the surface tension of a liquid, thereby causing it to spread more readily on a solid surface.

whiskers—metallic filamentary growths, often microscopic, sometimes formed during electrodeposition and sometimes spontaneously during storage or service, after finishing.

work (plating)—the material being plated or otherwise finished.

DEFINITIONS RELATING SPECIFICALLY TO PLATING ON PLASTIC SUBSTRATES

cleaning—the removal of unwanted materials, such as mold release agents.

conditioning—the conversion of a surface to a suitable state for successful treatment in succeeding steps.

nucleation—the preplating step in which a catalytic material, often a palladium or gold compound, is absorbed on a surface to act as sites for initial stages of deposition.

post-nucleation—the step where, if necessary, the catalyst is converted to its final form. This is the final step prior to electroless plating.

sensitization—the absorption of a reducing agent, often a stannous compound, on the surface.

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Standard Test Method of Corrosion Testing of Decorative Electrodeposited Coatings by the Corrodkote Procedure

1 This method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on General Test Methods.

The original work in developing the Corrodkote procedure was initiated by the American Electroplaters' Society Research Committee, Project No. 15.


3 Annual Book of ASTM Standards, Vol 03.02.

4 Annual Book of ASTM Standards, Vol 02.05.


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Designation: B 380 – 97 (Reapproved 2002)

Standard Test Method of Corrosion Testing of Decorative Electrodeposited Coatings by the Corrodkote Procedure

This standard is issued under the fixed designation B 380; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This standard describes the Corrodkote2 method of evaluating the corrosion performance of copper/nickel/chromium and nickel/chromium coatings electrodeposited on steel, zinc alloys, aluminum alloys, plastics and other substrates.

NOTE 1—The following ASTM standards are not requirements. They are reference for information only: Practice B 537, Specification B 456, Test Method B 602, and Specification B 604.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 117 Practice for Operating Salt Spray (Fog) Apparatus3
B 456 Specification for Electrodeposited Coatings of Copper Plus Nickel Plus Chromium and Nickel Plus Chromium4
B 537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure4
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings4
B 604 Specification for Decorative Electroplated Coatings of Copper/Nickel/Chromium on Plastics4
D 1193 Specification for Reagent Water5

3. Summary of Method

3.1 The test is conducted by applying a slurry containing corrosive salts to test specimens, allowing the slurry to dry, and exposing the specimens coated with the slurry to a high relative humidity for a specified period of time.

4. Significance and Use

4.1 Nickel/chromium and copper/nickel/chromium electrodeposited coatings are widely used for decorative and protective applications. The Corrodkote test provides a method of controlling the quality of electroplated articles and is suitable for manufacturing control, as well as research and development.

5. Apparatus

5.1 The apparatus shall consist of a humidity chamber, specimen supports, provision for heating the chamber, and provisions for air circulation in the chamber.

5.2 Drops of moisture that might accumulate on the ceiling or cover of the chamber of specimen supports shall not be permitted to fall on the specimens being tested.

NOTE 2—Suitable apparatus may be constructed from salt-spray equipment by eliminating fog-spray nozzles, substituting water for the salt solution in the reservoir, adding a manifold for bubbling air through the water in the reservoir, and adding a fan for circulating the air in the chamber with the fan discharge directed across the surface of the water in the reservoir.

5.3 Materials of construction shall not affect the corrosiveness of the test.

6. Procedure

6.1 Corrodkote Slurry—Prepare the Corrodkote slurry in a glass beaker by dissolving 0.035 g of reagent grade cupric nitrate (Cu(NO3)2·3H2O), 0.165 g of ACS reagent grade ferric chloride (FeCl3·6H2O), and 1.0 g of ACS reagent grade ammonium chloride (NH4Cl) in 50 mL of water conforming to Specification D 1193, Type IV. Stir 30 g of water-washed, ceramic-grade kaolin into the solution. Mix the slurry thoroughly and allow it to stand for about 2 min so that the kaolin will become saturated. Mix the slurry thoroughly just before using.

6.1.1 An alternative method of preparing the Corrodkote slurry is as follows. Weigh out 2.5 g of cupric nitrate (Cu(NO3)2·3H2O), and dissolve and dilute with distilled water in a volumetric flask to exactly 500 mL. Weigh out 2.50 g of ferric chloride (FeCl3·6H2O), and dissolve and dilute with...
distilled water in a second volumetric flask to exactly 500 mL. (The ferric chloride solution shall be kept in a dark place and stoppered with a rubber or glass stopper when not in use.) Weigh out 50.0 g of ammonium chloride (NH₄Cl) and dissolve and dilute with distilled water in a volumetric flask to exactly 500 mL. Then measure out exactly 7.0 mL of the cupric nitrate solution, 33.0 mL of ferric chloride solution, and 10.0 mL of the ammonium chloride solution; place these in a beaker to which 30.0 g of kaolin is now added. Stir with a glass stirring rod.

6.2 Test Specimens—The type and number of test specimens to be used, as well as the criteria for evaluation of the test results, shall be defined in the specifications covering the material or product being tested, or shall be mutually agreed upon by the manufacturer and the purchaser.

6.3 Preparation of Test Specimens—Metallic and metallic-coated test specimens may be solvent cleaned before testing, using a suitable solvent such as alcohol, ethyl ether, acetone or petroleum ether. Solvents that are corrosive or will deposit protective films shall not be used.

6.4 Slurry Application—Apply the Corrodkote slurry to the specimen using a clean paint brush. Dip the brush in the Corrodkote slurry and with a circular motion, completely cover the specimen. Then smooth out the coating by brushing lightly in one direction. Allow the specimens to dry at room temperature and at a relative humidity less than 50% for 1 h before placing them in the humidity chamber. Should it be desirable to cut test specimens from parts or from preplated, or otherwise coated steel sheet, the cut edges shall be protected by coating them with paint, wax, tape, or other effective media so that the development of a galvanic effect between such edges and the adjacent plated or otherwise coated-metal surfaces, is prevented.

6.5 Position of Specimens During Test—The position of the specimens in the humidity chamber during the test is not critical as long as they do not touch each other and the supports do not touch the test areas that have been coated with the Corrodkote slurry.

6.6 Conditions in the Humidity Chamber—The exposure zone of the humidity chamber shall be maintained at 38 ± 2°C. The relative humidity of the exposure zone of the chamber shall be maintained between 80 and 90% shall not produce condensation on any of the parts.

Note 3—A fan or blower may be used in the chamber to maintain uniform temperature and humidity. The amount of air circulation in the chamber necessary to maintain these conditions must be determined for each chamber. An Abbeon certified hygrometer Model No. 167, or its equivalent, is a suitable instrument for measuring the relative humidity of the exposure zone of the humidity chamber.

6.7 Test Cycle—Exposure in the humidity chamber shall be continuous for periods up to 20 h. A single test period up to 20 h shall be considered as one cycle. Continuous operation implies that the humidity chamber be closed and operated continuously except for the short interruptions necessary to place or remove test specimens. Operations shall be so scheduled that these interruptions are held to a minimum. When more than one test cycle of 20 h or less is specified, the test specimen shall be treated as outlined in 6.9 after each test cycle. Fresh slurry shall be applied for each succeeding cycle. After the last test cycle, the test specimen shall be treated as outlined in 6.9.

6.8 Period of Test—The period of each test cycle as well as the number of test cycles required, shall be as specified in the specifications for the material or product being tested, or as mutually agreed upon between the manufacturer and the purchaser.

6.9 Cleaning of Testing Specimens—Treat the specimen after each test cycle as outlined in 6.9.1. After the last test cycle and if the specimen is to be inspected between test cycles, treat the specimens as outlined in 6.9.1 and 6.9.2.

6.9.1 Remove the specimen from the humidity chamber. Clean the specimen using fresh running water and a piece of clean cheesecloth or suitable synthetic sponge to remove all of the slurry. A mild abrasive such as a water paste of tripoli may be used to remove any adhering materials.

6.9.2 Since the cleaning operation described in 6.9.1 will remove any corrosion products, it will be necessary to redevelop the points of failure. This may be done in several ways, such as exposure for 4 h in the salt spray cabinet described in Practice B 117, exposure for 24 h in a humidity cabinet operated at 38°C and 100% relative humidity with condensation, or some other method that will cause corrosion of the basis metal without any significant damage to the coating.

Note 4—Corrosion products visible on the test specimen with the slurry in place are not necessarily caused by points of failure in the coating. Particles of metal accidentally deposited on the test specimen before humidity exposure will also cause visible corrosion products.

7. Evaluation of Results

7.1 After redevelopment of points of failure, as outlined in 6.9.2, make a careful and immediate examination for the extent of corrosion of the test specimen or for other failures as required in the specifications covering the material or product being tested or by agreement between the manufacturer and the purchaser.

8. Report

8.1 The report shall contain the following information, unless otherwise prescribed in the specifications covering the material or product being tested:

8.1.1 The title, designation, and year of issue of this method,

8.1.2 Readings of temperature within the exposure zone of the humidity chamber,

8.1.3 Readings of relative humidity within the exposure zone of the chamber,

8.1.4 Type of specimen and its dimensions or number, or description of part,

8.1.5 Exposure period for each cycle and number of cycles,

8.1.6 Results of all inspections, and

8.1.7 Method of redeveloping corrosion spots.

9. Precision and Bias

9.1 The Corrodkote test provides reliable results if the
composition of the slurry, the humidity in the chamber, the
temperature, and other factors discussed in this standard are
carefully controlled. The precision and bias are being deter-
mined in interlaboratory test programs.

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Standard Practice for Evaluating Apparent Grain Size and Distribution of Cemented Tungsten Carbides

This standard is issued under the fixed designation B 390; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This procedure for the visual comparison and classification of the apparent grain size and distribution of cemented tungsten carbides is limited to cemented tungsten carbides that contain approximately 6, 10, and 18 % cobalt.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 657 Test Method for Metallographic Determination of Microstructure in Cemented Carbides
B 665 Practice for Metallographic Sample Preparation of Cemented Tungsten Carbides

3. Terminology

3.1 Definitions of Terms Specific to This Standard:
3.1.1 apparent grain size—the average diameter of a tungsten carbide particle as measured on an etched metallographically polished surface of a specimen of sintered cemented carbide. The apparent grain size is expressed in micrometres and is generally reported as a range.

4. Significance and Use

4.1 The apparent size and distribution of tungsten carbide grains in cemented carbides affects the material’s wear resistance and fracture. For a given chemical composition, an increase in the average grain size will result in increased toughness and decreased wear resistance. This practice illustrates representative micro-structures for a wide range of tungsten carbide-cobalt grades. This is not intended to be used as a specification for carbide grades; producers and users may use the micrographs and the grain size chart as a guide in developing their own specifications.

5. Specimen Preparation

5.1 Specimens shall be prepared for metallographic examination in accordance with a procedure suitable for this type of cemented carbides.

NOTE 1—A suitable procedure is described in Method B 657 and Practice B 665.

6. Procedure

6.1 Examine metallographically the entire surface of an etched specimen at a magnification of 1500 diameters. Select a representative area and make a comparison with the photomicrographs in Fig. 1, Fig. 2, and Fig. 3.

6.2 The nominal analyses of the cemented tungsten carbide samples that were used to prepare the nine photomicrographs shown in Fig. 1, Fig. 2, and Fig. 3 are as follows:

6.2.1 Fig. 1—Tungsten carbide plus 6 % cobalt,
6.2.2 Fig. 2—Tungsten carbide plus 10 % cobalt, and
6.2.3 Fig. 3—Tungsten carbide plus 18 % cobalt.

6.3 The grain size for each of these specimens may be designated as follows:

Type F—Fine grain,
Type M—Medium grain, and
Type C—Coarse grain.

7. Evaluation

7.1 The grain size rating of a cemented tungsten carbide specimen is accomplished by selecting the photomicrographs in Fig. 1, Fig. 2, or Fig. 3 that most nearly match the field observed on the specimen being evaluated (see Appendix X1).

7.2 A range of numerical values for various concentrations of tungsten carbide particles, even though arbitrary, is suggested in Table 1.

8. Report

8.1 After visual comparison of a specimen with the photomicrographs contained in this practice, the apparent grain size of a particular specimen shall be rated relative to one or more of the photomicrographs. Cobalt contents should always be specified. For example, a cemented tungsten carbide specimen containing approximately 6 % cobalt, having a fine grain size and a grain size distribution similar to the one shown in Fig. 1(a) would be reported as Type 6-F. The “F” indicates a fine grain size and the “6” identifies the cobalt content.
9. Precision and Bias

9.1 Visual comparison of cemented carbide microstructures with the Carbide Grain Size Chart is subjective. No precision and bias statement can be made for this practice.

10. Keywords

10.1 apparent grain size; cemented carbides; cemented tungsten carbides; grain size distribution; hardmetals; microstructure; powder metallurgy
FIG. 3 Apparent Grain Size of Tungsten Carbide plus 18 % Cobalt (1500×)

<table>
<thead>
<tr>
<th>Figure Number</th>
<th>Grain Size</th>
<th>High Concentration, µm</th>
<th>Low Concentration, µm</th>
<th>Rating</th>
</tr>
</thead>
<tbody>
<tr>
<td>1(a)</td>
<td>fine</td>
<td>0.25 to 1.00</td>
<td>1.25 to 2.00</td>
<td>6-F</td>
</tr>
<tr>
<td>1(b)</td>
<td>medium</td>
<td>2.00 to 5.00</td>
<td>0.75 to 1.00</td>
<td>6-M</td>
</tr>
<tr>
<td>1(c)</td>
<td>coarse</td>
<td>4.00 to 6.00</td>
<td>0.50 to 3.00</td>
<td>6-C</td>
</tr>
<tr>
<td>2(a)</td>
<td>fine</td>
<td>0.25 to 0.75</td>
<td>1.00 to 2.00</td>
<td>10-F</td>
</tr>
<tr>
<td>2(b)</td>
<td>medium</td>
<td>2.00 to 4.00</td>
<td>0.50 to 1.00</td>
<td>10-M</td>
</tr>
<tr>
<td>2(c)</td>
<td>coarse</td>
<td>3.00 to 6.00</td>
<td>0.75 to 2.00</td>
<td>10-C</td>
</tr>
<tr>
<td>3(a)</td>
<td>fine</td>
<td>0.25 to 0.50</td>
<td>1.00 to 2.00</td>
<td>18-F</td>
</tr>
<tr>
<td>3(b)</td>
<td>medium</td>
<td>1.50 to 4.00</td>
<td>0.50 to 1.00</td>
<td>18-M</td>
</tr>
<tr>
<td>3(c)</td>
<td>coarse</td>
<td>3.00 to 6.00</td>
<td>0.75 to 2.00</td>
<td>18-C</td>
</tr>
</tbody>
</table>
X1. CARBIDE GRAIN SIZE CHART

X1.1 Fig. X1.1 illustrates the relationship of grain sizes from 1 to 10 µm as observed at a magnification of 1500 diameters (1 µm = 0.00003937 in.).

FIG. X1.1 Relationship of Grain Sizes at Magnification of 1500 Diameters
Standard Test Method for Transverse Rupture Strength of Cemented Carbides

This standard is issued under the fixed designation B 406; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the transverse rupture strength of cemented carbides.

1.2 The values stated in inch-pound units are to be regarded as the standard. The SI values in parentheses are provided for information only.

1.3 This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ISO Standard:
ISO-3327 Hardmetals—Determination of Transverse Rupture Strength

3. Significance and Use

3.1 This test method is used as a means of determining the quality of cemented carbide grade powders by measuring their sintered strength. It is performed on test specimens prepared to specified shape, dimensions, and surface finish; test specimens may be prepared from finished parts if size permits. There is no known standard material for this test method. The transverse rupture strength of cemented carbides is not a design value.

3.1.1 Most commercial cemented carbides have mechanical behavior that is best classified as brittle (negligible ductility). Fracture strengths are dependent on internal or surface flaws. Examples of incoherent internal flaws are macropores, Type B porosity, and inclusions of foreign particles. Such flaws are randomly distributed spatially and in size within the sintered material. This imparts a statistical nature to any transverse rupture strength measurement.

3.1.2 The stress distribution in a beam in three-point loading is non-uniform. It increases linearly along the span to a maximum at the center, and varies linearly through any section from compression on the top to tension on the bottom. The maximum tensile stress therefore occurs at center span in the bottom most fibers of the sample, and is defined as the transverse rupture strength at failure. Failure is initiated at a random flaw site, which is most probably not coincident with the maximum stress. This imparts an additional statistical nature to transverse rupture strength measurements.

4. Apparatus

4.1 Either a specially adapted machine for applying the load or a special fixture suitable for use with a conventional load-applying machine may be used. In either case, the apparatus shall have the following parts:

4.1.1 Two ground-cemented-carbide cylinders 0.250 ± 0.001 in. (6.35 ± 0.02 mm) in diameter, at least 0.500 in. (13 mm) in length with the long axes parallel, and center to center spacing of 0.563 ± 0.005 in. (14.3 ± 0.1 mm).

4.1.2 A movable member (free to move substantially only in a line perpendicular to the plane established by the axes of the two cylinders) containing a 0.4 ± 0.05-in. (10 ± 1.3-mm) cemented-tungsten-carbide ball or a ground-cemented-carbide cylinder of the same dimensions as, and with axis parallel to, those of the two previously mentioned cylinders (see 4.1.1). This ball or cylinder shall be so positioned that movements of the member will cause the ball or cylinder to contact a specimen placed on the two lower cylinders at the midpoint of the span between them.

4.1.3 The apparatus shall be so constructed that the application of a sufficient load to the movable member to effect breaking of a specimen will not cause appreciable deflection of the line of movement of the movable member and the plane established by the two fixed cylinders. The apparatus shall be capable of applying sufficient load to break the specimen. The apparatus shall be capable of registering the load required (within ±1 % of the load) to break the specimen. The cemented-tungsten-carbide ball and cylinders shall not show permanent deformation after use.

5. Specimen Size

5.1 The cemented carbide specimens shall be ground to the following dimensions: 0.200 ± 0.010 in. (5.00 ± 0.25 mm) thick by 0.250 ± 0.010 in. (6.25 ± 0.25 mm) wide by 0.750 in. (19.0 mm) minimum long.

6. Specimen Preparation

6.1 Specimens shall be ground to a surface finish of 15 µin.
(0.381 µm) rms maximum on four sides, and to the tolerances specified in Section 5. All grinding marks shall be parallel to the length, 0.750 in. (19.05 mm), axis. Opposite ground faces shall be parallel within 0.001 in. (0.0254 mm). The two faces that are perpendicular to the length axis need not be ground. Careful grinding techniques should be used to prevent various forms of surface cracking (flaws) that will degrade the measured strength. Long-established practice recommends the use of soft resin bonded diamond wheels, and copious quantities of coolant. For surface grinding, no pass shall exceed 0.0005 in. (0.0127 mm) in depth.

6.2 The four edges of the specimen representing the intersection of the ground faces shall be chamfered or honed to a maximum of 0.010 in. (0.25 mm) by 45 degrees. Any grinding marks shall be parallel to the long axis of the specimen.

6.3 Each specimen shall be measured to within 0.001 in. (0.02 mm) in both directions perpendicular to the length axis. Adjacent ground sides shall be at right angles to each other within 2 degrees.

6.4 Each specimen shall be visually inspected after grinding. Any specimen on which cracks, chips, or obvious structural defects appear on the ground surfaces shall be eliminated from the test.

7. Procedure

7.1 Visually examine the cylinders and ball in the fixture for cracks, chips, deformation, or misalignment and check the movable member for freedom of movement. Correct any defects prior to use.

7.2 Place a properly prepared and measured specimen on the fixture with the long axis perpendicular to the cylinders and with the 0.250-in. (6.25-mm) face resting on the two cylinders. Then adjust the movable member so that the ball or upper cylinder contacts the specimen without substantial impact. If a ball is used, place the specimen so that the ball touches the midpoint of the specimen width. Apply the load at a rate not exceeding 350 lbf/s (1.5 kN/s). Fracture should occur within the middle one third of the span between the supporting cylinders on the tension side of the specimen. Record the number of pounds required to cause fracture.

7.3 Perform all tests at room temperature but not lower than 65°F (18°C).

7.4 Five specimens shall be tested.

8. Calculation

8.1 Calculate the transverse rupture strength as follows:

\[ S = \frac{3PL}{2bh^2} \]  

where:

\[ S = \text{transverse rupture strength, psi (MPa)}, \]
\[ P = \text{load, lb (N) required to fracture}, \]
\[ L = \text{length of span, in. (mm)}, \]
\[ b = \text{specimen width, in. (mm)}, \]
\[ h = \text{specimen thickness, in. (mm)}. \]

9. Report

9.1 One, but only one, of the five values obtained will be considered invalid if its deviation from the mean of the other four values is excessive as determined by the following:

9.1.1 Take the average of the other four values.

9.1.2 Find the deviation of the values from the average.

9.1.3 Total the four deviations.

9.1.4 If the value omitted has a greater deviation than the total of the four other deviations, it is dropped. Otherwise, all five values must be considered valid.

9.1.5 Example:

<table>
<thead>
<tr>
<th>Values Determined,</th>
<th>Deviation from Average of 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>180 000</td>
<td>20 000</td>
</tr>
<tr>
<td>200 000</td>
<td>0</td>
</tr>
<tr>
<td>205 000</td>
<td>5 000</td>
</tr>
<tr>
<td>215 000</td>
<td>15 000</td>
</tr>
<tr>
<td>150 000</td>
<td>(50 000)</td>
</tr>
</tbody>
</table>

Average of 5 190 000
Average of 4 200 000

The last value is dropped. Had it been 160 000 to 240 000 psi, it would have to be included in the average.

9.2 Report the transverse rupture strength as the mean of the valid values. Also report the standard deviation of these valid values. If less than five valid values are used in calculating the mean, the number of valid values used in the calculation of the mean and the standard deviations of these valid values are to be referenced in the report.

10. Precision and Bias

10.1 The statistical nature of transverse rupture strength in cemented carbide was discussed in 3.1. This causes the precision and bias of the test to be inseparable from statistical nature of the material behavior. This dilemma is compensated for by requiring the reporting of the standard deviation of the test values.

11. Keywords

11.1 cemented carbides; fracture strength; hardmetals; tensile stress; transverse rupture strength
Standard Test Method for
Apparent Density of Non-Free-Flowing Metal Powders Using
the Carney Funnel

1. Scope

1.1 This test method covers a procedure for determining the
apparent density of non-free-flowing metal powders. It is
designed for those metal powders that do not freely flow
through the Hall flowmeter funnel.

1.2 This standard does not purport to address all of the
safety concerns, if any, associated with its use. It is the
responsibility of the user of this standard to establish appro-
priate safety and health practices and determine the applica-

2. Referenced Documents

2.1 ASTM Standards:
B 215 Practices for Sampling Finished Lots of Metal Pow-
ders
B 243 Terminology of Powder Metallurgy
B 873 Test Method for Measuring the Volume of Apparent
Density Cup Used in Test Methods B 212, B 329, and
B 417

3. Terminology

3.1 Definitions—Terms in Terminology B 243 are appli-
cable to this test method.

4. Summary of Test Method

4.1 A container of definite volume is filled with non-free-
flowing powder under controlled conditions. The mass of
powder per unit volume is determined and reported as apparent
density, Carney.

5. Significance and Use

5.1 This test method provides a guide for evaluation of the
apparent density physical characteristic of powders. The den-
sity measured bears a relationship to the mass of powder that
will fill a fixed volume die cavity when parts are being made.
The degree of correlation between the results of this test
method and the quality of powders in use will vary with each
particular application.

6. Apparatus

6.1 Powder Funnel—A Carney funnel (Fig. 1).
6.2 Density Cup—A cylindrical brass cup (Fig. 2) having a
capacity of 25 cm³. The actual cup volume shall be determined
according to Test Method B 873.
6.3 Stand—A stand (Fig. 1) to support the powder funnel
concentric with the density cup so that the bottom of the
powder funnel is approximately 25 mm (1 in.) above the top of
the density cup when the apparatus is assembled as shown in
Fig. 1.
6.4 Base—A level, vibration-free base to support the pow-
der flowmeter.
6.5 Balance, having a capacity of at least 200 g and a
sensitivity of 0.01 g.
6.6 Wire, approximately 2.5 mm (0.10 in.) in diameter by
150 mm (6 in.) in length.

7. Test Specimen

7.1 The test specimen shall consist of a volume of approxi-
mately 30 to 40 cm³ of metal powder obtained in accordance
with Practice B 215.
7.2 The test specimen shall be tested as sampled. Note,
however, that temperature, moisture, oils, stearic acid, stear-
ates, waxes, and so forth may alter the characteristics of the
powder.

8. Procedure

8.1 Weigh the empty density cup to the nearest 0.1 g or,
alternatively, place the empty density cup on the balance and
tare the balance to zero.
8.2 Load the test specimen carefully into the flowmeter
funnel and permit it to run into the density cup through the
discharge orifice. If necessary, it may be agitated or pushed by
use of the length of wire but take care to prevent the wire from
entering the density cup. The density cup should not be moved
during the filling operation.
8.3 When the powder completely fills and overflows the
periphery of the density cup, rotate the funnel approximately
90° in a horizontal plane so that the remaining powder falls
away from the cup.
8.4 Using a nonmagnetic spatula, with the blade held

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1 This test method is under the jurisdiction of ASTM Committee B09 on Metal
Powders and Metal Powder Products and is the direct responsibility of Subcommit-
tee B09.02 on Base Metal Products.


2 Annual Book of ASTM Standards, Vol 02.05.

3 The flowmeter funnel, density cup, and stand are available from Acu Powder
International, LLC.
perpendicular to the top of the cup, level off the powder flush with the top of the density cup. Take care to avoid jarring the apparatus at any time.

8.5 After the leveling operation, tap the density cup lightly on the side to settle the powder to avoid spilling in transfer.

8.6 Transfer the filled density cup to the balance and weigh to the nearest 0.1 g to determine the mass \( M \) of powder.

9. Calculation

9.1 Calculate the apparent density as follows:

\[
\text{Apparent density, g/cm}^3 = \frac{M}{V} \tag{1}
\]

where:

- \( M \) = mass of powder in the density cup in grams
- \( V \) = volume of the density cup in cubic centimetres.

10. Report

10.1 Report the results as apparent density, Carney, to the nearest 0.01 g/cm³.

11. Precision and Bias

11.1 Precision—The precision of this test method has not been determined by a statistically valid interlaboratory test. Results obtained by eight laboratories testing a sample of tin powder suggest the following:

11.1.1 Repeatability \( r = 1 \% \) (tin powder)—Duplicate analysis of a tin powder by the same operator and same apparatus should not differ by more than 1 % at the 95 % confidence level.

11.1.2 Reproducibility \( R = 4 \% \) (tin powder)—The difference between two single and independent results obtained by different operators working in different laboratories on tin powder should not differ by more than 4 % at the 95 % confidence level.

11.2 Bias—Since there is no accepted reference material for determining the bias for the procedure in Test Method B 417 for measuring apparent density (Carney), bias has not been determined.

12. Keywords

12.1 apparent density; Carney; flowmeter funnel; metal powders
Standard Test Method for Particle Size Distribution of Refractory Metal Powders and Related Compounds by Turbidimetry

This standard is issued under the fixed designation B 430; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of particle size distribution of refractory metal powders with a turbidimeter (1). Experience has shown that this test method is satisfactory for the analysis of elemental tungsten, molybdenum, rhenium, tantalum metal powders, and tungsten carbide powders. Other refractory metal powders, for example, elemental metals, carbides, and nitrides, may be analyzed using this test method with caution as to significance until actual satisfactory experience is developed. The procedure covers the determination of particle size distribution of the powder in two conditions:

1.1.1 As the powder is supplied (as-supplied), and

1.1.2 After the powder has been de-agglomerated by rod milling (laboratory milled) according to Practice B 859.

1.2 Where dual units are given, inch-pound units are to be regarded as standard.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 330 Test Method for Fisher Number of Metal Powders and Related Compounds

B 821 Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis

B 859 Practice for De-Agglomeration of Refractory Metal Powders and Their Compounds Prior to Particle Size Analysis

E 456 Terminology Relating to Quality and Statistics

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Method

3.1 A uniform dispersion of the powder in a liquid medium is allowed to settle in a glass cell. A beam of light is passed through the cell at a level having a known vertical distance from the liquid level. The intensity of the light beam is determined using a photo cell. This intensity increases with time as sedimentation of the dispersion takes place.

3.2 The times at which all particles of a given size have settled below the level of the transmitted light beam are calculated from Stokes’ law for the series of sizes chosen for the particle size analysis.

3.3 The intensity of the light beam at these times is measured as percent of the light transmitted through the cell with the clear liquid medium. The size distribution in the powder can be calculated from these relative intensities using the Lambert-Beer law in the modified form (also see Refs 2, 3, 4).

\[ \Delta W_{1-2} = \frac{d_m (\log I_{d1} - \log I_{d2})}{100} \]  

where \( I_{d1} \) and \( I_{d2} \) are the intensities measured at the times when all particles having diameters larger than \( d_1 \) and \( d_2 \) respectively have settled below the level of the light beam. \( d_m \) is the arithmetic mean of particle sizes \( d_1 \) and \( d_2 \), and \( \Delta W_{1-2} \) refers to the relative weight for the particle size range between \( d_1 \) and \( d_2 \). Values of \( \Delta W \) are determined for each of the particle size ranges chosen. The sum of these values is \( \sum \Delta W \). The weight percent of particles in the size range from \( d_1 \) to \( d_2 \) can then be calculated as:

\[ \text{Weight, } \% = \frac{(\Delta W_{1-2} - \sum \Delta W)}{2} \times 100 \]  

4. Significance and Use

4.1 Knowledge of the particle size distribution of refractory metal powders is useful in predicting powder-processing behavior, and ultimate performance of powder metallurgy parts. Particle size distribution is closely related to the flowability, compressibility, and die-filling characteristics of a powder, as well as to the final structure and properties of the finished parts. However, the degree of correlation between the results of this test method and the quality of powders in use has not been fully determined quantitatively.

4.2 This test method is suitable for manufacturing control
and research and development in the production and use of refractory metal-type powders, as indicated in 1.1.

4.3 Reported particle size measurement is a function of both the actual particle dimension and shape factor, as well as the particular physical or chemical properties being measured. Caution is required when comparing data from instruments operating on different physical or chemical parameters or with different particle size measurement ranges. Sample acquisition, handling, and preparation also can affect reported particle size results.

5. Apparatus

5.1 Turbidimeter (5)—The recommended instrument is one using a cell rectangular in cross section, approximately 50 mm high, 40 mm wide, and 10-mm sedimentation medium thickness, and having optically parallel faces.

5.2 Millivolt Recorder, 0 to 10-mV range, 10-in. (254-mm) wide strip chart, 0 to 100 graduations, 120 in./h (50 mm/min) chart speed, or microammeter with 0 to 100 graduations, 15-µA full scale, 4.5-mV full scale.

NOTE 1—While a 120-in./h (50-mm/min) chart speed is recommended, other speeds may be satisfactory.

5.3 Ultrasonic Cleaning Tank, with tank dimensions approximately 5 by 5 by 3 in. (127 by 127 by 76 mm) deep and an output of 50 W, or approximately 3½ by 3½ by 2½ in. (89 by 89 by 67 mm) deep and an output of 25 W.7

5.4 Glass Vial, nominal 2 dram, flat-bottom, with a tight-fitting cap. The vial shall be approximately 2 in. (51 mm) in height with a 5/8-in. (16-mm) outside diameter and approximately a 5/32-in. (0.8-mm) wall.8

6. Reagents

6.1 Sedimentation Medium:

6.1.1 Base Medium, distilled or deionized water (see Note 4).

6.1.2 Use either one of the following as recommended in Guide B 821:

6.1.2.1 Daxad (No. 11)9—Dissolve 25 mg in 1 L of base medium.

6.1.2.2 Sodium Hexametaphosphate—Dissolve 0.1 g in 1 L of base medium.

NOTE 2—Use water that is pure. Do not store the sedimentation medium longer than a week, and do not use rubber tubing in any storage container. Clean thoroughly all sedimentation medium containers every week.

7. Preparation of Apparatus

7.1 Warm up equipment by turning on the light source and recorder for a minimum of 1 h prior to use.

7.2 Fill the cell with sedimentation medium to a height sufficient to cover the light beam path by at least 10 mm and place the cell in the turbidimeter (Note 3). If a microammeter is used to measure light intensity, adjust the light transmission to 100 % using the diaphragm. If a millivolt recorder is used, adjust the potentiometer so that the photovoltaic cell output is 10 mV or 100 %. In this case, the diaphragm is not adjusted and is completely open.

NOTE 3—For convenience in filling the cell to the proper height, inscribe a line on each face of the cell at the desired liquid-level height. The height of fall is usually 25 mm. To determine the location of the line, the center of the light beam path must be established and 25 mm added to this value.

7.3 After the instrument is adjusted to 100 % light transmission through the sedimentation cell and medium, move the cell carriage until light is passing through a reference glass held in another slot of the cell carriage. Read and record the percent of reference light transmission. Having been selected to have approximately 70 to 95 % of the transmission of the sedimentation cell and medium, the reference glass will indicate 100 % light transmission through the sedimentation cell when the recorder reads this value through the reference cell.

8. Calculation of Times at Which Light Intensity is Measured

8.1 The times at which the light transmission values should be read are calculated from Stokes’ law. A uniform 1-µm interval should be used in making measurements through the 10-µm size and, depending upon the particular powder, either 1-µm or 5-µm intervals thereafter. The form of Stokes’ law used is as follows:

$$t = (18 \times 10^5 Nh)d^3(\rho_s - \rho_m)g$$

where:

- $t$ = time, s,
- $N$ = viscosity of settling medium at ambient temperature, P (Note 4),
- $h$ = height of fall, cm (distance from liquid level height to midpoint of light beam),
- $d$ = diameter of particle, µm ($d_1$, $d_2$, et al),
- $\rho_s$ = theoretical density of the powder being tested (for tungsten, use 19.3 g/cm$^3$),
- $\rho_m$ = density of settling medium at ambient temperature (Note 4), and
- $g$ = gravitational constant (980 cm/s$^2$).

NOTE 4—The viscosity and density values at different temperatures that are used for the sedimentation medium in this procedure are the same as for pure water. Some viscosity (from the Handbook of Chemistry and Physics, 65th Edition, CRC Press, 1984) and density (from Metrological Handbook 145, NIST, 1990) values are given as follows:

<table>
<thead>
<tr>
<th>Temperature, °C</th>
<th>Viscosity, cP</th>
<th>Density, g/cm$^3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>18</td>
<td>64.4</td>
<td>1.0530</td>
</tr>
<tr>
<td>19</td>
<td>66.2</td>
<td>1.0270</td>
</tr>
<tr>
<td>20</td>
<td>68.0</td>
<td>1.0020</td>
</tr>
<tr>
<td>21</td>
<td>69.8</td>
<td>0.9779</td>
</tr>
</tbody>
</table>

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8 The recommended instrument is a Cenco Photometer (not made anymore) of original or modified designs or any proven equivalent instrument. A schematic diagram of the Photometer is shown in the papers referenced at the end of this test method. Copies of detailed drawings of an acceptable instrument are available from ASTM Headquarters. Order ADJA0430. A fabricated instrument can be secured from WAB Instruments Co., 5171 Hickory Dr., Cleveland, OH 44124.

9 The 69800-Q1, Model S, Type G, Speedomax W, or XL630 Series recorder as made by the Leeds and Northrup Co., have been found satisfactory.

10 Two-dram Titeseal vials, as made by Chemical Rubber Co., Cleveland, OH, have been found satisfactory.

11 Ultrasonic tank Model Nos. 2 or 12 as made by Branson Instrument Co., Stanford, CT, have been found satisfactory.

12 The 69800-Q1, Model S, Type G, Speedomax W, or XL630 Series recorder as made by Bransonic Instrument Co., Stamford, CT, have been found satisfactory.

13 Ultrasonic tank Model Nos. 2 or 12 as made by Bransonic Instrument Co., Stamford, CT, have been found satisfactory.

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15UMB.65th Edition, CRC Press, 1984) and density (from Metrological Handbook 145, NIST, 1990) values are given as follows:

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<tr>
<td>21</td>
<td>69.8</td>
<td>0.9779</td>
</tr>
</tbody>
</table>
9. Conditioning (or De-agglomeration) of the Powder Prior to Analysis

9.1 For as-supplied particle size distribution determinations, this step is not needed.

9.2 For laboratory-milled particle size distribution determinations, follow the procedure specified in Practice B 859.

 NOTE 5—Since milled powder has a greater tendency than as-supplied powder to pick up moisture and oxidize, the analysis procedure should be initiated immediately after milling is completed. This is particularly important if the powder is to be dispersed using the 5-min hand-shake procedure (see Section 8) where a difference can be seen between determinations made in succession on powders having significant amounts of 1-µm size powder. This difference, related to the size of the powder, is greater for finer powders. For all practical purposes, however, two runs can be made in succession on each milled powder. If more than two runs on the same milled powder are desired using the 5-min shake procedure, provisions may be taken to lessen (elimination is not possible) the effect of humidity on the milled powder such as immediate splitting of the sample and storage under dry nitrogen or in a desiccator. If the 5-min ultrasonic procedure is used to disperse the powder for analysis, the milled powder may be stored for several days without any effect being seen in the distribution results.

10. Dispersion

10.1 The powder, either as supplied, or laboratory milled in accordance with 9.2, may be dispersed in the sedimentation medium either by a 5-min ultrasonic treatment procedure or by a 5-min continuous hand-shake procedure. The 5-min ultrasonic treatment procedure is the preferred and recommended procedure.

 NOTE 6—The weight of the sample used should give a preferred initial light transmission of between 20 and 30 %. Transmissions between 15 and 40 % are acceptable. If it is desired to change the initial light transmission, reweigh another sample, increasing or decreasing the weight accordingly.

 NOTE 7—Table 1 gives likely sample weight ranges for lab-milled tungsten powders having known Fisher sub-sieve sizer average particle diameters in the as-supplied condition. (See Test Method B 330.) These likely sample weight ranges apply for powders that have been lab-milled tungsten powders known having Fisher sub-sieve sizer average particle diameters in the as-supplied condition, or other powders, proper weights should be determined by trial and error.

10.2 The 5-min ultrasonic treatment dispersion procedure is as follows:

10.2.1 Fill the vial with 2 mL of sedimentation medium or to a height of approximately ¼ in. (7.0 mm). Add weighed amount of powder and cap the vial. Place into the ultrasonic tank, handholding the vial for 5 min.

 NOTE 8—Depth of the liquid in the tank should be 1 ½ to 2 in. (approximately 40 to 50 mm) from the bottom. Liquid in the tank is distilled or deionized water, room temperature, with a small amount of detergent. A 1-min warm-up of the ultrasonic tank is recommended prior to vial immersion.

 NOTE 9—If any of the powder sample is on the walls of the vial, the liquid may be swirled before and during the ultrasonic treatment to rinse the powder down into the bottom. The vial need not be held in a stationary position nor perpendicular to the bottom. Depth of immersion and location of the vial are generally at the center portion of the tank, but may vary. Where cavitation within the vial is noticeable, as evidenced by rapid agitation of the powder dispersion, the bottom of the vial could even be at the surface of the tank liquid. Agitation within the vial should be noticeable. Where agitation is not evident within the vial, the vial should be moved until agitation is evident. The vial generally is immersed to a depth where powder dispersion is at or below tank liquid level with the vial bottom not closer than ½ in. (about 10 mm) to the bottom of the tank. Immersion is generally not within 1 in. (about 25 mm) from any tank wall. During ultrasonic treatment, a slight tingling feeling at the fingertips, where they touch the vial, might be present. Also, while vial and contents are slightly warmed during treatment, no temperature correction need be made because of the subsequent dilution in the sedimentation cell.

10.2.2 Wipe dry or rinse the outside of the vial immediately after ultrasonic treatment to prevent ultrasonic tank liquid contamination in the sedimentation cell.

10.2.3 Quantitatively transfer the powder dispersion into an empty sedimentation cell. Thoroughly rinse the vial, making sure that all the powder is in the cell.

 NOTE 10—A 250 or 500-mL plastic wash bottle that has had the nozzle straightened to an upright position has been found to be convenient to flush the vial of remaining traces of powder as it is inverted over and into the sedimentation cell at a slight angle. Care must be taken not to flush the vial so strongly that liquid and powder splashes out over the sedimentation cell. (See Note 2 regarding cleansing of this equipment.)

 NOTE 11—Usually no difficulty is encountered in the transfer of fine powders into the sedimentation cell. However, where coarse powders are ultrasonically dispersed, there is a tendency for some of this powder to remain in the vial and the transfer is a little more difficult. Experience will solve this problem.

10.2.4 Fill the sedimentation cell to the proper height (see Note 3). Adjustment of the final liquid level may be done by using an eye-dropper filled with sedimentation medium.

10.2.5 Close the cell and redisperse the powder in the sedimentation medium by holding it at the top and the bottom and turning it upside down and shaking it for approximately 5

<table>
<thead>
<tr>
<th>Micrometre Sizes Read</th>
</tr>
</thead>
<tbody>
<tr>
<td>Each µm interval from 1 to 5</td>
</tr>
<tr>
<td>Each µm interval from 1 to 10</td>
</tr>
<tr>
<td>Each µm interval from 1 to 20 plus at 15, 20, and 25</td>
</tr>
<tr>
<td>Each µm interval from 1 to 25 plus at 20 and 25</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>TABLE 1 Lab-Milled Tungsten Metal Powders</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter by Fisher Sub-Sieve Sizer, µm</td>
</tr>
<tr>
<td>----------------------------------------</td>
</tr>
<tr>
<td>0 to 1.0</td>
</tr>
<tr>
<td>1.0 to 1.8</td>
</tr>
<tr>
<td>1.8 to 3.0</td>
</tr>
<tr>
<td>3.0 to 5.0</td>
</tr>
<tr>
<td>5.0 to 7.0</td>
</tr>
<tr>
<td>7.0 to 12.0</td>
</tr>
</tbody>
</table>
to 10 s to remove any powder that has settled to the bottom. Then give the cell 1½ to 2-min second shake (not quite as vigorous as described in 10.3.2) ending with a gentle end-over-end complete 360° facewise rotation that allows the air bubble contained in the cell to “wipe” both faces for approximately 10 s to rehomogenize the contents. Continue this facewise rotation until the cell is placed in the instrument. During this time, visually check the contents of the cell for uniformity of dispersion and recheck the liquid level.

10.2.6 Proceed immediately to step 11.1.

10.3 The 5-min hand-shake dispersion procedure is as follows:

10.3.1 Fill the sedimentation cell with sedimentation medium to approximately 1 to 2 mm below the graduated line that signifies a 25-mm height of fall.

10.3.2 Transfer the weighed sample into the sedimentation cell. Close the cell with a cover and, holding it at the top and bottom between the forefingers and the thumb, shake vigorously for 4½ to 4¾ min. Shake the cell by moving it in an arc of 12 to 15 in. (305 to 380 mm) in length, back and forth approximately one cycle per second. The sedimentation medium movement is distinctly heard as the cell is shaken. After the vigorous shake, remove the cover from the cell and adjust the liquid level to the graduated line using an eye dropper filled with sedimentation medium.

10.3.3 Continue by performing steps listed in 10.2.5 and 10.2.6 except to eliminate 1½ to 2-min second shake.

11. Procedure

11.1 During the last 2 to 10 s of the gentle shake, start the chart paper motor, recheck the reference light transmission value (see 7.3), adjusting the instrument accordingly, and move the cell carriage to block completely the light transmission. The reading of the millivolt recorder or microammeter changes from 100 to 0 % transmission. As soon as the transmission is 0 %, cautiously drop (Note 12) the cell into the carriage, and then immediately position in the light path. Exercise care to position the cell vertically (top to bottom) in the carriage before moving it into the light path and that the cell carriage is recentered before starting the run.

11.2 As the powder settles, record the light transmission values either manually at the appropriate times determined in 8.1, or continuously through the use of the potentiometer and millivolt recorder. If a recorder is used, read the light transmission for the appropriate times from the graph paper after sedimentation is complete.

Note 12—If the cell is dropped too hard, it might crack. To reduce this possibility, place the thumb and forefinger of the other hand around the cell carriage at the side so they are on top of the block that the cell will sit on during the run. Then drop the cell into the cell carriage, and as soon as it hits the thumb and forefinger, remove them, allowing the cell to have a reduced shock.

12. Calculations

12.1 Use Eq 2 to calculate the $\Delta W$ values from the light intensity measured (either in percent or millivolts) at the upper and lower limit of each chosen range of particle diameters and from the arithmetic means of the particle range.

13. Report

13.1 The report may be of a single determination or an average with or without the individual determinations being listed, and should be so identified.

13.2 The report shall be identified with the condition of the powder analyzed, that is, either “as supplied” or “lab milled”, and, if dispersed by the 5-min hand-shake procedure, with “hand-shake.” Conversely, if the powder is dispersed by the 5-min ultrasonic treatment procedure, only the powder condition is identified.

13.3 Values shall be reported in weight percent to the nearest 0.1 % for each micrometre size interval calculated.

14. Precision and Bias

14.1 Precision—At this time no full interlaboratory study on the precision of this test method exists. However, the user of this test method may get some indication of its precision from ASTM Research Report No. B9-1007, which presents the results of a study done in only three laboratories on tungsten and tungsten carbide powders with the two dispersants included in 6.1 (analyzed according to Practice E 691).

14.1.1 The within-laboratory repeatability limit, $r_a$, as defined by Terminology E 456, was found to be 3 to 5 weight % in each individual particle size range.

14.1.2 The between-laboratory reproducibility limit, $R_a$, as defined by Terminology E 456, was found to be 5 to 7 weight % in each individual particle range.

14.2 Bias—No absolute method of determining particle size distribution is universally recognized. Therefore, it is not possible to discuss the bias of results by this test method.

15. Keywords

15.1 de-agglomeration; laboratory milled; light-attenuation; particle size distribution; Photelometer; rod milled; sedimentation; turbidimeter

10 Supporting data are available from ASTM Headquarters. Request RR: B9-1007.
REFERENCES


Standard Specification for
Sintered Bronze Bearings (Oil-Impregnated)\textsuperscript{1}

This standard is issued under the fixed designation B 438/B 438M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (\(\epsilon\)) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This specification covers sintered bronze, oil-impregnated bearings made primarily from elemental copper, tin, and graphite powders. The manufacturer may, at his discretion, use prealloyed bronze powder in the blend.

1.2 This specification covers the following variables:

1.2.1 Grades—Available in four bronze base composition grades identifiable by different graphite contents.

1.2.2 Type—Grades 1 and 2 are available in four types described by specific density ranges.

1.3 Bearings ordered to this specification will normally be sized after sintering and will be impregnated with a lubricating oil unless otherwise specified by print.

1.4 The values stated in either inch-pound or SI units are to be regarded separately as standard. Within the text, the SI units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the specification.

2. Referenced Documents

2.1 ASTM Standards: \textsuperscript{2}

B 328 Test Method for Density, Oil Content, and Interconnected Porosity of Sintered Metal Structural Parts and Oil-Impregnated Bearings

E 9 Test Methods of Compression Testing of Metallic Materials at Room Temperature

2.2 Government Standards:

MIL-PRF-6085 Lubricating Oil: Instrument, Aircraft, Low Volatility\textsuperscript{3}

MIL-PRF-17331 Lubrication Oil: Steam Turbine and Gear, Moderate Service\textsuperscript{3}

\textsuperscript{1} This specification is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.04 on Bearings.


\textsuperscript{2} For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard’s Document Summary page on the ASTM website.

\textsuperscript{3} Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111–5094, Attn: NPODS.

3. Ordering Information

3.1 Orders for parts under this specification shall include the following information:

3.1.1 Dimensions and tolerances (Section 9),

3.1.2 Grade and type,

3.1.3 Density specification (Table 1 and Table 2), and

3.1.4 Oil type.

4. Materials and Manufacture

4.1 Sintered bronze bearings shall be made by molding or briquetting metal powder mixtures to the proper density. The green bearing shall be sintered at a time–temperature relationship to produce a microstructure that is essentially alpha bronze and contains no tin-rich phases visible at 300\(\times\). Sintered bronze bearings are normally sized after sintering to maintain the dimensional characteristics required of the bearing. After sizing and inspection, they are impregnated with a lubricating oil unless otherwise specified.

5. Chemical Composition

5.1 The material shall conform to the requirements as to the chemical composition prescribed in Table 3.

6. Physical Properties

6.1 Density—The density of bearings supplied impregnated with lubricant shall be within the limits prescribed in Table 1 and Table 2, when determined in accordance with Test Method B 328.

6.2 Oil Content—Oil content of bearings shall not be less than shown in Table 4 for each grade and type when determined in accordance with Test Method B 328.

7. Mechanical Properties

7.1 The manufacturer and purchaser shall agree on a representative number of specimens for tests.

7.2 Radial Crushing Force—Radial crushing force shall be determined by compressing the test specimen between two flat steel surfaces at a “no load” speed not greater than 0.2 in./min [5.0 mm/min], the direction of the load being normal to the longitudinal axis of the specimen. The point at which the load
drops as a result of the first crack shall be considered the crushing strength. This test shall be applied to plain cylindrical bearings. Flanged bearings shall be tested by cutting off the flange and compressing the two sections separately. Each section shall meet the minimum strength requirements prescribed in Table 5.

### TABLE 1 Density Requirements (Oil Impregnated)

<table>
<thead>
<tr>
<th>Type</th>
<th>Density, g/cm³</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grades 1 and 2</td>
<td>1</td>
</tr>
<tr>
<td>Grades 1, 2, and 4</td>
<td>2</td>
</tr>
<tr>
<td>Grades 1 and 2</td>
<td>3</td>
</tr>
<tr>
<td>Grades 1 and 2</td>
<td>4</td>
</tr>
</tbody>
</table>

⁴ Maximum density limit of 6.2 g/cm³ has been established on Type 1 to ensure meeting an oil content of 27% minimum. Satisfactory bearings can also be produced between Type 1 and Type 2. These bearings have slightly higher strength constants and slightly lower oil content.

### TABLE 2 Density Requirements (Oil Impregnated)

<table>
<thead>
<tr>
<th>Type</th>
<th>Density, g/cm³</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grade 3</td>
<td>1</td>
</tr>
<tr>
<td>Grade 3</td>
<td>2</td>
</tr>
</tbody>
</table>

### TABLE 3 Chemical Requirements (Composition, %)

<table>
<thead>
<tr>
<th>Element</th>
<th>Grade 1</th>
<th>Grade 2</th>
<th>Grade 3</th>
<th>Grade 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper</td>
<td>87.2–90.5</td>
<td>85.7–90.0</td>
<td>82.8–88.3</td>
<td>80.9–88.0</td>
</tr>
<tr>
<td>Tin</td>
<td>9.5–10.5</td>
<td>9.5–10.5</td>
<td>9.2–10.2</td>
<td>9.5–10.5</td>
</tr>
<tr>
<td>Graphite</td>
<td>0–0.3</td>
<td>0.5–1.8</td>
<td>2.5–5.0</td>
<td>0.50–1.75</td>
</tr>
<tr>
<td>Iron, max</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>Total other elements</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
<td>0.5</td>
</tr>
</tbody>
</table>

**Note:**—Grade 4 to be used for special government needs.

### TABLE 4 Oil Content (Oil Content, Volume %, Min)

<table>
<thead>
<tr>
<th>Type</th>
<th>Grade 1</th>
<th>Grade 2</th>
<th>Grade 3</th>
<th>Grade 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>27</td>
<td>25</td>
<td>11⁵</td>
<td>...</td>
</tr>
<tr>
<td>2</td>
<td>19</td>
<td>17</td>
<td>...</td>
<td>17</td>
</tr>
<tr>
<td>3</td>
<td>12</td>
<td>9</td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>4</td>
<td>9</td>
<td>7</td>
<td>...</td>
<td>...</td>
</tr>
</tbody>
</table>

⁵ At 3% graphite, Type 1 will contain 14% min oil content. At 5% graphite, Type 2 will contain only a minimal amount of oil.

### TABLE 5 Strength Constant K (Strength Constants, Min), A psi [MPa]

<table>
<thead>
<tr>
<th>Type</th>
<th>Grade 1</th>
<th>Grade 2</th>
<th>Grade 3</th>
<th>Grade 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>15 000 [105]</td>
<td>13 000 [90]</td>
<td>10 000 [70]</td>
<td>... [...]</td>
</tr>
<tr>
<td>2</td>
<td>26 000 [180]</td>
<td>23 000 [160]</td>
<td>15 000 [105]</td>
<td>23 000 [160]</td>
</tr>
<tr>
<td>3</td>
<td>37 000 [255]</td>
<td>30 000 [205]</td>
<td>... [...]</td>
<td>... [...]</td>
</tr>
<tr>
<td>4</td>
<td>40 000 [275]</td>
<td>34 000 [235]</td>
<td>... [...]</td>
<td>... [...]</td>
</tr>
</tbody>
</table>

A For the K value specification to be valid, wall thickness must be less than one third of the outside diameter.

where:
- \( P \) = radial crushing force, pounds [N],
- \( D \) = outside diameter of bearing, inches [mm],
- \( T \) = wall thickness of bearing, inches [mm],
- \( K \) = strength constant as shown in Table 5 for grade and type specified, psi [MPa], and
- \( L \) = length of bearing, inches [mm].

7.2.2 Concerning spherical bearings, sample parts from a lot will be machined to a straight wall and radially crushed to calculate the \( K \) value. Sample parts from the same lot will be radially crushed as is (whole part). By correlation, the minimum radial crush value will be established on the whole bearing and so specified as the minimum radial crush value for the part.

8. Chemical Analysis

8.1 If required by purchase agreement, one sample for chemical analysis shall be taken from each lot. A representative sample of chips may be obtained by milling, drilling, filing, or crushing a bearing with clean dry tools without lubrication. To obtain oil-free chips, the parts selected for test shall have the oil extracted in accordance with Test Method B 328 if necessary.

8.2 The chemical analysis shall be made in accordance with the methods prescribed in Vol 03.05 of the Annual Book of ASTM Standards or by any other method agreed upon between the manufacturer and the purchaser.

9. Dimensions and Tolerances

9.1 Permissible variations in dimensions shall be within the limits specified on the drawings accompanying the order or shall be within the limits specified on the order.

10. Workmanship, Finish, and Appearance

10.1 Bearings shall be uniform in composition, clean, and conform to applicable drawings.

11. Sampling

11.1 Lot—Unless otherwise specified, a lot shall consist of parts of the same form and dimensions made from powders of the same composition, formed and sintered under the same conditions, and submitted for inspection at one time.

12. Inspection

12.1 Unless otherwise specified, inspection of parts supplied on contract shall be made by the purchaser at the destination.

13. Rejection

13.1 Parts that fail to conform to the requirements of this specification may be rejected. Rejection should be reported to the producer or supplier promptly and in writing.

14. Certification

14.1 When specified in the purchase order or contract, a producer’s certification shall be furnished to the purchaser that the parts were manufactured, sampled, tested, and inspected in accordance with this specification and have been found to meet...
the requirements. When specified in the purchase order or contract, a report of the test results shall be furnished.

14.2 The purchase order must specify whether or not the certification includes chemistry.

14.3 Upon request of the purchaser in the contract or order, the certification of an independent third party indicating conformance to the requirements of this specification may be considered.

15. Supplementary Requirements

15.1 For some materials, supplementary requirements may be specified. Usually these apply only when specified by the purchaser in the inquiry, contract, or order. These supplementary requirements shall appear separately.

15.2 Special Government Requirements—Requirements that are special to government needs are listed in 15.2.1 through 15.2.9.

15.2.1 Materials shall conform to Table 3, Grade 4. Contractor shall furnish a percent composition analysis on an oil-free basis for each lot showing the percentage for each element as specified in Table 3. Bearing shall conform to this specification and supporting military specification as applicable.

15.2.2 High-grade nongumming petroleum lubricants, such as MIL-PRF-6085, MIL-PRF-17331 (Military Symbol 2190–TEP), or as specified on referenced military standard specification sheets shall be used to impregnate the bearings.

15.2.3 When specified, a first-article inspection shall be performed on bearings. Four samples shall be made available for first-article inspection and tested for chemical requirements, density, porosity, radial crushing strength, oil excretion, and dimensional characteristics as specified herein, Test Method B 328, FED-STD-151, or in an otherwise specified document. Any defect or failure shall be cause for rejection of the lot. Waivers for minor defects may be addressed to the contracting officer.

15.2.4 When procured from a contractor versus the actual manufacturer, a certificate of quality conformance (COQC) supplied by the manufacturer of the bearing may be furnished in lieu of actual performance of such testing by the contractor, provided lot identity has been maintained and can be demonstrated to the Government. The certificate shall include the name of the contractor, contractor number, name of manufacturer, NSN, item identification, name of the component or material, lot number, lot size, dimensions, date of testing, test method, individual test results, and specification requirements.

15.2.5 When specified in the contract or purchase order, packaging and marking shall be completed in accordance with the provisions of the contract.

15.2.6 Oil excretion of the bearing shall be verified by placing the bearing in the chamber of a preheated oven. Oven temperature shall be nominally 300°F [149°C]. Exposure shall be 5 min. During the period, beads shall exude uniformly from the bearing surface. Lack of appreciable sweating of the lubricant on the bearing surface will be cause for rejection. Lubricant content may be verified using Test Method B 328.

15.2.7 Unless otherwise specified, the contractor is responsible for testing. The contractor may use their own or any other suitable facility for the performance of testing and inspection, unless an exception is stated. The Government reserves the right to perform an inspection set forth herein to assure supplies and sources conform to the prescribed requirements.

15.2.8 Records of examination and tests performed by or for the contractor shall be maintained and made available to the Government by the contractor for a period of three years after delivery of the products and associate material.

15.2.9 All requirements shall be as specified herein. Reference military standard specification sheets shall take precedence unless otherwise specified in the contract or purchase order.

16. Related Specifications

16.1 MPIF Standards:


16.2 ISO Standards:

16.2.1 2795—Plain Bearings Made From Sintered Material—Dimensions and Tolerances.

16.2.2 5755—Sintered Metal Material Specifications.

17. Keywords

17.1 density; K strength constant; oil content; oil-impregnated bearings; porosity

APPENDIXES

(Nonmandatory Information)

X1. EXPLANATORY INFORMATION

X1.1 Design Information

X1.1.1 In calculating permissible loads, the operating conditions, housing conditions, and construction should be considered. The maximum static bearing load should not exceed 8500 psi [60 MPa] of projected bearing area (length times inside diameter of bearing) for this material. This figure is 75% of the value for the compression deformation limit [yield strength, permanent set of 0.001 in. [0.025 mm] for specimens 1⅛ in. [30 mm] in diameter and 1 in. [25 mm] in length] as determined in accordance with Test Methods E 9.
X1.2 Permissible Loads

X1.2.1 Permissible loads for various operating conditions are given in Table X1.1.

X1.3 Dimensional Tolerances

X1.3.1 Commercial dimensional tolerances are included in Table X1.2. Closer tolerances can be held with special tooling or processing, or both.

X1.3.2 The commercial tolerances listed in Table X1.2 are intended for bearings with a 4 to 1 maximum length to inside diameter ratio and a 24 to 1 maximum length to wall thickness ratio.

X1.3.3 Fig. X1.1, Fig. X1.2, and Fig. X1.3 illustrate standard sleeve, standard flange bearings, and standard thrust bearings, respectively. Their dimensions are referenced throughout the tolerance tables. Standard chamfer tolerances are also listed in Table X1.2.

X1.4 Press Fits

X1.4.1 Plain cylindrical journal bearings are commonly installed by press fitting the bearing into a housing with an insertion arbor. For housings rigid enough to withstand the press fit without appreciable distortion and for bearings with wall thickness approximately one eighth of the bearing outside diameter, the press fits shown in Table X1.3 are recommended.

X1.5 Running Clearance

X1.5.1 Proper running clearance for sintered bearings depends to a great extent on the particular application. Therefore, only minimum recommended clearances are listed in Table X1.4. The maximum running clearances will automatically be held within good design practice for average conditions. It is assumed that ground steel shafting having a recommended finish of 4 to 16 root mean square (rms) will be used and all bearings will be oil impregnated.

### TABLE X1.1 Permissible Loads

<table>
<thead>
<tr>
<th>Shaft Velocity, ft/min [m/s]</th>
<th>Permissible Loads, psi [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Grades 1, 2, 3, and 4</td>
</tr>
<tr>
<td></td>
<td>Type I</td>
</tr>
<tr>
<td>50 to 100 [0.25–0.50], incl</td>
<td>500 [3.4]</td>
</tr>
<tr>
<td>Over 100 to 150 [0.50–0.75], incl</td>
<td>325 [2.2]</td>
</tr>
<tr>
<td>Over 150 to 200 [0.75–1.00], incl</td>
<td>250 [1.7]</td>
</tr>
<tr>
<td>Over 200 [1.00]</td>
<td>A</td>
</tr>
</tbody>
</table>

Note: 1—With a shaft velocity of less than 50 ft/min [0.25 m/s] and a permissible load greater than 1000 psi [0.15 MPa], an extreme pressure lubricant should be used.

Note 2—With good heat dissipation and heat removal techniques, higher PV ratings can be obtained.

### TABLE X1.2 Commercial Dimensional Tolerances

<table>
<thead>
<tr>
<th>Inside Diameter, d, and Outside Diameter, D, in. [mm]</th>
<th>Total Diameter Tolerances, in. [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Over</td>
<td>Through</td>
</tr>
<tr>
<td>...</td>
<td>1 [25]</td>
</tr>
<tr>
<td>...</td>
<td>1 [25]</td>
</tr>
<tr>
<td>...</td>
<td>1 [25]</td>
</tr>
<tr>
<td>...</td>
<td>1 [25]</td>
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<tr>
<td>...</td>
<td>1 [25]</td>
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<td>...</td>
<td>1 [25]</td>
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<tr>
<td>...</td>
<td>1 [25]</td>
</tr>
<tr>
<td>...</td>
<td>1 [25]</td>
</tr>
</tbody>
</table>

### TABLE X1.3 Concentricity, ± in. [mm]

<table>
<thead>
<tr>
<th>Over</th>
<th>Through</th>
</tr>
</thead>
<tbody>
<tr>
<td>...</td>
<td>1 [25]</td>
</tr>
<tr>
<td>...</td>
<td>1 [25]</td>
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<td>1 [25]</td>
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<td>...</td>
<td>1 [25]</td>
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<tr>
<td>...</td>
<td>1 [25]</td>
</tr>
<tr>
<td>...</td>
<td>1 [25]</td>
</tr>
</tbody>
</table>

### FIG. X1.1 Standard Sleeve Bearing

X1.6 Flange and Thrust Bearing Specifications

X1.6.1 Diameter and thickness specifications for flange and thrust washers are shown in Table X1.5.
X1.7 Lubricating Oil-Impregnant

X1.7.1 It was found that the high-grade turbine oil containing rust and oxidation inhibitors and antifoam additives is the most desirable type of oil to be used. The viscosity should be specified by the user in accordance with the application.
TABLE X1.5 Flange and Thrust Bearings Diameter and Thickness Tolerances

<table>
<thead>
<tr>
<th>Diameter Range, in. [mm]</th>
<th>Standard (Tolerance), in. [mm]</th>
<th>Special (Tolerance), in. [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Over Through</td>
<td></td>
<td></td>
</tr>
<tr>
<td>0 [0]</td>
<td>±0.005 [±0.13]</td>
<td>±0.0025 [±0.06]</td>
</tr>
<tr>
<td>1½ [40]</td>
<td>±0.010 [±0.25]</td>
<td>±0.005 [±0.13]</td>
</tr>
<tr>
<td>3 [75]</td>
<td>±0.025 [±0.63]</td>
<td>±0.010 [±0.25]</td>
</tr>
</tbody>
</table>

Flange Bearings, Flange Diameter Tolerances

<table>
<thead>
<tr>
<th>Diameter Range, in. [mm]</th>
<th>Standard (Tolerance), in. [mm]</th>
<th>Special (Tolerance), in. [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Over Through</td>
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<tr>
<td>0 [0]</td>
<td>±0.005 [±0.13]</td>
<td>±0.0025 [±0.06]</td>
</tr>
<tr>
<td>1½ [40]</td>
<td>±0.010 [±0.25]</td>
<td>±0.007 [±0.20]</td>
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<td>3 [75]</td>
<td>±0.015 [±0.40]</td>
<td>±0.010 [±0.25]</td>
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Flange Bearings, Flange Thickness Tolerances

<table>
<thead>
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<tbody>
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<tr>
<td>0 [0]</td>
<td>±0.005 [±0.13]</td>
<td>±0.0025 [±0.06]</td>
</tr>
<tr>
<td>0.475 [12]</td>
<td>0.012 [0.3]</td>
<td></td>
</tr>
<tr>
<td>1.200 [30]</td>
<td>0.024 [0.6]</td>
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</tr>
<tr>
<td>1.20 [30]</td>
<td>±0.031 [0.8]</td>
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Flange Bearings, Radius, r, Tolerance

<table>
<thead>
<tr>
<th>Outside Diameter, D, in. [mm]</th>
<th>Radius, r, max, in. [mm]</th>
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Thrust Bearings (¼-in. [6.35-mm] Thickness, max), Thickness Tolerances for All Diameters

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Parallelism of Faces, max

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<th>Standard (Tolerance), in. [mm]</th>
<th>Special (Tolerance), in. [mm]</th>
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<td>[0.10]</td>
<td>[0.08]</td>
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</table>

A Standard and special tolerances are specified for diameters, thickness, and parallelism. Special tolerances should not be specified unless required since they require additional or secondary operations and, therefore, are costlier.

B Outside diameter tolerances are the same as for flange bearings.

X2.2.1 Requirements

X2.1.1 These bearings are not intended for reaming on assembly.

X2.1.2 Material—Sintered bronze, oil-impregnated bearings made primarily from elemental copper, tin and graphite powders in accordance with ASTM B 438/B 438M, type 2, grade 2 or type 2, grade 4 (note: the grade must be specified in the contract).

X2.2 Notes

X2.2.1 The military part or identifying number (PIN) shall consist of the letters B09, 438X2 where X2 is the B438 appendix number, and a dash number from Table X2.1.

Example: B09-438X2-104

where:

B09 = B09
438X2 = ASTM Standard and Appendix number
104 = Dash number

Note X2.1—The B09-438X2-104 identifying number equates to the old MS 17796-104 designation where the MS 17796 represented the military sheet specification number and 104 was the dash number. The dash numbers remain unchanged from the MS 17796.

X2.2.2 Referenced documents shall be of the issue in effect on the date of invitations for bids or request for proposals, except that referenced adopted industry documents shall give the date of the issue adopted.

X2.2.3 In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence.

X2.2.4 The bearings described in this document are not recommended for military airframe applications.
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<th>Nominal ID</th>
<th>d-basic ID</th>
<th>r-radius (max)</th>
<th>D-basic OD</th>
<th>D₁-flange OD</th>
<th>E-flange thick</th>
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Designation: B 439 – 00\textsuperscript{1}

Standard Specification for Iron-Base Sintered Bearings (Oil-Impregnated)\textsuperscript{1}


1. Scope
1.1 This specification covers sintered metal powder, oil-impregnated, bearings of four iron-base compositional grades:
   Grades 1 and 2—iron-carbon
   Grades 3 and 4—iron-copper

1.2 The values stated in inch-pound units are to be regarded as the standard. The metric equivalents of inch-pound units may be approximate.

2. Referenced Documents
2.1 ASTM Standards:
   B 328 Test Method for Density, Oil Content, and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings\textsuperscript{2}
   E 9 Test Methods of Compression Testing of Metallic Materials at Room Temperature\textsuperscript{3}

3. Ordering Information
3.1 Orders for material under this specification shall include the following information:
   3.1.1 Grade (Section 5),
   3.1.2 Density (6.1),
   3.1.3 Dimensions, and
   3.1.4 Certification (12.1).

4. Manufacture
4.1 Bearings shall be made by briquetting and sintering metal powders, with or without sizing, so as to produce finished parts conforming to the requirements of this specification.

5. Chemical Requirements
5.1 The material shall conform to the requirements as to the chemical composition prescribed in Table 1.

6. Physical Properties
6.1 Density—The density of bearings supplied fully impregnated with lubricant shall be within the limits prescribed in Table 2.
6.2 Oil Content—Oil content of bearings shall not be less than that shown in Table 2.
6.3 Radial Crushing Strength—Radial crushing strength shall not be less than the value calculated as follows:

\[
P = \frac{KL^2(T - D)}{2}
\]

\textsuperscript{1} This specification is under the jurisdiction of ASTM Committee B-09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.04 on Bearings.

\textsuperscript{2} Annual Book of ASTM Standards, Vol 02.05.

\textsuperscript{3} Annual Book of ASTM Standards, Vol 03.01.

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where:

\[ P = \text{radial crushing strength, lbf (or N);} \]
\[ D = \text{outside diameter of bearing, in. (or mm);} \]
\[ T = \text{wall thickness of bearing, in. (or mm);} \]
\[ K = \text{strength constant as shown in Table 3 for grade specified, psi (MPa); and} \]
\[ L = \text{length of bearing, in. (or mm).} \]

7. Workmanship, Finish, and Appearance

7.1 Bearings shall be uniform in composition. When cut or fractured, the exposed surface shall be of uniform appearance. The parts shall be free from defects which would affect their serviceability.

8. Sampling

8.1 Lot—Unless otherwise specified, a lot shall consist of parts of the same form and dimensions made from powders of the same composition, formed and sintered under the same conditions, and submitted for inspection at one time.

8.2 Sample for Chemical Analysis —At least one sample for chemical analysis shall be taken from each lot. A representative sample of chips may be obtained by milling, drilling, or crushing at least two pieces with clean dry tools without lubrication. To obtain oil-free chips, the parts selected for test shall have the oil extracted in accordance with Test Method B 328, if necessary.

8.3 Mechanical Tests— The manufacturer and purchaser shall agree on a representative number of specimens for mechanical tests.

9. Test Methods

9.1 Density and Oil Content—Density and oil content shall be determined in accordance with Test Method B 328.

9.2 Radial Crushing Strength—Radial crushing strength shall be determined by compressing the test specimens between two flat surfaces at a “no-load” speed no greater than 0.2 in./min (5.0 mm/min), the direction of the load being normal to the longitudinal axis of the specimen. The point at which the load drops as a result of the first crack shall be considered the crushing strength. This test shall be applied to plain cylindrical bearings. Flanged bearings shall be tested by cutting off the flange and compressing the two sections separately. Each section shall meet the maximum minimum strength requirements prescribed in 6.3.

10. Inspection

10.1 Unless otherwise specified, inspection of parts supplied on contract shall be made by the purchaser at the destination.

11. Rejection

11.1 Rejection based on tests made in accordance with this specification shall be reported to the manufacturer within 30 days of receipt of shipment; however, the rejected parts should not be returned without authority from the producer.

### TABLE 1 Chemical Requirements

<table>
<thead>
<tr>
<th>Element</th>
<th>Grade 1</th>
<th>Grade 2</th>
<th>Grade 3</th>
<th>Grade 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper</td>
<td>0–1.5</td>
<td>0–1.5</td>
<td>9.0–11.0</td>
<td>18.0–22.0</td>
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<tr>
<td>Iron</td>
<td>balance</td>
<td>balance</td>
<td>balance</td>
<td>balance</td>
</tr>
<tr>
<td>Combined carbon (on basis of iron only)</td>
<td>0.3 max</td>
<td>0.3–0.6</td>
<td>0.3 max</td>
<td>0.3 max</td>
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</table>

*The combined carbon may be a metallographic estimate of the carbon in the iron.*

### TABLE 2 Density and Oil Content Requirements

<table>
<thead>
<tr>
<th>Grade</th>
<th>Density, g/cm³</th>
<th>Oil Content, Volume, %</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>min</td>
<td>max</td>
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<tr>
<td>1 and 2</td>
<td>5.6</td>
<td>6.0</td>
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<tr>
<td>3 and 4</td>
<td>5.8</td>
<td>6.2</td>
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</table>

### TABLE 3 Strength Constants

<table>
<thead>
<tr>
<th>Grade</th>
<th>K (Strength Constant), psi (MPa)</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>15 000 (105)</td>
</tr>
<tr>
<td>2</td>
<td>20 000 (140)</td>
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<tr>
<td>3</td>
<td>30 000 (205)</td>
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<tr>
<td>4</td>
<td>30 000 (205)</td>
</tr>
</tbody>
</table>
12. Certification

12.1 A certification based on the manufacturer’s quality control that the material conforms to the requirements of this specification, shall be the basis of shipment of the material. A certificate covering the conformance of the material to these specifications shall be furnished by the manufacturer upon request of the purchaser.

13. Keywords

13.1 density; iron-base bearings; $K$ strength constant; oil content; oil impregnated; porosity

APPENDIX

(Nonmandatory Information)

X1. EXPLANATORY INFORMATION

X1.1 Design Information

X1.1.1 In calculating permissible loads, the operating conditions, housing conditions, and construction should be considered. In general, this material has less resistance to seizure and corrosion than copper-base material. The maximum static bearing load should not exceed 15 000 psi (105 MPa) of projected bearing area (length times inside diameter of bearing) for this material. This figure is 75% of the value for the compression deformation limit (yield strength permanent set of 0.001 in. (0.025 mm) for specimens 1⅛in. (28.6 mm) in diameter and 1 in. (25.4 mm) in length) as determined in accordance with Test Methods E 9.

X1.2 Permissible Loads

X1.2.1 Permissible loads for various operating conditions are given in Table X1.1.

X1.3 Dimensional Tolerances

X1.3.1 Commercial dimensional tolerances are given in Table X1.2.

X1.4 Press Fits

X1.4.1 Plain cylindrical journal bearings are commonly installed by press fitting the bearing into a housing with an insertion arbor. For housings rigid enough to withstand the press fit without appreciable distortion and for bearings with wall thickness approximately one eighth of the bearing outside diameter, the press fits shown in Table X1.3 are recommended.

X1.5 Running Clearance

X1.5.1 Proper running clearance for sintered bearings depends to a great extent on the particular application. Therefore, only minimum recommended clearances are listed in Table X1.4. It is assumed that ground steel shafting will be used and that all bearings will be oil impregnated.

X1.6 Flange and Thrust Bearing Specifications

X1.6.1 Diameter and thickness specifications for flange and thrust washers are shown in Table X1.5

<table>
<thead>
<tr>
<th>TABLE X1.1 Permissible Loads</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shaft Velocity, ft/min (m/min)</td>
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<td></td>
</tr>
<tr>
<td>Slow and intermittent</td>
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<td>25 (7.6)</td>
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<tr>
<td>50 to 100 (15.2 to 30.4), incl</td>
</tr>
<tr>
<td>Over 100 to 150 (30.4 to 45.7), incl</td>
</tr>
<tr>
<td>Over 150 to 200 (45.7 to 61), incl</td>
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<td>Over 200 (61)</td>
</tr>
</tbody>
</table>

*For shaft velocities over 200 ft/min the permissible loads may be calculated as follows:

\[ P = \frac{50 000}{V} \]

where:

- $P$ = safe load, psi of projected area
- $V$ = shaft velocity, ft/min.
X1.7 Lubrication

X1.7.1 It was found that a circulating type oil containing rust and oxidation inhibitors is the most desirable type of oil to be used. The viscosity should be specified by the user in accordance with the application.
### TABLE X1.5 Flange and Thrust Bearings Diameter and Thickness Tolerances

<table>
<thead>
<tr>
<th>Diameter Range</th>
<th>Flange Diameter Tolerances</th>
<th>Standard</th>
<th>Special</th>
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</thead>
<tbody>
<tr>
<td>in.</td>
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<td>mm</td>
<td>mm</td>
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<td>0 to 1 1/2</td>
<td>0 to 38</td>
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<td>±0.13</td>
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<tr>
<td>Over 1 1/2 to 3</td>
<td>39 to 76</td>
<td>±0.010</td>
<td>±0.25</td>
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<tr>
<td>Over 3 to 6</td>
<td>77 to 152</td>
<td>±0.025</td>
<td>±0.64</td>
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<table>
<thead>
<tr>
<th>Diameter Range</th>
<th>Flange Thickness Tolerances</th>
<th>Standard</th>
<th>Special</th>
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<td>mm</td>
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<tr>
<td>0 to 1 1/2</td>
<td>0 to 38</td>
<td>±0.005</td>
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<td>Over 1 1/2 to 3</td>
<td>39 to 76</td>
<td>±0.010</td>
<td>±0.25</td>
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<tr>
<td>Over 3 to 6</td>
<td>77 to 152</td>
<td>±0.015</td>
<td>±0.38</td>
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</table>

<table>
<thead>
<tr>
<th>Diameter Range</th>
<th>Thrust Bearings (1/4 in, 6.35 mm Thickness, max), Thickness Tolerances</th>
<th>All Diameters</th>
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<tr>
<td>in.</td>
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<td>mm</td>
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<tr>
<td>0 to 1 1/2</td>
<td>±0.005</td>
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<td>Over 1 1/2 to 3</td>
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<table>
<thead>
<tr>
<th>Diameter Range</th>
<th>Parallelism on Faces, max</th>
<th>Standard</th>
<th>Special</th>
</tr>
</thead>
<tbody>
<tr>
<td>in.</td>
<td>mm</td>
<td>mm</td>
<td>mm</td>
</tr>
<tr>
<td>0 to 1 1/2</td>
<td>0 to 38</td>
<td>±0.005</td>
<td>0.13</td>
</tr>
<tr>
<td>Over 1 1/2 to 3</td>
<td>39 to 76</td>
<td>±0.007</td>
<td>0.18</td>
</tr>
<tr>
<td>Over 3 to 6</td>
<td>77 to 152</td>
<td>±0.010</td>
<td>0.25</td>
</tr>
</tbody>
</table>

*Standard and special tolerances are specified for diameters, thickness, and parallelism. Special tolerances should not be specified unless required since they require additional or secondary operations and, therefore, are costlier.*

*Outside diameter tolerances same as for flange bearings.*

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Standard Specification for
Chromates on Aluminum¹

This standard is issued under the fixed designation B 449; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This specification covers the requirements relating to rinsed and nonrinsed chromate conversion coatings on aluminum and aluminum alloys intended to give protection against corrosion and as a base for other coatings. This edition of the specification has been coordinated with ISO 10546 and is technically equivalent.

1.2 Aluminum and aluminum alloys are chromate coated in order to retard corrosion; as a base for organic films including paints, plastics, and adhesives; and as a protective coating having a low electrical contact impedance.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards: ²
B 117 Practice for Operating Salt Spray (Fog) Apparatus
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 767 Guide for Determining Mass Per Unit Area of Electrodeposited and Related Coatings by Gravimetric and Other Chemical Analysis Procedures
D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting
D 3359 Test Methods for Measuring Adhesion by Tape Test

2.2 ISO Standards:
ISO 2409 Paint and Varnishes—Cross-Cut Test
ISO 3768 Metallic Coatings—Neutral Salt Spray Test
ISO 3892 Conversion Coatings on Metallic Materials—Determination of Mass Per Unit Area—Gravimetric Method
ISO 4519 Electrodeposited Metallic Coatings and Related Finishes—Sampling Procedures for Inspection by Attributes
ISO/ DIS 10546 Chemical Conversion Coatings—Rinsed and Nonrinsed Chromate Conversion Coatings—On Aluminum and Aluminum Alloys

2.3 Federal Standard:
Fed. Std. No. 141 Paints, Varnish, Lacquer, and Related Materials; Methods of Inspection

2.4 Military Specification:
MIL-C-5541 Chemical Films for Aluminum and Aluminum Alloys

3. Terminology

3.1 Definitions:
3.1.1 nonrinsed—chromate coatings that are dried immediately after the chromating step without receiving a water rinse.
3.1.1.1 Discussion—This special type of coating is typically used on long coils of aluminum sheet stock that receive an immediate subsequent paint or adhesive coating.

NOTE 1—Nonrinsed chromate coatings are finding increased usage on fabricated parts and castings.

3.1.2 rinsed—chromate coatings that are rinsed in water prior to drying.
3.1.2.1 Discussion—This type of coating is typically applied to extruded aluminum fabricated parts and castings.

4. Classification

4.1 Chromate finishes can be applied ranging in color from brown, thick coatings (Class 1) providing maximum corrosion protection to yellow, intermediate thickness coatings (Class 2) suitable as an organic film base or to colorless, thin coatings (Class 3) suitable for low electrical contact resistance. The yellow coatings vary from golden yellow to iridescent light yellow. Chromate-phosphate finishes (Class 4) can be applied

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¹ This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.07 on Chemical Conversion Coatings.


² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard’s Document Summary page on the ASTM website.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

⁴ Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098

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ranging in color from green to iridescent light green. The Class 4 coatings comply with the requirements of MIL-C-5541.

4.2 Finishers can seldom guarantee to supply exact shades of color with chromate conversion coatings. If it is necessary to have exact shades of color, it is possible to dye chromate coatings having a coating mass greater than 0.4 g/m² to obtain a wide range of colors, but they can only be expected to give an order of added corrosion resistance similar to that provided by the undyed coatings. It should be noted that color and color uniformity will vary somewhat between one alloy and another and from a polished surface to an etched surface. Iridescence and variations in color density from one area of the surface to another are normal and shall not be considered a sign of poor quality.

4.3 The finishes are divided into four classes; their most important characteristics are listed in Table 1.

5. Surfaces Preparation

5.1 The surfaces of the parts to be chromated must be clean and free of any oxidation, scale, or soils such as metal turnings, grinding dust, oil, grease, lubricants, hand-sweat, or any other contamination detrimental to the chromating process. The parts must therefore, as far as necessary, be cleaned before chromating and if necessary be pickled. Fig. X2.1 shows the various processing step options.

6. Methods of Application of Chromate Coatings

6.1 Metallic material other than aluminum should not be treated with the parts to be chromated.

6.2 Chromate conversion coatings are normally applied by dipping: the coating may also be applied by inundation, spraying, roller coating, or by wipe-on techniques. The application method used should be taken from the operating instructions for the chromating process employed. Chromating solutions are usually acidic and may contain hexavalent chromium salts together with other salts that may be varied to affect the appearance and hardness of the film. The color of the film, and, therefore, the type of conversion coating, depends on the composition of the chromating solution, but it is also affected by the pH and temperature, the duration of the treatment, and the nature and surface condition of the alloy being treated.

6.3 These coatings receive a final water rinse. If the coating is meant to be a basis for additional coatings, the detail shall be subject to a rinse in deionized water with a conductivity less than 100 µS/cm. If hot water is used as the final rinse after the chromating process, it is essential that the time of rinsing should be kept as short as possible in order to prevent the dissolution of the hexavalent chromium. The drying of the coating shall be carried out at a temperature not exceeding 60°C to prevent cracking due to dehydration, which causes loss of adhesion and performance of the chromate coating.

6.4 Any additional subsequent treatments depend upon the purpose for which the chromated parts are intended.

7. Coating Requirements

7.1 General—Chromate conversion coatings harden with age by gradual dehydration. They should, therefore, be handled carefully for the first 24 h after treatment, and any tests (including corrosion tests) shall be deferred until the expiration of that period. The green chromate-phosphate coatings usually continue to improve in corrosion resistance after initial formation. They achieve their maximum corrosion resistance after 1 to 2 months at room temperature. It is not required to store parts for this purpose.

7.2 Electrical Resistance—Colorless, light yellow, or light green iridescent chromate layers of low mass per unit area increase the electrical resistance between an electrical contact and the aluminum to a very small extent. When measured at 9-V and a 2-A current the resistance should be less than 0.1 Ω. Highly colored brown, yellow, or green coatings show a marked increase in electrical contact resistance with increasing mass per unit area of the chromate layer and may reach resistances of 10,000 Ω or more.

7.3 Adhesion—The coatings shall be adherent and non-powdery. There are no practical tests for measuring the adhesion of a chromate conversion coating on aluminum. However, a practical evaluation of the adhesion can be made by measuring the adhesion of a secondary organic film applied to the chromated aluminum. When specified, the chromate conversion coating shall pass the organic coating adhesion test in Test Methods D 3359 or the equivalent ISO 2409.

7.3.1 Class 4 coatings intended for use under MIL-C-5541 shall have their adhesion evaluated by Method 6301 of Fed. Std. No. 141.

7.3.2 Additional treatments applied under MIL-C-5541 can be found in Practices D 1730, Methods 5, 6, and 7.

7.4 Corrosion Resistance—When subjected to the neutral salt spray test specified in Practice B 117 or in the equivalent ISO 3768, three separate test specimens of the coating shall withstand exposure for the hours shown in Table 2 without giving evidence, to the unaided eye, of more than a total of 8 isolated spots or pits. None shall be larger than 1 mm in diameter. Each individual test specimen shall not have more than 5 isolated spots or pits, none larger than 1 mm in diameter on their respective surfaces. Spots within 10 mm of the edges of the panels are not counted.

7.5 Coating Mass Per Unit Area—The mass of the coating per unit area of coated surface shall conform to the values given in Table 1. Use the procedure in ISO 3892 as referenced in Guide B 767.

7.5.1 Coating Mass Significance—It should be recognized that heavier coating masses do not always provide better
performance, particularly when using the coating for an organic film or adhesive base.

7.6 Confirming Class 2 Coatings—The presence of a Class 2 coating shall be confirmed by the spot test in Appendix X1.

7.7 Coating Identification:
7.7.1 Colorless, Yellow, or Brown Coatings—The determination of chromium in the coating will identify the layer as a chromate conversion coating. Use the test in Appendix X1.
7.7.2 Green Coatings—It is necessary to determine the presence of phosphate and the absence of zinc in order to distinguish chromate-phosphate coatings from anodized coatings or zinc phosphate coatings. Use the test in Appendix X1.
7.8 Test Specimens and Samplings—Unless otherwise specified the sampling plans of Test Method B 602 or the equivalent ISO 4519 shall be used to test the coatings.

8. Keywords
8.1 chromate; clear chromate; conversion coating; non-rinsed; passivation coating; rinsed; yellow chromate

APPENDIXES
(Nonmandatory Information)

X1. QUALITATIVE TESTING OF THE LAYER COMPOSITION

X1.1 Reagents
X1.1.1 Only analytical grade reagents should be used. The water used shall be distilled or deionized water. Solutions without indication of the solvent are solutions in distilled or completely demineralized water.

X1.1.1.1 Sodium Hydroxide, approximately 5 % solution of NaOH.
X1.1.1.2 Sodium Hydroxide, approximately 20 % solution of NaOH.
X1.1.1.3 Hydrogen Peroxide, approximately 30 % solution of H₂O₂.
X1.1.1.4 Acetic Acid, approximately 10 % solution of CH₃·COOH.
X1.1.1.5 Lead Nitrate Solution, approximately 10 % solution of Pb(NO₃)₂.
X1.1.1.6 Nitric Acid, approximately 65 % solution of HNO₃ (density, 1.40 g/cm³ at 20°C).
X1.1.1.7 Nitric Acid, approximately 38 % solution of HNO₃, made up for instance by mixing 1 part by volume of nitric acid of density 1.40 g/cm² at 20°C with 1 part by volume of water.
X1.1.1.8 Ammonium Molybdate-Reagent—88.5 g ammonium molybdate (NH₄)₆Mo₇O₂₄·4 H₂O, 34-mL aqueous ammonium hydroxide solution with 25 % NH₃, and 240 g ammonium nitrate NH₄NO₃ are dissolved in water up to 1 L total.
X1.1.1.9 Ammonium Nitrate, NH₄NO₃.
X1.1.1.10 Hydrochloric Acid, approximately 25 % solution of HCl.

X1.1.1.11 Potassium Ferrocyanide Solution, approximately 5 % solution of K₄Fe(CN)₆.
X1.1.1.12 Sulfuric Acid, approximately 25 % solution of H₂SO₄.

X1.2 Determination of Chromium in the Layer
X1.2.1 A sample of about 300-cm² surface is treated with 50 mL of 5 % sodium hydroxide solution, to which 5 mL of 30 % hydrogen peroxide is added; the solution, at 50 to 60°C, is poured over, repeatedly if necessary, until the layer is completely removed. The resulting solution is poured off, boiled until all hydrogen peroxide is destroyed (about 5 to 6 min), cooled down and precipitated with an approximately 10 % lead nitrate solution. A yellow precipitate shows the presence of hexavalent chromium. The least quantity of total chromium detectable by this method corresponds to about 5 mg chromium per square metre of surface.

X1.3 Testing for Phosphate
X1.3.1 To test for the presence of phosphate in the chromated layer, a sample is taken whose surface to be tested is about 100 cm²; this is treated with 100 mL of 5 % sodium hydroxide solution at 80 to 90°C until the layer is completely dissolved or at least until the surface has been clearly attacked. The resulting solution is filtered, and 25 mL of the filtrate are acidified with approximately 38 % nitric acid then 10 mL ammonium molybdate reagent and 5 g ammonium nitrate are added. The sample is allowed to stand for at least 15 min. A yellow precipitate shows the presence of phosphate. The least
quantity of phosphate detectable by this process corresponds to about 40 mg P₂O₅ per square metre of surface.

X1.4 Testing for Zinc

X1.4.1 To test for the presence of zinc in the layer, a sample is taken whose surface to be tested is about 100 cm²; this is treated with 50 mL of approximately 65 % nitric acid at room temperature until the layer is either completely dissolved or at least until the surface has clearly been attacked. The resulting solution is filtered through glass wool, and 25 mL of the filtrate are neutralized with 20 % caustic soda until phenolphthalein indicator gives a red coloration. The solution is then weakly acidified by addition of about 10 drops of 25 % sulfuric acid. After addition of 5 mL of 5 % potassium ferrocyanide solution, a greenish-white precipitate shows the presence of zinc. The least quantity of zinc detectable by this process corresponds to about 20 mg zinc per square metre of surface.

X2. TREATMENT SEQUENCE OPTIONS

X2.1 The chromate process necessitates a specified sequence of treatments that depends upon the surface condition of the parts to be treated, the aluminum alloy used, and the desired appearance of the parts. See Fig. X2.1.
1. Scope

1.1 This specification covers requirements for several types and grades of electrodeposited copper plus nickel plus chromium or nickel plus chromium coatings on steel, nickel plus chromium coatings on copper and copper alloys, nickel plus chromium coatings on Type 300 and 400 series stainless steel and copper plus nickel plus chromium coatings on aluminum and its alloys and zinc alloys for applications where both appearance and protection of the basis metal against corrosion are important. Five grades of coatings are provided to correspond with the service conditions under which each is expected to provide satisfactory performance: namely, extended very severe, very severe, severe, moderate, and mild. Definitions and typical examples of these service conditions are provided in Appendix X1.

1.2 This specification does not cover the requirements for the plating on plastics, see Specification B 604.

1.3 The following hazards caveat pertains only to the test methods portions, Appendix X2, Appendix X3, Appendix X4, and Appendix X5 of this specification: This standard does not purport to address all of safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Guide for Preparation of High-Carbon Steel for Electroplating
B 252 Guide for Preparation of Zinc Alloy Die Castings for Electroplating and Conversion Coatings
B 253 Guide for Preparation of Aluminum Alloys for Electroplating
B 254 Practice for Preparation of and Electroplating on Stainless Steel
B 281 Practice for Preparation of Copper and Copper Base Alloys for Electroplating and Conversion Coatings
B 320 Practice for Preparation of Iron Castings for Electroplating
B 368 Method for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test)
B 380 Test Method of Corrosion Testing of Decorative Electrodeposited Coatings by the Corrodkote Procedure
B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section
B 489 Practice for Bend Test for Ductility of Electrodeposited and Autocatalytically Deposited Metal Coatings of Metal
B 490 Practice for Micrometer Bend Test for Ductility of Electrodeposits
B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
B 530 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Electrodeposited Nickel Coatings on Magnetic and Nonmagnetic Substrates
B 537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure
B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 604 Specification for Decorative Electroplated Coatings of Copper Plus Nickel Plus Chromium on Plastics
B 659 Guide for Measuring Thickness of Metallic and Inorganic Coatings
3. Terminology

3.1 Definitions:
3.1.1 significant surfaces—those surfaces normally visible (directly or by reflection) that are essential to the appearance or serviceability of the article, or both, when assembled in normal position; or that can be the source of corrosion products that deface visible surfaces on the assembled article. When necessary, the significant surfaces shall be specified by the purchaser and shall be indicated on the drawings of the parts, or by the provision of suitably marked samples.

4. Classification

4.1 Five grades of coatings designated by service condition numbers and several types of coatings defined by classification numbers are covered by this specification.

4.2 Service Condition Number:
4.2.1 The service condition number indicates the severity of exposure for which the grade of coating is intended:
SC 5 extended severe service
SC 4 very severe service,
SC 3 severe service,
SC 2 moderate service, and
SC 1 mild service.

4.2.2 Typical service conditions for which the various service condition numbers are appropriate are given in Appendix X1.

4.3 Coating Classification Number—The coating classification number comprises:
4.3.1 The chemical symbol for the basis metal (or for the principal metal if an alloy) followed by a slash mark, except in the case of stainless steel. In this case, the designation shall be SS followed by the designated AISI number followed by a slash, that is, SS463/.

4.3.2 The chemical symbol for copper (Cu) (if copper is used),
4.3.3 A number indicating the minimum thickness of the copper coating in micrometres (if copper is used),
4.3.4 A lower-case letter designating the type of copper deposit (if copper is used) (see 4.4 and 6.2.3),
4.3.5 The chemical symbol for nickel (Ni),
4.3.6 A number indicating the minimum thickness of the nickel coating, in micrometres,
4.3.7 A lower-case letter designating the type of nickel deposit (see 4.4 and 6.2.4),
4.3.8 The chemical symbol for chromium (Cr), and
4.3.9 A letter (or letters) designating the type of chromium deposit and its minimum thickness in micrometres (see 4.4 and 6.2.5).

4.4 Symbols for Expressing Classification—The following lower-case letters shall be used in coating classification numbers to describe the types of coatings:
a —ductile copper deposited from acid-type baths
b —single-layer nickel deposited in the fully-bright condition
d —double- or triple-layer nickel coatings
r —regular (that is, conventional) chromium
mc —microporous chromium
mp —microporosity chromium

4.5 Example of Complete Classification Numbers—A coating on steel comprising 15 µm minimum (ductile acid) copper plus 25 µm minimum (duplex) nickel plus 0.25µm minimum (micro-cracked) chromium has the classification number: Fe/Cu15aNi25d Cr mc (see 4.3 and 6.2 for explanation of symbols).

5. Ordering Information

5.1 When ordering articles to be electroplated in conformance with this standard, the purchaser shall state the following:
5.1.1 The ASTM designation number of this standard.
5.1.2 Either the classification number of the specific coating required (see 4.3) or the substrate material and the service condition number denoting the severity of the conditions it is required to withstand (see 4.2). If the service condition number is quoted and not the classification number, the manufacturer is free to supply any of the types of coatings designated by the classification numbers corresponding to the specified service condition number, as given in Table 1, Table 2, Table 3, Table 4, or Table 5. On request, the manufacturer shall inform the purchaser of the classification number of the coating applied.

### Table 1: Nickel Plus Chromium Coatings on Steel

<table>
<thead>
<tr>
<th>Service Condition No.</th>
<th>Classification No.</th>
<th>Nickel Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>SC 5</td>
<td>Fe/Ni35d Cr mc</td>
<td>35</td>
</tr>
<tr>
<td>SC 4</td>
<td>Fe/Ni30d Cr mc</td>
<td>30</td>
</tr>
<tr>
<td>SC 3</td>
<td>Fe/Ni25d Cr mc</td>
<td>25</td>
</tr>
<tr>
<td>SC 2</td>
<td>Fe/Ni20b Cr r</td>
<td>20</td>
</tr>
<tr>
<td>SC 1</td>
<td>Fe/Ni15b Cr mc</td>
<td>15</td>
</tr>
</tbody>
</table>

Note: 1—When permitted by the purchaser, copper may be used as an undercoat for nickel but is not substitutable for any part of the nickel thickness specified. If the use of copper is permitted, Table 2 may be used to obtain the same service conditions.

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1 Annual Book of ASTM Standards, Vol 11.01.
3 Annual Book of ASTM Standards, Vol 03.05.
4 Available from International Standards Organization, 1 Rue de Varembe, Geneva 20, Switzerland.
TABLE 2 Copper Plus Nickel Plus Chromium Coatings on Steel

<table>
<thead>
<tr>
<th>Service Condition No.</th>
<th>Classification No.</th>
<th>Nickel Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>SC 5</td>
<td>Fe/Cu15a Ni30d Cr mc</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td>Fe/Cu15a Ni30d Cr mp</td>
<td>30</td>
</tr>
<tr>
<td>SC 4</td>
<td>Fe/Cu15a Ni25d Cr mc</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td>Fe/Cu15a Ni25d Cr mp</td>
<td>25</td>
</tr>
<tr>
<td>SC 3</td>
<td>Fe/Cu12a Ni20d Cr mc</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>Fe/Cu12a Ni20d Cr mp</td>
<td>20</td>
</tr>
</tbody>
</table>

TABLE 3 Copper Plus Nickel Plus Chromium Coatings on Zinc Alloy

<table>
<thead>
<tr>
<th>Service Condition No.</th>
<th>Classification No.</th>
<th>Nickel Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>SC 5</td>
<td>Zn/Cu5 Ni35d Cr mc</td>
<td>35</td>
</tr>
<tr>
<td></td>
<td>Zn/Cu5 Ni35d Cr mp</td>
<td>35</td>
</tr>
<tr>
<td>SC 4</td>
<td>Zn/Cu5 Ni30d Cr mc</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td>Zn/Cu5 Ni30d Cr mp</td>
<td>30</td>
</tr>
<tr>
<td>SC 3</td>
<td>Zn/Cu5 Ni25d Cr mc</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>Zn/Cu5 Ni25d Cr mp</td>
<td>20</td>
</tr>
<tr>
<td>SC 2</td>
<td>Zn/Cu5 Ni20b Cr r</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>Zn/Cu5 Ni15b Cr mc</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td>Zn/Cu5 Ni15b Cr mp</td>
<td>15</td>
</tr>
<tr>
<td>SC 1</td>
<td>Zn/Cu5 Ni10b Cr r</td>
<td>10</td>
</tr>
</tbody>
</table>

TABLE 4 Nickel Plus Chromium Coatings on Copper or Copper Alloy

<table>
<thead>
<tr>
<th>Service Condition No.</th>
<th>Classification No.</th>
<th>Nickel Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>SC 4</td>
<td>Cu/Ni25d Cr mc</td>
<td>25</td>
</tr>
<tr>
<td></td>
<td>Cu/Ni25d Cr mp</td>
<td>25</td>
</tr>
<tr>
<td>SC 3</td>
<td>Cu/Ni20d Cr mc</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>Cu/Ni20d Cr mp</td>
<td>20</td>
</tr>
<tr>
<td>SC 2</td>
<td>Cu/Ni15b Cr r</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td>Cu/Ni10b Cr mc</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>Cu/Ni10b Cr mp</td>
<td>10</td>
</tr>
<tr>
<td>SC 1</td>
<td>Cu/Ni5b Cr r</td>
<td>5</td>
</tr>
</tbody>
</table>

5.1.3 The appearance required, for example, bright, dull, or satin. Alternatively, samples showing the required finish or range of finish shall be supplied or approved by the purchaser.

5.1.4 The significant surfaces, to be indicated on drawings of the parts, or by the provision of suitably marked specimens (see 3.1).

5.1.5 The positions on significant surfaces for rack or contact marks, where such marks are unavoidable (see 6.1.1).

5.1.6 The extent to which defects shall be tolerated on nonsignificant surfaces.

5.1.7 The elongation of copper if other than the standard value (see 6.4).

5.1.8 The ductility of the nickel if other than the standard value (see 6.5).

5.1.9 The extent of tolerable surface deterioration after corrosion testing (see 6.7.3).

5.1.10 Sampling methods and acceptance levels (see Section 7).

5.1.11 The minimum values of the electrode potential differences between individual nickel layers as measured in accordance with Test Method B 764 within the limits given in 6.8.

5.1.12 Adhesion Test—The adhesion test to be used (see 6.3).

6. Product Requirements

6.1 Visual Defects:

6.1.1 The significant surfaces of the electroplated article shall be free of clearly visible plating defects, such as blisters, pits, roughness, cracks, and uncoated areas and shall not be stained or discolored. On articles where a visible contact mark is unavoidable, its position shall be agreed upon by the purchaser and the plater. The electroplated article shall be clean and free of damage.

6.1.2 Defects in the surface of the basis metal, such as scratches, porosity, nonconducting inclusions, roll and die marks, cold shuts, and cracks, may adversely affect the appearance and the performance of coatings applied thereto despite the observance of the best electroplating practices. Accordingly, the plater’s responsibility for defects in the coating resulting from such conditions shall be waived.

NOTE 1—To minimize problems of this type, the specifications covering the basis material or the item to be electroplated should contain appropriate limitations on such basis metal conditions.

6.2 Process and Coating Requirements:

6.2.1 Proper preparatory procedures and thorough cleaning of the basis metal surface are essential for satisfactory adhesion and corrosion performance of the coating. Accordingly, the applicable practices for the preparation of various basis metals for electroplating shall be followed. Practices B 183, B 242, B 252, B 281, and B 320 are examples of practices that may be used for the preparation of basis metals.

6.2.2 Following the preparatory operations, the parts (articles) to be electroplated are introduced in such plating baths as required to produce the types of deposits described by the specific coating classification numbers or one of the coating classification numbers listed in Table 1, Table 2, Table 3, Table 4, or Table 5 appropriate for the specified service condition number.

6.2.3 Type of Copper and Deposit Thickness:
6.2.3.1 Type of Copper—The type of copper is designated by the following symbols that are placed after the thickness value:

- a for ductile copper deposited from acid-type baths containing additives that promote leveling by the copper deposit and have an elongation not less than 8%.
- No symbol is placed after the thickness value if a minimum elongation is not required or if a deposit from a non-leveling bath is permitted.

6.2.3.2 Thickness of Copper Deposits—The number following the chemical symbol for copper (Cu) indicates in micrometres the minimum thickness of the copper deposit at points on significant surfaces (see 3.1).

6.2.4 Type of Nickel and Deposit Thickness

6.2.4.1 Type of Nickel—The type of nickel is designated by the following symbols, which are placed after the thickness value (Note 5):

- b for nickel deposited in the fully bright condition.
- d for a double-layer or triple-layer nickel coating.

The bottom layer of this coating system shall contain less than 0.005 mass % sulfur (Note 3), and a minimum ductility of 67% (see Practice B 490). The top layer of this system shall contain more than 0.04 mass % sulfur (Note 2 and Note 3), and have a minimum ductility of 11%. Its thickness shall be not less than 20% nor more than 40% (see Table 6) of the total nickel thickness. The thickness of the bottom layer in double-layer coatings shall not be less than 60% nor more than 80% of the total nickel thickness. In triple-layer coatings, the bottom layer shall be not less than 50% nor more than 70%. If there are three layers, the intermediate layer shall contain not less than 0.15 mass % sulfur and shall not exceed 10% of the total nickel thickness. These requirements for multilayer nickel coatings are summarized in Table 6.

Note 2—The sulfur contents are specified in order to indicate which type of nickel electroplating solution must be used. Although at present, no simple method is available for determining the sulfur content of a nickel deposit on a coated article, chemical determinations are possible using specially prepared test specimens (see Appendix X3).

Note 3—It will usually be possible to identify the type of nickel by microscopical examination of the polished and etched section of an article prepared in accordance with Test Method B 487. The thickness of the individual nickel layers in double-layer and triple-layer coatings, as well as the electrochemical relationships between the individual layers, can also be measured by the STEP test, in accordance with Test Method B 764.

6.2.4.2 Thickness of Nickel Deposit—The number following the chemical symbol Ni indicates, in micrometres, the minimum thickness of the nickel electrodeposited at points on the significant surface (see 3.1).

6.2.5 Type of Chromium and Deposit Thickness

6.2.5.1 Type of Chromium—The type of chromium deposit is designated by the following symbols placed after the chemical symbol Cr:

- r for “regular” (that is, conventional) chromium.
- mp for microporous chromium containing a minimum of 10 000 pores/10 mm by 10 mm square (10 000/cm²). The pores shall be invisible to the unaided eye (see 6.10).
- mc for microcracked chromium, having more than 30 cracks/mm in any direction over the whole of the significant surface. The cracks shall be invisible to the unaided eye (see 6.10).

Note 4—A specially formulated nickel strike in between the bright nickel and the chromium deposits may be used to induce micropores or microcracks in the chromium deposits. Controlled particle impingement of the plated standard chromium deposit may also be used to induce microporous chromium. Trivalent chromium deposits, as plated, may be microporous, microcracked, or both.

6.2.5.2 Thickness of Chromium Deposit—The minimum thickness of the chromium deposit shall be 0.25 μm on significant surfaces (see 3.1), except that for service condition SC 1 (see 4.2.1) the minimum thickness may be reduced to 0.13 μm. The thickness of chromium is designated by the same symbol as the type instead of by numerals as in the case of copper and nickel.

6.2.5.3 When plating chromium over a nickel strike containing micro-particles used to induce microporosity in the subsequent chromium deposit, excess chromium thickness will bridge the nonconductive particles within the nickel layer. A maximum of 0.5 μm is recommended.

6.3 Adhesion—The coating shall be sufficiently adherent to the basis metal, and the separate layers of multilayer coatings shall be sufficiently adherent to each other, to pass the appropriate tests detailed in Test Methods B 571. The particular test or tests to be used shall be specified by the purchaser.

6.4 Elongation—The elongation of copper shall be such that it will not be less than stated in 6.2.3.1 when tested by the method given in Appendix X2. Greater elongation may be requested but shall be subject to agreement between the purchaser and the manufacturer.

6.5 Ductility—The ductility of the composite nickel deposit on a finished part is considered acceptable when foils plated out of the individual nickel processes meet or exceed the values listed in Table 6. See test details in Test Method B 490.

6.6 Coating Thickness:

6.6.1 The minimum coating thickness shall be as designated by the coating classification number.

6.6.2 It is recognized that requirements may exist for thicker coatings than are covered by this specification.

6.6.3 The thickness of a coating and its various layers shall be measured at points on the significant surfaces (See 3.1.1 and Note 5).

Note 5—When significant surfaces are involved on which the specified thickness of deposit cannot readily be controlled, such as threads, holes,
shall be cause for rejection. It is to be understood that coating. Any basis metal corrosion or blistering of the coating examined for corrosion of the basis metal or blistering of the designation.

Table 5. The test is described in detail in the referenced ASTM corresponding to a specified classification number) as shown in condition number (or for the service condition number corre-

6.6.3.1 The coulometric method described in Test Method B 504 may be used to measure thickness of the chromium, the total thickness of the nickel, and the thickness of the copper. The STEP test, Test Method B 764, which is similar to the coulometric method, may be used to closely estimate the thicknesses of individual layers of nickel in a multilayer coating.

6.6.3.2 The microscopical method described in Test Method B 487 may be used to measure the thickness of each nickel layer and of the copper layer.

6.6.3.3 The X-ray method described in Test Method B 568 may be used when the total thickness of a copper/nickel/chromium composite coating is to be measured, without any indication of the thickness of each individual layer.

6.6.3.4 Other methods may be used if it can be demonstrated that the uncertainty of the measurement is less than 10 %, or less than that of any applicable method mentioned in 6.6.3. Other methods such as B 499 and B 530, as outlined in Guide B 659, may be used if agreed upon between the purchaser and manufacturer.

6.7 Corrosion Testing:

6.7.1 Coated articles shall be subjected to the corrosion test for a period of time that is appropriate for the particular service condition number (or for the service condition number corresponding to a specified classification number) as shown in Table 5. The test is described in detail in the referenced ASTM designation.

Note 6—There is no direct relation between the results of an accelerated corrosion test and the resistance to corrosion in other media, because several factors, such as the formation of protective films, influence the progress of corrosion and vary greatly with the conditions encountered. The results obtained in the test should, therefore, not be regarded as a direct guide to the corrosion resistance of the tested materials in all environments where these materials may be used. Also, performance of different materials in the test cannot always be taken as a direct guide to the relative resistance of these materials in service.

6.7.2 After the article has been subjected to the treatment described in the relevant corrosion test method, it shall be examined for corrosion of the basis metal or blistering of the coating. Any basis metal corrosion or blistering of the coating shall be cause for rejection. It is to be understood that occasional widely scattered, small corrosion defects such as surface pits may be observed after the testing period. In general, “acceptable resistance” shall mean that such defects are not, when viewed critically, significantly defacing or otherwise deleterious to the function of the electroplated part. A method of rating corrosion is given in Practice B 537.

6.7.3 Surface deterioration of the coating itself is expected to occur during the testing of some types of coatings. The extent to which such surface deterioration will be tolerated shall be specified by the purchaser.

6.8 STEP Test Requirements:

6.8.1 The electrode potential differences between individual nickel layers shall be measured for multilayer coatings corresponding to SC5, SC4, and SC3 in accordance with Test Method B 764 (STEP Test).

Note 7—Universally accepted STEP values have not been established but some agreement in the value of ranges has been obtained. The STEP values depend upon which two nickel layers are being measured.

The STEP potential difference between the semi-bright nickel layer and the bright nickel layer has an accepted potential range of 100 to 200 mV with a typical range of 110 to 140 mV. For all combinations of nickel layers, the semi-bright nickel layer is more noble (cathodic) than the bright nickel layer.

The STEP potential difference between the high-activity nickel layer and the bright nickel layer in triple-layer coatings has an accepted potential range of 15 to 35 mV. The high-activity nickel layer is more active (anodic) than the bright nickel layer.

The STEP potential difference between the bright nickel layer and a nickel (particle nickel) layer between the bright nickel layer and the chromium layer has an accepted potential range of 0 to 30 mV. The bright nickel layer is more active (anodic) than the particle nickel layer prior to chromium.

6.9 Sulfur Content:

6.9.1 The sulfur content of the nickel deposit shall meet the maximum or minimum values as stated in 6.2.4.1 and Table 6.

6.9.2 A method to determine sulfur is presented in Appendix X3. Any reliable method may be used.

6.10 Density and Measurement of the Discontinuities in Chromium:

6.10.1 The density of cracks or pores in microcracked or microporous chromium deposits shall meet minimum values. Microcracked chromium shall have more than 30 cracks/mm (300 cracks/cm) in any direction over the whole of the significant surface. Microporous chromium shall contain a minimum of 10,000 pores/10 by 10 mm square (10,000

<table>
<thead>
<tr>
<th>Basis Metals</th>
<th>Service Condition No.</th>
<th>CASS Method B 368</th>
<th>Corrodokote Method B 380</th>
<th>Acetic-salt Method G 85</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steel, zinc alloy, or copper and</td>
<td>SC 5</td>
<td>66</td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>copper alloy, stainless steel and aluminum alloys</td>
<td>SC 4  SC 3  SC 2  SC 1</td>
<td>22  16  8</td>
<td>Two 16-h cycles 16 4 8</td>
<td>114 96 24 8</td>
</tr>
</tbody>
</table>

TABLE 7 Corrosion Tests Appropriate for Each Service Condition Number

Basis Metals | Service Condition No. | CASS Method B 368 | Corrodokote Method B 380 | Acetic-salt Method G 85 |
---|---|---|---|---|
Steel, zinc alloy, or copper and stainless steel and aluminum alloys | SC 5  SC 4  SC 3  SC 2  SC 1 | 66  22  16  8 | Two 16-h cycles 16 4 8 | 114 96 24 8 |

B 456 – 03
pores/cm²) in any location over the whole of the significant surface. The cracks and pores shall be invisible to the unaided eye.

6.10.2 A method for measuring the discontinuities, referred to as Dubpernell sites, is given in Appendix X4. A method for measuring the number of corrosion sites formed during corrosion, referred to as active sites, is given in Appendix X5.

7. Sampling Requirements

7.1 The sampling plan used for the inspection of a quantity of coated articles shall be as agreed upon by the purchaser and supplier.

Note 8—Usually, when a collection of coated articles, the inspection lot, is examined for compliance with the requirements placed on the coating, a relatively small number of the articles, the sample, is selected at random and is inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Three standards, Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for the sampling inspection of coatings.

Test Method B 602 contains four sampling plans, three for use with tests that are non-destructive and one when they are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 identifies the plan to be used.

Guide B 697 provides a large number of plans and also gives guidance on the selection of a plan. When Guide B 697 is specified, the buyer and seller need to agree on the plan to be used. Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numerical value and certain statistical requirements must be met. Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Test Method B 762 identifies the plan to be used.

Note 9—When both destructive and nondestructive tests exist for the measurement of a characteristic, the purchaser needs to state which is to be used so the proper sampling plan is selected. A test may destroy the coating but in a noncritical area; or, although it may destroy the coating, a tested part may be reclaimed by stripping and recoating. The purchaser needs to state whether the test is to be considered destructive or nondestructive.

7.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specifications, that have been coated by a single supplier at one time or approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

7.3 If separate test specimens are used to represent the coated articles in a test, the specimens shall be of the nature, size, and number and be processed as required in Appendix X2, Appendix X3, Appendix X4, and Appendix X5. Unless a need can be demonstrated, separately prepared specimens shall not be used in place of production items for nondestructive tests and visual examination. For destructive tests including determination of adhesion, ductility, sulfur contents, the number of discontinuities, and corrosion testing, separately prepared specimens may be used.

8. Packaging

8.1 Parts plated for the U.S. Government and military, including subcontracts, shall be packaged in accordance with Practice D 3951.

9. Keywords

9.1 corrosion; decorative; electrodeposited chromium; electrodeposited copper; electrodeposited nickel

APPENDIXES

(Nonmandatory Information)

X1. DEFINITIONS AND EXAMPLES OF SERVICE CONDITIONS FOR WHICH THE VARIOUS SERVICE CONDITION NUMBERS ARE APPROPRIATE

X1.1 Service Condition No. SC 5 (Extended Very Severe)—Service conditions that include likely damage from denting, scratching, and abrasive wear in addition to exposure to corrosive environments where long-time protection of the substrate is required; for example, conditions encountered by some exterior components of automobiles.

X1.2 Condition No. SC 4 (Very Severe)—Service conditions that include likely damage from denting, scratching, and abrasive wear in addition to exposure to corrosive environments; for example, conditions encountered by exterior components of automobiles and by boat fittings in salt water service.

X1.3 Service Condition No. SC 3 (Severe)—Exposure that is likely to include occasional or frequent wetting by rain or dew or possibly strong cleaners and saline solutions; for example, conditions encountered by porch and lawn furniture; bicycle and perambulator parts; hospital furniture and fixtures.

X1.4 Service Condition No. SC 2 (Moderate)—Exposure indoors in places where condensation of moisture may occur; for example, in kitchens and bathrooms.

X1.5 Service Condition No. SC 1 (Mild)—Exposure indoors in normally warm, dry atmospheres with coating subject to minimum wear or abrasion.
X2. ELONGATION TEST

Note X2.1—Practice B 489 is used to ensure compliance of the type of copper deposit with the appropriate definition given in 6.4. Practice B 489 should be followed with these conditions.

X2.1 Preparation of Test Piece:

X2.1.1 Prepare an electroplated test strip, 150 mm long, 10 mm wide, and 1 mm thick by the following method:

X2.1.1.1 Polish a sheet of the appropriate basis metal, similar to that of the articles being electroplated, except that if the basis metal is zinc alloy the sheet may be of soft brass. (Use a sheet sufficiently large to allow the test strip to be cut from its center after trimming off a border 25 mm wide all around.) Electroplate the polished side of the sheet with copper to a thickness of 25 µm under the same conditions and in the same bath as the corresponding articles.

X2.1.2 Cut the test strip from the electroplated sheet with a flat shear. Round or chamfer the longer edges of the strip, at least on the electroplated side, by careful filing or grinding.

X2.2 Procedure—Bend the test strip with the electroplated side in tension (on the outside), by steadily applying pressure, through 180° over a mandrel of 12 mm diameter. Ensure that contact between the test strip and the mandrel is maintained during bending.

X2.3 Assessment—The electroplating is deemed to comply with the minimum requirement of an elongation of 8% if after testing there are no cracks passing completely across the convex surface. Small cracks at the edges do not signify failure.

X3. DETERMINATION OF SULFUR IN ELECTRODEPOSITED NICKEL (NOTE X3.1)

The following two methods for the determination of sulfur in electroplated nickel are given as guidelines for use to test compliance of the type of nickel deposit with the appropriate definition given in 6.2.4.1. They represent methods that have been used with success commercially; they are not ASTM standards, nor is it the intent in publishing these methods to preclude the use of other methods or variations in these methods.

X3.1 Total Sulfur in Electroplated Nickel by Combustion-Iodate Titration

X3.1.1 Scope—This method covers the determination of sulfur in concentrations from 0.005 to 0.5 mass %.

X3.1.2 Summary of Method—A major part of the sulfur in the sample is converted to sulfur dioxide (SO\(_2\)) by combustion in a stream of oxygen using an induction furnace. During the combustion, the SO\(_2\) is absorbed in an acidified starch-iodide solution and titrated with potassium iodate solution. The latter is standardized against steels of known sulfur content to compensate for characteristics of a given apparatus and for day-to-day variation in the percentage of sulfur recovered as SO\(_2\). Compensation is made for the blank because of accelerators and crucibles.

Note X3.1—Instruments are available for measuring the sulfur dioxide from combustion by infrared detection methods and using built-in computers to integrate and display the sulfur content as a percentage.

X3.1.3 Interferences—The elements ordinarily present in electroplated nickel do not interfere.

X3.1.4 Apparatus—Induction heating apparatus for determination of sulfur by direct combustion as described in Practices E 50 (Apparatus No. 13).

X3.1.5 Reagents:

X3.1.5.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first determined that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

X3.1.5.2 Purity of Water—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D 1193.

X3.1.5.3 Hydrochloric Acid (3 + 97)—Mix 3 volumes of concentrated hydrochloric acid (HCl) (sp gr 1.19) with 97 volumes of water.

X3.1.5.4 Iron (Low-Sulfur) Accelerator—Chips.

X3.1.5.5 Iron (Low-Sulfur) Accelerator—Powder.

X3.1.5.6 Potassium Iodate, Standard Solution A (1 mL = 0.2225 mg S)—Dissolve 0.2225 g of potassium iodate (KIO\(_3\)) in 900 mL of water and dilute to 1 L.

X3.1.5.7 Potassium Iodate, Standard Solution B (1 mL = 0.02 mg S)—Transfer 200 mL of potassium iodate Solution A (1 mL = 0.1 mg S) to a 1-L volumetric flask, dilute to volume, and mix.

Note X3.2—The sulfur equivalent is based on the complete conversion of sulfur to sulfur dioxide. The recovery of sulfur as the dioxide may be less than 100%, but it is consistent when the temperature and the rate of oxygen flow are maintained constant. An empirical factor must be determined by an analysis of a standard sample.

X3.1.5.8 Starch-Iodide Solution—Transfer 1 g of soluble or arrowroot starch to a small beaker, add 2 mL of water, and stir until a smooth paste is obtained. Pour the mixture into 50 mL of boiling water. Cool, add 1.5 g of potassium iodide (KI), stir until dissolved, and dilute to 100 mL.

X3.1.5.9 Tin (low sulfur) Accelerator, granular.

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8 Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K.; and the United States Pharmacopoeia and National Formulary, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.
X3.1.6 Standards—Standards for calibration are National Institute of Standards and Technology steels of the proper sulfur content.

X3.1.7 Sample Preparation:
X3.1.7.1 Prepare a test panel of cold-rolled steel 150 mm long by 100 mm wide by 1 mm thick or any other convenient size. Clean, acid dip, and electroplate with approximately 7.5 \( \mu \)m of an adherent nickel deposit and thoroughly rinse. Buffed nickel or buffed stainless steel may also be used as alternatives to steel electroplated with nickel.

X3.1.7.2 Passivate the test panel anodically at 3 V for 5 to 10 s in a hot alkaline cleaner (temperature 70 to 80°C) containing 30 g/L of sodium hydroxide (NaOH) and 30 g/L of trisodium phosphate (Na\(_3\)PO\(_4\)) or 60 g/L of any other suitable anodic alkaline cleaner.

X3.1.7.3 Coat the passivated test panel with 25 to 37 \( \mu \)m of nickel deposited from the same solution using the same parameters as for the coated articles represented by the test specimen.

X3.1.7.4 Remove the edges of the electroplated panel with a hand or power shear or any other convenient method that permits ready separation of the test foil.

X3.1.7.5 Separation from the panel, wash the nickel foil electroplate with water to remove salts and blot dry. Cut into pieces 2 to 3 mm per side with a scissors. Transfer to a 100-mL beaker, cover with water, and heat to boiling. Pour off the water and cover. With the oxygen flow adjusted, raise the crucible, close the furnace, and turn on the power. Burn the sample for 8 to 10 min. Titrate continuously with the KIO\(_3\) solution at such a rate as to maintain as nearly as possible the original intensity of the blue color. The end point is reached when the original blue color is stable for 1 min. Record the final buret reading and drain the titration vessel through the exhaust stopcock.

X3.1.10.4 Blank—Determine the blank by placing the same amount of accelerators used in the test sample in a pre-ignited crucible. Cover and proceed as in X3.1.10.3.

X3.1.11 Calculation—Calculate the sulfur factor of the potassium iodate as follows:

\[
\text{Sulfur factor, g/unit volume} = \frac{A \times B}{(C - D) \times 100}
\]

where:
- \(A\) = grams of standard sample used,
- \(B\) = percent sulfur in the standard sample,
- \(C\) = millilitres of KIO\(_3\) solution required for titration of the standard sample (Note X3.5), and
- \(D\) = millilitres of KIO\(_3\) solution required for titration of the blank (Note X3.5).

Note X3.5—Or apparent percentage of sulfur for “direct-reading” burets.

X3.1.11.1 Calculate the percentage of sulfur in the test sample as follows:

\[
\text{Sulfur, mass %} = \frac{(E - D)F}{G} \times 100
\]

where:
- \(E\) = KIO\(_3\) solution required for titration of the test sample (Note X3.5), mL,
- \(D\) = KIO\(_3\) solution required for titration of the blank, mL,
- \(F\) = average sulfur factor of the KIO\(_3\) for the standards used (see X3.1.11), g/unit volume, and
- \(G\) = sample used, g.

X3.2 Determination of Sulfur in Electroplated Nickel by the Evolution Method

X3.2.1 Scope—This method covers the determination of sulfide sulfur in electroplated nickel in the range from 0.005 to 0.2 mass %.

X3.2.2 Summary of Method—Sulfide sulfur is evolved as hydrogen sulfide (H\(_2\)S) on dissolving the sample of hydrochloric acid (HCl) containing a small amount of platinum as an accelerator for dissolution. The sulfur is precipitated as zinc sulfide (ZnS) in the receiving vessel and then titrated with standard potassium iodate solution. Values are based on potassium iodate (KIO\(_3\)) as the primary standard.

X3.2.3 Apparatus:
X3.2.3.1 The apparatus is shown in Fig. X3.1. It may be assembled using a 50-mL Erlenmeyer flask with a No. 19/38 outer joint. A wash bottle fitting with a No. 19/38 inner joint can be cut to fit the 50-mL flask. The exit tube can be bent and connected to the 6-mm gas tube with tubing.

Note: X3.3—Always fill the titration vessel to the same point.

Note: X3.4—The oxygen flow rate may be adjusted to meet the requirements of individual operators or equipment; however, the flow rate must be the same for the test samples and the standard samples.

X3.1.10.3 After the unit has been at operating temperature for at least 45 s, place the covered crucible containing the sample and accelerators on the pedestal. With the oxygen flow

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X3.2.3.2 A nitrogen cylinder with valves and pressure regulator.

X3.2.3.3 Buret, 10-mL.

X3.2.4 Reagents:

X3.2.4.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.8 Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

X3.2.4.2 Purity of Water—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D 1193.

X3.2.4.3 Ammoniacal Zinc Sulfate Solution—Dissolve 50 g of zinc sulfate (ZnSO₄·7H₂O) in 250 mL of water, add 250 mL of ammonium hydroxide (NH₄OH, sp gr 0.90) and mix. Transfer to a flask and allow to stand about 24 h and filter into a polyethylene bottle.

X3.2.4.4 Hexachloroplatinic Acid Solution (10 g/L)—Dissolve 0.5 g of hexachloroplatinic acid (H₂PtCl₆·6H₂O) in about 40 mL of water, add 5 mL of hydrochloric acid (HCl sp gr 1.19), and dilute to 50 mL.

X3.2.4.5 Hydrochloric Acid-Platinum Chloride Solution—Prepare 500 mL of diluted hydrochloric acid (HCl sp gr 1.19 part acid in 1 part water). Add 2.5 mL of the hexachloroplatinic acid solution and mix.

X3.2.4.6 Potassium Iodate, Standard Solution (0.1 N)—Dry the crystals of potassium iodate (KIO₃) at 180°C for 1 h. Dissolve 3.570 g of the KIO₃ in about 200 mL of water, transfer to a 1-L volumetric flask, dilute to volume, and mix.

X3.2.4.7 Potassium Iodate, Standard Solution (0.005 N)—Transfer 25 mL of the 0.1 N KIO₃ solution to a 500-mL volumetric flask with a pipet, dilute to volume, and mix.

X3.2.4.8 Starch Solution (10 g/L)-Potassium Iodide (50 g/L) Solution—Add about 5 mL of water to 1 g of soluble starch with stirring until a paste is formed and add to 100 mL of boiling water. Cool, add 5 g of potassium iodide (KI), and stir until the KI is dissolved.

X3.2.5 Sample Preparation—Prepare sample as outlined in X3.1.7.

X3.2.6 Weight of Sample—Select and weigh to the nearest 0.1 mg an amount of sample as follows:

<table>
<thead>
<tr>
<th>Expected Sulfur Content, mass %</th>
<th>Weight of Sample, g</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.005 to 0.07</td>
<td>1.0 ± 0.02</td>
</tr>
<tr>
<td>0.05 to 2</td>
<td>0.4 ± 0.02</td>
</tr>
</tbody>
</table>

X3.2.7 Procedure:

X3.2.7.1 Weigh the specified amount of sample to the nearest 0.1 mg and transfer to the 50-mL evolution flask.

X3.2.7.2 Add 20 mL of water and 3 mL of ammoniacal zinc sulfate solution to the receiving flask.

X3.2.7.3 Adjust the hot plate to maintain the temperature of 25 mL of water in a 50-mL Erlenmeyer flask at 80°C.

X3.2.7.4 Add 15 mL of the hydrochloric acid-hexachloroplatinic acid solution to the sample. Assemble the apparatus as shown in Fig. X3.1 and start a very gentle stream of nitrogen through the system.

Note X3.6—A flow of about 30 cm³/min is satisfactory. If the sample dissolves rapidly, the flow should be decreased during the time hydrogen is freely liberated.

X3.2.7.5 Continue the heating and flow of nitrogen until the sample is completely dissolved, then continue for 5 min (Note X3.6). Separate the gas delivery tube from the evolution head and remove the receiving flask with the delivery tube.

Note X3.7—The solution in the receiving flask will remain alkaline throughout the dissolution period if the hot plate temperature and the nitrogen flow are properly adjusted. Additional ammoniacal zinc sulfate solution may be added, if necessary, but the sample should be discarded if the receiving solution becomes acidic (less than pH 7 by test paper).

X3.2.7.6 Add 1 mL of the starch-iodide solution and 5 mL of diluted HCl (1 + 1) and mix. Titrate immediately with standard potassium iodate from a 10-mL buret to the first blue color. Draw some of the solution into the delivery tube with a rubber bulb and release along the neck of the flask to wash down any adhering zinc sulfide. Swirl the solution to wash the outside of the tube. Continue the titration to a permanent blue color.

X3.2.7.7 Run a blank titration to the same starch-iodine color on a mixture of 20 mL of water, 3 mL of ammoniacal zinc sulfate, 1 mL of starch-iodate solution and 5 mL of diluted hydrochloric acid (1 part HCl sp gr 1.19 and 1 part water) in a 50-mL Erlenmeyer flask.

X3.2.8 Calculations—Calculate the mass percent of sulfide sulfur as follows:

\[
\text{Sulfide sulfur, mass %} = \frac{(A-B) \times 0.005 \times 0.016 \times 100}{W} \quad (X3.3)
\]

where:

\[A\] is the weight of sample

\[B\] is the weight of blank

\[W\] is the weight of sample expressed in grams
\[ A = 0.005 \text{ N KIO}_3 \text{ solution used for the sample titration, mL,} \]
\[ B = 0.005 \text{ N KIO}_3 \text{ solution used in the blank, mL, and} \]
\[ W = \text{sample used, g.} \]

**X4. DETERMINING THE NUMBER OF DISCONTINUITIES IN CHROMIUM ELECTROPLATING (DUBPERNELL TEST)**

**X4.1 Principle of the Method**—Copper will be deposited on nickel exposed through discontinuities in chromium but not on the chromium, provided that potential is properly controlled (kept low enough to avoid activation of passive chromium).

**X4.2 Preparation of Test Piece:**

**X4.2.1** Mask all edges not covered by the chromium with a nonconductive paint or pressure sensitive tape, including the wire used to make contact to the cathode bar. After masking, clean the specimen by soaking in a hot alkaline cleaner until the surface is free of water breaks. A mild scrubbing with a soft brush is helpful. Follow the cleaning by a thorough rinse in cold deionized water, then a dip in a 5% by mass solution of H\textsubscript{2}SO\textsubscript{4}.

**X4.2.2** Make freshly cleaned sample anodic at 0.8V for 30 s in the copper plating bath, then switch to cathodic (see Fig. X4.1) at approximately 0.2 to 0.4V, for 2 min (see Note X4.1 and Note X4.2). (Warning—Do not go beyond the specified anodic voltage or time because nickel will slowly dissolve or become passivated.)

Bath formulation—(non-critical)
- CuSO\textsubscript{4}·5H\textsubscript{2}O 1 m (250 g/L)
- H\textsubscript{2}SO\textsubscript{4} (SpG 1.84) 0.5 m (50 g/L)
- Temperature (20 to 25°C)
- Anode (copper)
- Live entry

**X4.2.3** Following copper electroplating, carefully remove the specimen, rinse in cold then hot deionized water, and air dry. The specimen should not be wiped where pores or cracks are to be counted, nor should the part be force air dried. Drying can be accelerated by following the last water rinse by a rinse with alcohol (ethanol) or other volatile water miscible solvent.

**X4.2.4** The copper deposits only on the underlying nickel that is exposed through discontinuities (pores and cracks) in the chromium.

**X4.3 Assessment:**

**X4.3.1** The number of discontinuities in the chromium can be estimated by counting the copper nodules deposited within a known area of the specimen or the number of cracks in a known length. These determinations are facilitated with a metallurgical microscope fitted with a calibrated reticle in the eyepiece, or from photomicrographs taken of a representative field of the specimen. (See Appendix X5 for a guide to the determination of active: corrosion sites in the chromium layer.)

**X4.3.2** Current measured or recorded during the cathodic cycle, or both, serves as a reliable indicator of porosity. If current remains low (<1 mA/cm\textsuperscript{2}) during the cathodic cycle, porosity is low. Rapidly rising current (\(\Delta I/\Delta t \approx 1\) to 2 mA/min) and high (2 to 4 mA/cm\textsuperscript{2}) final current is indicative of high porosity. Use of a strip chart recorder provides a permanent record of the test current. With experience, direct counts of nodules of Cu deposited can be reduced to periodic verifications as the \(I-t\) signature. A qualitative visual check (microscopically) will then suffice for regular routine use.

**Note X4.1**—Exact potential used is dependent on anode-cathode spacing. At a distance of 8 to 10 cm, 0.2 V usually produces the desired deposit. As spacing increases, the potential can be increased to 0.4 V.

**Note X4.2**—After cleaning, anodic treatment to repassivate chromium

\[ E_A = \frac{R_1 + R_3}{R_1 + R_2 + R_3} \]
\[ E_C = \frac{R_1}{R_2 + R_3 + R_4} \]

**FIG. X4.1 Schematic Diagram of a Switching Apparatus to Conveniently Control Polarity and Voltage During Porosity Testing via Copper Deposition**
is essential. Plating time can be varied from 1 to 5 min. Two minutes has been found to be near optimum. With highly porous chromium, longer times incur risk of merging the deposit nodules, giving rise to ambiguities in counting pores (nodules).

X4.3.3 Precautions—Do not exceed 0.6 V cathodic. High cathodic potentials can activate chromium locally, giving rise to spuriously high nodule counts. If this condition is suspected, it can be tested by gently wiping the copper off with a tissue. If copper adheres to specimen, it is probable the cathodic potential was too high, thus depositing copper on the chromium instead of just in the pores.

X5. Determining the Number of Active Sites After Corrosion

X5.1 Principle of the Method—Some of the discontinuities determined in Appendix X4.1 might not become corrosion sites. The sites that actually corrode are determined by stripping the chromium after corrosion and the surface pits in the nickel are counted and referred to as “active sites.”

X5.2 Before testing, the part should be cleaned to eliminate water breaks. Magnesium oxide, warm water and soap, or solvents, or a combination thereof, might be necessary for thorough cleaning.

X5.3 To develop the active corrosion sites, subject the part to the number of hours of CASS exposure corresponding to the corrosion specification for the required service condition and thickness specification. After CASS testing, rinse the part in warm water to remove the salt layer. If the part contains corrosive product staining, it can be washed with a very mild sponge in warm water but the part should not be subjected to any abrasive cleaning. After drying either by hot air or solvent such as methanol, the part should be examined at a magnification between 100 and 200x and the active pore size and density determined. The corroding pore sites are typically distinctly larger than the uncorroded sites and have a darker and rougher texture. By means of photographs at a known magnification, the active pore sites can be counted and the active sites per area calculated. Different photographs should be taken at the same magnification at all significant surfaces and current densities to record the differences in pore density and size after corrosion. As a guide, the average diameter of the active sites should not exceed 0.032 mm (0.00125 in.) and the largest individual site should not exceed 0.0635 mm (0.0025 in.). Comparisons with known pore density charts can be used for determination of the sites per square centimetre.

X5.4 Even though the necessary work has not been conducted to establish a correlation between active corrosion sites and starting sites as measured by Dubpernell, it appears that for a given current density and deposit system there is a correlation between the number of pores before corrosion and the number of active corrosion sites after CASS testing. Once this correlation has been established at a given installation, a simple 100 to 200 X viewing of a part might nondestructively indicate the service performance of the part after corrosion. The desired number of active corrosion sites has not been definitely established. This number depends somewhat on the size of the pores before and after corrosion, the thickness and potentials of the deposit system, and the desired trade-off between basis metal corrosion and staining in CASS.
Standard Test Method for Measurement of Impedance of Anodic Coatings on Aluminum¹

This standard is issued under the fixed designation B 457; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes the conditions and equipment for measuring the impedance of anodic coatings on aluminum. Such measurements have been used to evaluate the quality of seal of an anodic coating. The test method does not prescribe the procedure for producing the anodic coating, nor the postanodizing treatment usually described as “sealing.”

1.2 This test method is applicable to the rapid, nondestructive testing of anodic coatings. The interpretation of results and correlation of data with service experience and other tests are not within the scope of this test method.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Nature of Test

2.1 Impedance is an electrical characteristic described as the total opposition of all circuit elements to the flow of alternating current. Inductive effects of anodic coatings are negligible and the impedance is presumed to be made up of resistance and capacitive reactance according to the following relationships:

\[ Z = \sqrt{R^2 + X_c^2} \]  

(1)

and

\[ X_c = 1/2 \pi f C \]  

(2)

where:

- \( Z \) = impedance,
- \( R \) = resistance,
- \( X_c \) = capacitive reactance,
- \( f \) = frequency, and
- \( C \) = capacitance.

2.2 Both resistance and capacitance are associated with a material constant and a geometric factor:

\[ R = r (l/A) \]  

(3)

\[ C = e(A/l) \]  

(4)

where:

- \( R \) = resistance,
- \( r \) = specific resistance,
- \( l \) = length of conductor, and
- \( A \) = area of cross section of conductor.

2.3 For measuring impedance of anodic coatings, the material constants are partly associated with the anodic coating and partly with the electrolyte employed in the test cell. For simplification these constants are assumed not to vary. The variations in measured impedance then depend directly on geometric factors.²

3. Apparatus

3.1 Impedance Bridges—Commercially available a-c impedance bridges with the following characteristics are satisfactory:³

3.1.1 Range—1 µF (1000 nF) to 0.0001 µF (0.1 nF) full scale with a dissipation factor of 0 to 2.

3.1.2 Frequency—1000 Hz.

3.1.3 Accuracy—1 %.

3.1.4 Voltage—Adjustable so as to be not greater than 0.5 V as applied to the test specimen.

² The Alcoa Impedance Test for Anodic Coatings (AZTAC) evaluates a 0.129-cm² (0.02-in.²) test area and expresses impedance in kilohms. Instructions are available from Alcoa Process Development Laboratories, P.O. Box 2970, Pittsburgh, PA 15230.

³ The Z-Scope, formerly manufactured by Twin City Testing Corp., Tonawanda, NY was designed to measure the impedance of anodic coatings on aluminum. AZTAC values (the impedance of a 0.129-cm² (0.02-in.²) test area) can be read directly in kilohms with this instrument.
3.2 Cell (Fig. 1) for containing the electrolyte during test. It may be constructed from the following components:

3.2.1 Glass Tube, 9 mm (0.35 in.) in outside diameter by 9 cm (3.54 in.).

3.2.2 Rubber Gasket or Seal.

3.2.3 Rubber Stopper—Any convenient size with a hole in center to accommodate the glass tube.

3.2.4 Platinum or Type 300 Stainless Steel Counter Electrode, 9 cm (3.54 in.) long by 0.75 to 1.0 mm (0.030 to 0.040 in.) diameter wire.

3.3 Wire Leads, two, insulated, approximately 1 m (40 in.) long complete with C-clamps or alligator clips shall be used for making connections to the test specimen and cell electrode.

3.4 Support Strand, complete with a utility clamp to hold the electrolytic test cell.

3.5 Glass or Stainless Steel Tank containing pure boiling water is required for conditioning the test specimen.

3.6 Container for Cell Standardization—A 150 to 500-mL Type 300 stainless steel beaker or standard 1-in. 2 test cell may be used.

4. Electrolyte

4.1 The electrolyte shall be a 3 1/2 % solution of sodium chloride prepared by dissolving 36 g of the salt in 1 L of distilled or deionized water.

5. Test Specimen

5.1 The area to be tested should be sufficiently large to accommodate the test cell, sufficiently flat to prevent leakage of the solution from the probe, and free from mechanical defects such as scratches. After an anodic coating has been sealed, moisture is lost from the coating thus producing an increase in impedance. The rate at which moisture is lost increases as the relative humidity decreases; therefore, the relative humidity of the testing environment should be controlled and never below 25 %. Specimens should be tested within 1 h after the anodic coating has been sealed. If this cannot be done or if the part has been subjected to an elevated temperature drying treatment, the test area should be conditioned as follows:

5.1.1 Wipe the test area with acetone, methylethylketone, trichloroethylene, or similar solvent.

5.1.2 Immerse the test area in pure boiling water for 30 s.

5.1.3 Rinse in cold water.

5.1.4 Blot dry and test without further delay.

6. Procedure

6.1 Penetrate the anodic coating in a small area with a sharp instrument and connect the lead from the low-impedance terminal of the bridge to the exposed aluminum substrate.

6.2 Connect the other lead from the bridge to the counter electrode of the test cell. Be certain the lead wires are not twisted around each other. Turn on the bridge and allow it to warm up.

6.3 Place the test cell on the area to be tested and apply enough pressure, 490 to 1230 MPa (71 to 178 psi) to prevent leakage of the electrolyte (the cell is spring-loaded to ensure sufficient and constant pressure). Make certain the bottom of the gasket on the test cell is level with the test surface.

6.4 Fill the test cell at least half full with electrolyte and commence a 2-min soak period. The counter electrode should not contact the test specimen.

6.5 Turn on and obtain a preliminary “balance” of the bridge in accordance with the manufacturer’s instructions (not necessary if a direct-reading instrument is used).

6.6 After the electrolyte has been in contact with the test surface for a period of 2 min, obtain a final balance of the bridge and record the dissipation factor (tan δ). Next, record the capacitance (in nanofarads) as indicated by either the balancing series component (CS) or the balancing parallel component (CP).

6.7 Calculate the impedance, Z, in kilohms, as follows: 7

\[ Z = \frac{159}{C_s \cos \delta} \]  (5)

6.8 Different locations should be measured until at least two values are within 10 %. These are then averaged and multiplied by the correlation factor (See Section 7).

7. Cell Standardization

7.1 Impedance is inversely proportional to the test area. The apparatus described herein tests an area of 0.129 cm² (0.02 in.²). Use of this relatively small test area facilitates the measurement of small parts and curved surfaces. It may be

---

4 Another test cell satisfactory for use with this method is the one used with the Kocour Electronic Thickness Tester Model 995 available from Kocour Co., 4800 S. St. Louis Ave., Chicago, IL.

5 Type A seal available from Kocour Co. or Twin City Testing Corp., 2440 Franklin Ave., St. Paul, MN, has been found satisfactory.

6 Available from Twin City Testing Corp.

7 Direct-reading instruments, such as the Z-Scope, are available, and the use of such equipment obviates mathematical computations (see 3.1).
desirable to modify this apparatus so that larger areas can be measured. Because this area may vary owing to variations in cell gasket size and wear during use, the cell should be standardized daily.

7.2 Cell standardization is accomplished by measuring the impedance on a 6.45-cm² (1-in.²) surface and comparing this measurement with results obtained with the cell. The 6.45-cm² (1-in.²) test surface must be free from surface scratches. The required area may be obtained by appropriately masking a larger anodized specimen using wax of lacquer. Several applications of the masking material will ensure against uncoated areas, especially at edges. Standard 1-in.² test cells are available and may be used as an alternative way of arriving at a 1-in.² area. If this type cell is used beaker immersion is not required.

7.3 Immerse the specimen with the 6.45-cm² (1-in.²) area of exposed anodic coating in a stainless steel beaker containing the 3½ % sodium chloride electrolyte. Connect one lead from the impedance bridge to the beaker, the other to an area of exposed aluminum on the test specimen.

7.4 Measure the impedance as outlined in Section 6, and record as \( A \).

7.5 Using the test cell, make at least two impedance measurements and average on this same area of the specimen used for standardization. Record this as \( B \). If the two measurements are not within 10 %, an additional measurement should be made and the two values within 10 % averaged.

7.6 Obtain the correction factor as follows:

\[
\text{Correction factor} = \frac{50A}{B}
\]

8. Temperature Correction

8.1 Impedance decreases with increasing temperature. If measurements are made at widely varying temperatures, they should be adjusted to a constant, that is, 25°C (77°F).

Example:

\[
Z_{27°F} = Z_{T}[1 - 0.011(T - 77)]
\]

where:

- \( Z_{T} \) = impedance value recorded at ambient,
- \( T \) = ambient temperature, °F, and
- \( Z_{27°F} \) = corrected reading.

9. Interpretation of Results

9.1 The relationship between impedance value and quality of the anodic coating shall be as agreed by the manufacturer and the purchaser. A minimum acceptable value can be established for any anodic coating thickness being used. Generally, higher values should be obtained with thicker anodic coatings. However, an increase in coating thickness does not compensate for a poor seal. Low values will be obtained regardless of coating thickness when the quality of seal is poor.
1. Scope

1.1 This guide describes two processes used for plating on magnesium and magnesium alloys: direct electroless nickel plating and zinc immersion. Some users report that the direct electroless nickel procedure does not produce quite as high a level of adhesion as zinc immersion.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see 5.1.1 and 5.2.9.2.

2. Referenced Documents

2.1 ASTM Standards:
   B 322 Practice for Cleaning Metals Prior to Electroplating

3. Significance and Use

3.1 Metals are electroplated on magnesium for various purposes: solderability, RF grounding, hermetic sealing, wear resistance, corrosion resistance, appearance, and electrical conductivity, for example. Because magnesium is covered with a naturally occurring oxide film, usual procedures for the preparation of metals for autocatalytic or electrolytic plating cannot be used.

4. Reagents

4.1 Purity of Reagents—All acids and chemicals used in this guide are of technical grade. Acid and base solutions are based on the following assay materials:

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Purity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonium hydroxide (NH₄OH)</td>
<td>30 mass %, density 0.895 g/L</td>
</tr>
<tr>
<td>Nitric acid (HNO₃)</td>
<td>67 mass %, density 1.16 g/L</td>
</tr>
<tr>
<td>Sulfuric acid (H₂SO₄)</td>
<td>93 mass %, density 1.40 g/L</td>
</tr>
<tr>
<td>Hydrofluoric acid (HF)</td>
<td>70 mass %, density 1.258 g/L</td>
</tr>
<tr>
<td>Phosphoric acid (H₃PO₄)</td>
<td>85 mass %, density 1.689 g/L</td>
</tr>
</tbody>
</table>

4.2 Purity of Water—All water used for solutions, whether new or recycled, should be monitored for cations, anions, and organic matter that are known to interfere with the plating process.

5. Processes

5.1 Procedures:

5.1.1 Wheel polish and buff parts for smooth, highly polished surfaces. Tumble and burnish small parts. Acid pickle the parts after use of wire brushing or steel wool. (Warning—Because of the high flammability of powdered magnesium, special precautions against fire are important. Polishing and buffing lathes should be kept scrupulously clean. Dust from grinding in and around lathes should be swept up and placed in closed containers for proper disposal. Exhaust systems should be cleaned frequently and the residues handled similarly. If abrasives are used in tumble finishing, similar precautionary techniques should be used for the solids from the abrasive slurry.)

5.1.2 Chemical—Remove oil and grease in an alkaline soak cleaner. Remove other soils and coatings in suitable acid pickling solutions.

Note 1—General information on the cleaning of metals is given in Practice B 322.

5.2 General Electroplating Procedure:

5.2.1 Remove oil, grease, and other soils left from preplating procedures by soak cleaning in an alkaline cleaner suitable for magnesium.

5.2.2 Rinse in cold water.

Note 2—As generally used in rinsing terminology, cold water refers to water from an unheated water supply as opposed to heated water used for drying or other purposes. In some areas, particularly in water, ambient water temperatures may be too low for effective rinsing. In those instances, the rinse water may need to be heated. A minimum temperature of 16°C is recommended for effective rinsing.

5.2.3 Electroclean parts in an alkaline electrocleaner suitable for magnesium. Make the magnesium cathodic at 7.5 to 13 A/dm² and 85°C.

5.2.4 Rinse in cold water.

5.2.5 Pickle in one of the following solutions:
5.2.5.1 Ferric Nitrate Pickle:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chromic acid (Cr₂O₃)</td>
<td>180 g/L</td>
</tr>
<tr>
<td>Ferric nitrate (Fe(NO₃)₃·9H₂O)</td>
<td>40 g/L</td>
</tr>
<tr>
<td>Potassium fluoride (KF)</td>
<td>3.5 g/L</td>
</tr>
<tr>
<td>Temperature</td>
<td>16 to 38°C</td>
</tr>
<tr>
<td>Time</td>
<td>15 s to 2 min</td>
</tr>
</tbody>
</table>

**Note 3**—This pickle removes metal from a surface at the rate of 3 µm/min at 38°C. Where no dimensional change can be tolerated, use of the chromic acid pickle in 5.2.5.2 is recommended.

5.2.5.2 Chromic Acid Pickle:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chromic acid (Cr₂O₃)</td>
<td>180 g/L</td>
</tr>
<tr>
<td>Temperature</td>
<td>16 to 93°C</td>
</tr>
<tr>
<td>Time</td>
<td>2 to 10 min</td>
</tr>
</tbody>
</table>

**Note 4**—The use of chromic acid pickles may leave chromate films on the surface that will reduce the adhesion of the subsequently deposited coating.

5.2.6 Rinse in cold water.
5.2.7 Activate in the following solution:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phosphoric acid (H₃PO₄)</td>
<td>20% by vol</td>
</tr>
<tr>
<td>Ammonium bifluoride (NH₄HF₂)</td>
<td>105 g/L</td>
</tr>
<tr>
<td>Temperature</td>
<td>16 to 38°C</td>
</tr>
<tr>
<td>Time</td>
<td>15 s to 2 min</td>
</tr>
</tbody>
</table>

5.2.8 Rinse in cold water.
5.2.9 Zinc coat in the following solution:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zinc sulfate (ZnSO₄·H₂O)</td>
<td>30 g/L</td>
</tr>
<tr>
<td>Tetrasodium pyrophosphate (Na₄P₂O₇)</td>
<td>120 g/L</td>
</tr>
<tr>
<td>Sodium fluoride (NaF)</td>
<td>5 g/L</td>
</tr>
<tr>
<td>Lithium fluoride (LiF)</td>
<td>2 g/L</td>
</tr>
<tr>
<td>Sodium carbonate (Na₂CO₃)</td>
<td>5 g/L</td>
</tr>
</tbody>
</table>

**Note 5.2.9.1**—Because of the low solubility of the tetrasodium pyrophosphate, it is generally necessary to mix this solution in an elevated temperature from 70 to 85°C. It is also advantageous to alternately add portions of the zinc sulfate and pyrophosphate. When these ingredients are completely dissolved, add and dissolve the balance of the ingredients in the order given.

**Note 5**—Either sodium fluoride or lithium fluoride may be used as a constituent of this bath. Sodium fluoride is widely used, but requires careful control. Potassium fluoride is too hygroscopic and should not be used because the variation in water content makes it impossible to be sure of how much is being added. Lithium fluoride has been found highly desirable because it is soluble only to the proper concentration level and is self-regulating. Thus, the use of lithium fluoride eliminates the need for fluoride dilution. During bath makeup, 3 g/L lithium fluoride is added. This small quantity saturates the solution, and an excess suspended in the bath in a canvas or nylon anode bag automatically replaces any fluoride consumed during the operation.

5.2.9.2 Immerse the parts for 3 to 10 min in the solution operated at 79 to 85°C and agitation mild. Do not use glass or fiberglass equipment of any kind. Careful control is essential for best results. Maintain the pH between 10.2 and 10.4 electrometric (glass electrode) measured at 25°C. (Warning—The glass electrode must be used with caution. Do not allow the electrode to remain in contact with the zincate solution for prolonged periods. Inspect and test the electrode regularly to ensure that no change has occurred from contact with fluoride ion. Colorimetric (paper) methods may be used. However, the colorimetric readings may vary ±0.5 pH units.)

5.2.10 Rinse thoroughly in cold water.

**Note 6**—For alloys M1660, M13120, M13312, or M13310, a double zinc immersion is required. After step 5.2.10, steps 5.2.7-5.2.10 are repeated followed by step 5.2.11. It is advisable to use separate solutions for steps 5.2.7 and 5.2.9 when the double zinc immersion technique is practiced.

5.2.11 Copper strike in either of the following:

**Bath 1:**

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper cyanide (CuCN)</td>
<td>38 to 42 g/L</td>
</tr>
<tr>
<td>Potassium cyanide (KCN)</td>
<td>64.5 to 71.5 g/L</td>
</tr>
<tr>
<td>Potassium fluoride (KF)</td>
<td>28.5 to 31.5 g/L</td>
</tr>
<tr>
<td>Free potassium cyanide</td>
<td>7 to 8 g/L</td>
</tr>
<tr>
<td>pH</td>
<td>9.6 to 10.4</td>
</tr>
<tr>
<td>Temperature</td>
<td>54 to 60°C</td>
</tr>
</tbody>
</table>

**Bath 2:**

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper cyanide (CuCN)</td>
<td>38 to 42 g/L</td>
</tr>
<tr>
<td>Sodium cyanide (NaCN)</td>
<td>50 to 55 g/L</td>
</tr>
<tr>
<td>Rochelle salt (KNaC₄H₄O₆·4H₂O)</td>
<td>40 to 48 g/L</td>
</tr>
<tr>
<td>Free sodium cyanide</td>
<td>7 to 8 g/L</td>
</tr>
<tr>
<td>pH</td>
<td>9.6 to 10.4</td>
</tr>
<tr>
<td>Temperature</td>
<td>54 to 60°C</td>
</tr>
</tbody>
</table>

5.2.11.1 Plate the parts about 6 min. Cathode rod agitation is suggested. With either bath make electrical contact quickly with initial current 5 to 10 A/dm², then lower current to 1 to 2.5 A/dm².
5.2.12 Rinse thoroughly in cold water.
5.2.13 Dip in diluted acid (1% by vol sulfuric acid + 99% by vol water).
5.2.14 Rinse in cold water.
5.2.15 Apply subsequent electrodeposits or autocatalytic nickel in accordance with standard commercial electroplating practice. Use plastisol-coated racks that are fitted with stainless steel or phosphor-bronze rack tips.

5.3 Autocatalytic Nickel Plating Magnesium:

5.3.1 Surface Conditioning—See 5.1.1 and 5.1.2, and 5.2.1-5.2.4.
5.3.2 Pickling—Pickle in (1) chromic acid as directed in 5.2.5.2 or in (2) phosphoric acid (90% by vol phosphoric acid + 10% by vol water) using a lead, glass, ceramic, or rubber-lined tank of polyethylene, polypropylene, or other suitably corrosion resistant material.
5.3.3 Rinse in cold water.
5.3.4 Chemical etch in one of the following:

**Etch 1**—For Alloys Containing Aluminum:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chronic acid (Cr₂O₃)</td>
<td>120 g/L</td>
</tr>
<tr>
<td>Nitric acid (HNO₃) (11% by vol nitric acid + 89% by vol water) to make 1L</td>
<td></td>
</tr>
</tbody>
</table>

**Etch 2**—For Other Magnesium Alloys:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chronic acid (Cr₂O₃)</td>
<td>60 g/L</td>
</tr>
<tr>
<td>Nitric acid (HNO₃) (9% by vol nitric acid + 91% by vol water) to make 1L</td>
<td></td>
</tr>
</tbody>
</table>

Stainless steel tank or tank lined with glass, ceramic, polyvinyl chloride, polyethylene, or other suitably resistant plastic material.

Immerse parts 20 to 60 s in solution at room temperature. Rinse thoroughly and proceed immediately to hydrofluoric acid dip.

**Note 7**—Use the chromic acid pickle where dimensional change cannot be tolerated. Do not use on alloys containing thorium. For thorium-containing alloys and on other alloys where dimensional change

---

1 Magnesium Finishing, The Dow Metal Products Co., Midland, MI.
is not critical, use phosphoric acid pickle. Immerse parts 30 s to 1 min in the phosphoric acid pickle at 21 to 32°C. Metal loss is about 13 µm/surface. If thorium alloys have critical dimensions, use chromic acid pickle and follow with an immersion in a strongly alkaline cleaner to remove possible chromate films.

5.3.5 Dip in hydrofluoric acid (5.5 vol % hydrofluoric acid + 94.5 vol % water) using a tank lined with polyvinyl chloride, polyethylene, or polypropylene. Immerse parts 10 min in solution at room temperature. For treating M11610 or M11800 use a 20 vol % hydrofluoric acid + 80 vol % water solution. Transfer to nickel bath immediately after rinsing. Use of ammonium bifluoride (NH₄HF₂), at 60 to 90 g/L (8 to 12 oz/gal) can often be substituted for hydrofluoric acid. Use of ammonium bifluoride eliminates the hazard of handling concentrated hydrofluoric acid.

**Note:** 8—The zinc immersion coating plus copper strike can be used instead of chemical etching (see Section 1). Follow steps 5.2.5-5.2.11.

5.3.6 Nickel plate in an autocatalytic bath specific for magnesium. The following is an example of a magnesium specific autocatalytic bath:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Basic nickel carbonate (2NiCO₃·3Ni(OH)₂·4H₂O)</td>
<td>10 g/L</td>
</tr>
<tr>
<td>Hydrofluoric acid (HF)</td>
<td>5.2 g/L</td>
</tr>
<tr>
<td>Citric acid (C₆H₈O₇)</td>
<td>(0.6 vol % hydrofluoric acid + 79.4 vol % water)</td>
</tr>
<tr>
<td>Ammonium bifluoride (NH₄HF₂)</td>
<td>10 g/L</td>
</tr>
<tr>
<td>Sodium hypophosphite (NaH₂PO₂·H₂O)</td>
<td>20 g/L</td>
</tr>
</tbody>
</table>

*This bath was patented (U.S. Patent 3,152,009, Oct. 6, 1964, DeLong, H.K.). It was assigned to the Dow Chemical Co., Midland, MI. Other autocatalytic plating solutions that may be used are described in U.S. Patent Nos. 2,983,634, May 9, 1961, Budininkas, P.; 3,121,644, Feb. 18, 1964, Gutzeit, G. and Lee, W.G.; and 3,211,578, Oct. 12, 1965, Gutzeit, G. There are also many specialized proprietary baths available which may be applied directly or after a minimum of 5 µm coating has been applied from any of the above baths. All the patents listed have expired.*

5.3.7 Rinse in cold water. For maximum corrosion resistance when the above plate is the final deposit, immerse for 10 min in (1) chromic acid (CrO₃) (2.5 g/L) or (2) sodium dichromate (Na₂Cr₂O₇·2H₂O) (120 g/L). Operate both solutions at 88 to 100°C. Proprietary compounds are available which operate at room temperature.

5.3.8 Thoroughly rinse in cold water and dry. To improve adhesion, bake at 200°C for 1 h. The deposit should remain adherent and free of blisters.

6. Keywords

6.1 activation; cleaning; deoxidizing; magnesium; preparation; striking

---

Ammonium hydroxide (NH₄OH) (3.9 % by vol ammonium hydroxide + 96.1 % by vol water)

Adjust pH to 5.5 to 6.3

Use a tank lined with polyvinyl chloride plastic, baked-phenolic based enamels, polyethylene, polypropylene, or other suitably resistant material. Use mild mechanical agitation. Immerse parts in nickel solution operated at 77 to 82°C with a pH range of 5.5 to 6.3, measured at 25°C. The bath should be filtered either periodically or continuously. The use of a pump which is sealed or designed for operation in fluoride and with electroless nickel solution is recommended. To ensure adequate filtration, the solution should be filtered in excess of 20 tank volumes per hour through a 5-micron filter. The use of a plastic, magnetically coupled pump will eliminate problems with shaft seals.

**Note:** 9—If heavy electroless nickel deposits are to be applied, the parts can be transferred to the appropriate bath after a thickness of 5 µm has been deposited from this bath.

6.3 Rinse in cold water. For maximum corrosion resistance when the above plate is the final deposit, immerse for 10 min in (1) chromic acid (CrO₃) (2.5 g/L) or (2) sodium dichromate (Na₂Cr₂O₇·2H₂O) (120 g/L). Operate both solutions at 88 to 100°C. Proprietary compounds are available which operate at room temperature.

6.3.8 Thoroughly rinse in cold water and dry. To improve adhesion, bake at 200°C for 1 h. The deposit should remain adherent and free of blisters.
INTRODUCTION

Full utilization of the light weight and high strength of titanium is prevented by the tendency it has to gall and seize and by its lack of corrosion resistance at elevated temperatures. Frequently these limitations can be overcome by electrodepositing upon the titanium a metal with satisfactory properties. Titanium is an active metal that rapidly forms an adherent oxide coating in the presence of oxygen and water. This coating prevents the application of adherent electrodeposits by the more familiar preparative processes. For this reason, the special processes described in this practice were developed.

1. Scope

1.1 This practice describes processes that have been found to be successful in producing adherent electrodeposits of good quality on titanium and certain titanium alloys. Not all of the processes that have been reported as successful are described, but rather three basic ones that have had the widest use. A rather complete listing of the published work on electroplating on titanium is given in the list of references which appear at the end of this practice.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For a specific hazard statement, see 3.1.

2. Referenced Documents

2.1 ASTM Standards:
B 343 Practice for Preparation of Nickel for Electroplating with Nickel

3. Reagents

3.1 Purity of Reagents—All acids and chemicals used in this practice are technical grade. Acid solutions are based upon the following assay materials (Warning—Use hydrofluoric acid with extreme care.):

<table>
<thead>
<tr>
<th>Acid</th>
<th>Purity</th>
<th>Density (g/mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrochloric acid</td>
<td>37 mass %</td>
<td>1.184</td>
</tr>
<tr>
<td>Hydrofluoric acid</td>
<td>60 mass %</td>
<td>1.235</td>
</tr>
<tr>
<td>Hydrofluoric acid</td>
<td>71 mass %</td>
<td>1.260</td>
</tr>
<tr>
<td>Hydrofluoric acid</td>
<td>100 mass %</td>
<td>1.0005</td>
</tr>
<tr>
<td>Nitric acid</td>
<td>69 mass %</td>
<td>1.409</td>
</tr>
</tbody>
</table>

3.2 Purity of Water—Use ordinary industrial or potable water for preparing solutions and rinsing.

4. Process No. 1

4.1 Cleaning—Remove oil, grease, and other soil by appropriate conventional processes such as vapor degreasing, alkaline cleaning, grinding, or blasting.

4.2 Activating—Activation may be done by chemical or electrochemical etching or liquid abrasive blasting. It is possible that all three processes will work equally well on pure titanium and all common alloys; however, only those for which each process has been demonstrated to be successful are given here. The suitability of a process for an alloy not listed should be experimentally determined before committing production parts.

4.2.1 Chemical Etch:

4.2.1.1 The following procedure is suitable for commercially pure titanium and for 6Al-4V, 4Al-4Mn, and 3Al-5Cr.
4.2.1.2 Pickle—Immerse in the following solution, at room temperature, until red fumes are evolved:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>HF (60 mass %)</td>
<td>1 volume and 3 volumes</td>
</tr>
<tr>
<td>HNO₃ (69 mass %)</td>
<td>3 volumes</td>
</tr>
</tbody>
</table>

4.2.1.3 Rinse.

4.2.1.4 Etch—Immerse in the following aequous solution for 20 min (Note that a special formulation is recommended for 3Al-5Cr alloy).

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na₂Cr₂O₇·2H₂O</td>
<td>250 g/L</td>
</tr>
<tr>
<td>Na₂Cr₂O₇·2H₂O</td>
<td>390 g/L</td>
</tr>
<tr>
<td>HF (60 % mass)</td>
<td>48 mL/L</td>
</tr>
<tr>
<td>HF (60 % mass)</td>
<td>25 mL/L</td>
</tr>
<tr>
<td>Temperature</td>
<td>82 to 100°C</td>
</tr>
</tbody>
</table>

NOTE 1—For platinum electroplating on commercially pure titanium, etching may be done by immersion for 5 min in hot (94°C min) concentrated hydrochloric acid followed by rinsing and platinum electroplating.

4.2.2 Rinse.

4.2.3 Electroplate—Electroplate with chromium, with copper from an acid bath, or with nickel from either a Watts or sulfamate bath, or deposit nickel in an autocatalytic bath. If a deposit of some metal other than these three is desired, first apply a nickel coating with a minimum thickness of 1 µm followed by the desired final metal.

4.2.4 Heat Treat:

4.2.4.1 The adhesion of the electrodeposition is mechanical and, therefore, although of a relatively high order of magnitude, it may be less than adequate. If a higher degree of adhesion is desired, use nickel as an intermediate coating and heat treatment. This causes interdiffusion of the nickel and titanium and produces a metallurgical bond. The heat treatment can be performed after all electroplating is applied or immediately after the nickel electroplating. This later approach is used in certain cases, for example, when undesirable diffusion can occur between the nickel and the subsequent deposit.

4.2.4.2 Heat treat in an inert gas atmosphere (for example, argon) for 1 to 4 h at 540 to 800°C. The exact time and temperature should be selected by subjecting electroplated test pieces to adhesion or performance tests or both.

4.2.4.3 If the heat treatment is performed before applying the subsequent deposit, the nickel will have to be activated by subjecting the electroplated coatings to anodic, raising the current above the operating value until local chemical attack of the metal is stopped as evidenced by the cessation of gassing. Then reduce the current to the operating value and etch anodically at 5.4 A/dm² for 15 to 30 min.

4.2.4.4 Excessive current densities will produce electropolishing and inadequate current densities will permit local chemical attack. Both conditions will result in lack of adhesion of the electroplating.

4.2.5 Rinse.

4.2.6 Electroplate—Electroplate with copper from an acid bath or copper from a cyanide bath preceded by a cyanide copper strike, with cadmium from a cyanide bath, with silver from a cyanide bath, or with nickel from a Watts bath.

5. Process No. 2

5.1 Clean—See 4.1.

5.2 Electrochemical Etch:

5.2.1 The following procedure is suitable for commercially pure titanium and 4Al-4Mn alloy. The adhesion produced is purely mechanical but sufficient to pass a bend test and heating in a gas flame.

5.2.2 Pickle—Immerse in the following solution at room temperature until red fumes are evolved:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>HF (60 mass %)</td>
<td>1 volume and 3 volumes</td>
</tr>
<tr>
<td>HNO₃ (69 mass %)</td>
<td>3 volumes</td>
</tr>
</tbody>
</table>

5.2.3 Rinse.

5.2.4 Etch—Immerse in the following solution and make anodic, raising the current above the operating value until local chemical attack of the metal is stopped as evidenced by the cessation of gassing. Then reduce the current to the operating value and etch anodically at 5.4 A/dm² for 15 to 30 min.

6. Process No. 3

6.1 Clean—See 4.1.

6.2 Liquid Abrasive Blasting:

6.2.1 The following procedure is suitable for commercially pure titanium, 3Al-5Cr, 5Al-2Cr-2Mo, 7Al-5Cr, 2.5Al-16V, 4Al-4Mn, 2Fe-2Cr-2Mo, 28Cr-1.5Fe, 6Al-4V, and 3Al-13V-11Cr.

6.2.2 Blasting—Blast all surfaces with a water-abrasive slurry until a uniform appearance is achieved. The grit may be as coarse as 100 mesh or as fine as 1250. Some grits require somewhat more time but do not cause roughening of the surface or dimensional changes. The grit should be used exclusively for blasting titanium to avoid imbedding of contaminants such as iron that can cause local failures in the coating.

6.2.3 Electroplating—Deposit nickel from either an electrolyte or autocatalytic bath. A minimum thickness of 1 µm is required when the nickel is used as a base for subsequent deposits.

6.2.4 Heat Treatment:

6.2.4.1 Heat treat in an inert gas atmosphere (for example, argon) for 1 to 4 h at 540 to 800°C. The exact time and temperature should be selected by subjecting electroplated test pieces to adhesion or performance tests or both.

6.2.4.2 The heat treatment can be performed after all electroplated coatings have been applied or immediately after the nickel electroplating. This latter approach is used in certain cases, for example, when undesirable diffusion can occur between the nickel and the subsequent deposit. If the heat treatment is performed before applying the subsequent deposit,
the nickel will have to be activated before continuing the electroplating. Methods of activation are given in Practice B 343.

REFERENCES

Standard Practice for Preparation of Tungsten and Tungsten Alloys for Electroplating

INTRODUCTION

Because of the high melting point of tungsten, (3410°C) this metal and its alloys are used in ultra-high-temperature applications. However, tungsten oxidizes readily and the oxides offer little or no protection to the metal because they melt or sublime below 2200°C. Tungsten has a body-centered-cubic structure as does chromium. Its coefficient of thermal expansion is 4.3 µm/m · °C; the coefficient of chromium is 6.1 µm/m · °C, therefore, chromium-coated tungsten offers a reasonable match based on crystal structure and coefficient of thermal expansion. The effect of hydrogen must be considered in the design of coating systems for tungsten. Molecular hydrogen when trapped under a coating can result in failure of the part. Although failure can occur if the adhesion of the coating to the tungsten is inadequate, failure within the basis metal as rupture at laminations of stringers has been observed with off-grade tungsten.

1. Scope

1.1 This practice makes recommendations of several reported processes for electroplating on tungsten and its alloys along with comments as a guide in the selection of a processing method for a given application. Because data on methods and results of evaluation with electroplated coatings on tungsten are limited, a recommendation of one method over another cannot be made.

1.2 This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Processes

2.1 The several processes reported for electroplating on tungsten can be classified as either (1) providing as-deposited adhesion of the coatings or (2) depending on diffusion alloy bonding to provide such adhesion. Appraisal of the relative merits of these processes depends on the application for the coating.

2.2 Precleaning:

2.2.1 The need for descaling, solvent cleaning, and alkaline-soak cleaning of tungsten is dictated by the degree of cleanliness of the surface to be coated. Remove gross scale and surface imperfections by mechanical means. Remove grease and oil by organic solvents. Remove saponifiable and dispersible surface dirt by soak cleaning in a hot alkaline cleaner.

2.2.2 Anodic treatment of tungsten surfaces in a 5 to 25 mass % sodium hydroxide solution at 71 ± 6°C and 16 to 25 A/dm² can be used to remove undesired surface metal. Anodic etching in a 10 mass % hydrofluoric acid electrolyte at 24 ± 3°C and 5.4 A/dm² can be used to remove light scale and undesired surface metal before activation and electroplating of tungsten.

2.3 Activation and Electroplating—Two alternative methods are presented below. The selection of one process over the other should be based on preliminary experimentation. Both procedures produce as-deposited adhesion of electroplated metals on tungsten.

2.3.1 Methods 1—Treat the precleaned tungsten in a hydrofluoric acid electrolyte (30 ± 10 mass % HF) at 24 ± 3°C, using 2 to 5 V, 60-Hz ac, and 2 to 5 A/dm² for 1 to 2 min. Either divide the work between two work rods or make one electrode of platinum. After rinsing, the work is electroplated in acid electrolytes.

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2.3.2 Method 2—Electroplate the precleaned tungsten with a chromium strike prior to subsequent electroplating with other metals\textsuperscript{3,4} using the following processing steps:

2.3.2.1 Anodic Etch—Etch anodically at 10 to 30 A/dm\textsuperscript{2} in the following solution for 2 to 5 min.

| KOH | Temperature |
| 30 mass % | 48 to 60°C |

2.3.2.2 Rinse.

2.3.2.3 Acid Neutralize—Immerse for 1 min in 10 mass % sulfuric acid at 27 ± 6°C.

2.3.2.4 Rinse.

2.3.2.5 Chromium Strike—Strike at 15 to 25 A/dm\textsuperscript{2} in the following chromium solution for 1 to 3 min. Enter the solution with the parts cathodic.

| CrO\textsubscript{3} | 250 g/L |
| H\textsubscript{2}SO\textsubscript{4} | 2.5 g/L |
| Temperature | 60 to 72°C |

2.3.2.6 Rinse.

2.3.2.7 Acid Activation—Immerse in 20 mass % hydrochloric acid at 21 to 33°C for 2 to 5 s.

2.3.2.8 Nickel Strike—Strike at 5 to 10 A/dm\textsuperscript{2} in the following nickel solution for 2 to 5 min.

| NiSO\textsubscript{4} \cdot 6H\textsubscript{2}O | 240 g/L |
| H\textsubscript{2}SO\textsubscript{4} | 40 g/L |
| Temperature | 21 to 33°C |

2.3.2.9 Rinse.

2.3.2.10 Electroplate—Electroplate with desired metal.

2.4 Alloying and Heating Effects:

2.4.1 A number of other processes, involving heat treating (927 ± 56°C) to diffusion-alloy bond an electrodeposited coating to the basis tungsten have been proposed but offer no apparent advantage.

2.4.2 Chromium-strike coated tungsten\textsuperscript{3,4} subsequently electroplated with nickel and heated for ½ h at 980°C shows no deterioration of the electroplated coatings. Chromium-tungsten diffusion does not form the weak brittle intermetallic alloy zones such as are evident when nickel, iron, or cobalt coatings on tungsten are similarly heat treated.


Standard Test Method for
Measurement of Metal and Oxide Coating Thickness by
Microscopical Examination of a Cross Section¹

This standard is issued under the fixed designation B 487; the number immediately following the designation indicates the year of
original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A
superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers measurement of the local
thickness of metal and oxide coatings by the microscopical
examination of cross sections using an optical microscope.

1.2 Under good conditions, when using an optical micro-
scope, the method is capable of giving an absolute measuring
accuracy of 0.8 µm. This will determine the suitability of the
method for measuring the thickness of thin coatings.

1.3 This standard does not purport to address all of the
safety concerns, if any, associated with its use. It is the
responsibility of the user of this standard to establish appro-
priate safety and health practices and determine the applica-
bility of regulatory limitations prior to use. (This is especially
applicable to the chemicals cited in Table X2.1.)

2. Referenced Documents

2.1 ASTM Standards:
E 3 Methods of Preparation of Metallographic Specimens²

3. Summary of Test Method

3.1 This test method consists of cutting out a portion of the
test specimen, mounting it, and preparing the mounted cross
section by suitable techniques of grinding, polishing, and
etching. The thickness of the cross section is measured with an
optical microscope.

Note 1—These techniques will be familiar to experienced metallogra-
phers but some guidance is given in Section 5 and in Appendix X1 for less
experienced operators.

4. Significance and Use

4.1 Coating thickness is an important factor in the perform-
ance of a coating in service and is usually specified in a
coating specification.

4.2 This method is suitable for acceptance testing.

5. Factors Influencing the Measurement Result

5.1 Surface Roughness—If the coating or its substrate has a
rough surface, one or both of the interfaces bounding the
covering cross section may be too irregular to permit accurate
measurement. (See X1.4)

5.2 Taper of Cross Section—If the plane of the cross section
is not perpendicular to the plane of the coating, the measured
thickness will be greater than the true thickness. For example,
an inclination of 10° to the perpendicular will contribute a
1.5 % error.

5.3 Deformation of the Coating—Detrimental deformation
of the coating can be caused by excessive temperature or
pressure during mounting and preparation of cross sections of
soft coatings or coatings melting at low temperatures, and also
by excessive abrasion of brittle materials during preparation of
cross sections.

5.4 Rounding of Edge of Coating—If the edge of the coating
cross section is rounded, that is, if the coating cross section is
not completely flat up to its edges, the true thickness cannot be
observed microscopically. Edge rounding can be caused by
improper mounting, grinding, polishing, or etching. It is
usually minimized by overplating the test specimen before
mounting. (See X1.2.)

5.5 Overplating—Overplating of the test specimen serves to
protect the coating edges during preparation of cross sections
and thus to prevent an erroneous measurement. Removal of
covering material during surface preparation for overplating can
cause a low-thickness measurement.

5.6 Etching—Optimum etching will produce a clearly de-
defined and narrow dark line at the interface of two metals. Excessive etching produces a poorly defined or wide line
which may result in an erroneous measurement.

5.7 Smearing—Improper polishing may leave one metal
smear over the other metal so as to obscure the true boundary
between the two metals. The apparent boundary may be poorly
defined or very irregular instead of straight and well defined.
To verify the absence of smearing, the coating thickness should
be measured and the polishing, etching, and thickness mea-
surement repeated. A significant change in apparent thickness

¹ This test method is under the jurisdiction of ASTM Committee B08 on Metallic
and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on
General Test Methods.

² Annual Book of ASTM Standards, Vol 03.01.
indicates that smearing was probably present during one of the measurements.

5.8 Magnification—For any given coating thickness, measurement errors generally increase with decreasing magnification. If possible, the magnification should be chosen so that the field of view is between 1.5 and 3 × the coating thickness.

5.9 Calibration of Stage Micrometer—Any error in calibration of the stage micrometer will be reflected in the measurement of the specimen. Errors of several percent are not unrealistic unless the scale has been calibrated or has been certified by a responsible supplier. The distance between two lines of a stage micrometer used for the calibration shall be known to within 0.2 µm or 0.1 %, whichever is the greater. If a stage micrometer is not certified for accuracy, it should be calibrated. A generally satisfactory means of calibration is to assume that the stated length of the full scale is correct, to measure each subdivision with a filar micrometer, and to calculate the length of each subdivision by simple proportion.

5.10 Calibration of Micrometer Eyepiece:

5.10.1 A filar micrometer eyepiece generally provides the most satisfactory means of making the measurement of the specimen. The measurement will be no more accurate than the calibration of the eyepiece. As calibration is operator dependent, the eyepiece shall be calibrated by the person making the measurement.

5.10.2 Repeated calibrations of the micrometer eyepiece can be reasonably expected to have a spread of less than 1 %.

5.10.3 Some image-splitting micrometer eyepieces have a nonlinearity that introduces an error of up to 1 % for short measurement distances.

5.11 Alignment—Errors can be introduced by backlash in the movement of the micrometer eyepiece. If the final motion during alignment of the hairline is always made in the same direction, this error will be eliminated.

5.12 Uniformity of Magnification—Because the magnification may not be uniform over the entire field, errors can occur if both the calibration and the measurement are not made over the same portion of the field with the measured boundaries centered about the optical axis.

5.13 Lens Quality—Lack of sharpness of the image contributes to the uncertainty of the measurement. Poor quality lenses could preclude accurate measurements. Sometimes image sharpness can be improved by using monochromatic light.

5.14 Orientation of Eyepiece—The movement of the hairline of the eyepiece for alignment has to be perpendicular to the boundaries of the coating cross section. For example, 10° misalignment will contribute a 1.5 % error.

5.15 Tube Length—A change in the tube length of the microscope causes a change in magnification and if this change occurs between the time of calibration and the time of measurement, the measurement will be in error. A change in tube length may occur when the eyepiece is repositioned within the tube, when the focus of the eyepiece tube is changed, and, for some microscopes, when the fine focus is adjusted or the interpupillary distance for binoculars is changed.

6. Preparation of Cross Sections

6.1 Prepare, mount, polish, and etch the specimen so that:
6.1.1 The cross section is perpendicular to the coating;
6.1.2 The surface is flat and the entire width of the coating image is simultaneously in focus at the magnification used for the measurement;
6.1.3 All material deformed by cutting or cross sectioning is removed.
6.1.4 The boundaries of the coating cross section are sharply defined by no more than contrasting appearance or by a narrow, well-defined line.

Note 2—Further guidance is given in Appendix X1. Some typical etchants are described in Appendix X2.

7. Procedure

7.1 Give appropriate attention to the factors listed in Section 5 and Appendix X1.
7.2 Calibrate the microscope and its measuring device with a certified or calibrated stage micrometer.
7.3 Measure the width of the image of the coating cross section at no less than five points distributed along a length of the microsection, and calculate the arithmetic mean of the measurements (see 8.1.5 and 8.1.6).

8. Test Report

8.1 The test report shall include the following information:
8.1.1 The date of test;
8.1.2 The number and title of this test method;
8.1.3 The identification of the test specimens;
8.1.4 The location on the coated item at which the cross section was made;
8.1.5 The measured thickness, in micrometres (millimetres if greater than 1 mm) at each point (7.3), and the length of section over which the measurements were distributed;
8.1.6 The local thickness, that is, the arithmetic mean of the measured thicknesses;
8.1.7 Any deviations from this test method;
8.1.8 Any factors that might influence interpretation of the reported results; and
8.1.9 The name of the operator and testing laboratory.

9. Precision and Bias

9.1 The microscope and associated equipment, its use, its calibration, and the method of preparation of the cross section shall be chosen so as to allow the coating thickness to be determined to within 1 µm or 10 %, whichever is the greater, of the actual coating thickness. Under good conditions, when using an optical microscope, the method is capable of giving an absolute measuring accuracy of 0.8 µm and for thicknesses greater than 25 µm a reasonable error is of the order of 5 % or better.
APPENDIXES

X1. GUIDANCE ON THE PREPARATION AND MEASUREMENT OF CROSS SECTIONS

X1.1 Introduction—The preparation of test specimens and measurement of coating thickness are greatly dependent on individual techniques and there is a variety of suitable techniques available. It is not reasonable to specify only one set of techniques, and it is impractical to include all suitable techniques. The techniques described in this appendix are intended as guidance for metallographers not experienced in measurements of coating thickness. For additional guidance see Methods E 3.

X1.2 Mounting:

X1.2.1 To prevent rounding of the edge of the cross section, the free surface of the coating should be supported so that there is no space between the coating and its support. This is usually achieved by overplating the specimen with a coating at least 10-µm thick of a metal of similar hardness to the coating. For hard, brittle coatings (for example oxide or chromium coatings) tightly wrapping the specimen in soft aluminum foil before mounting has proved successful.

X1.2.2 If the coating is soft, overplating with a metal which is softer will make polishing more difficult, because the softer metal tends to be polished away more rapidly.

X1.2.3 Overplating of zinc or cadmium coatings with copper may cause difficulty because of the tendency, during subsequent etching, of dissolved copper to deposit on the coatings. It is better to overplate zinc with cadmium and vice versa.

X1.3 Grinding and Polishing:

X1.3.1 It is essential to keep the cross-section surface of the mount perpendicular to the coating. This is facilitated by incorporating additional pieces of a similar metal in the plastic mounting, near the outer edges, by periodically changing the direction of grinding (rotating through 90°) and by keeping the grinding time and pressure to a minimum. If, before grinding, reference marks are inscribed on the side of the mount, any inclination from horizontal is easily measurable.

X1.3.2 Grind the mounted test specimens on suitable abrasive paper, using an acceptable lubricant, such as water or mineral spirits, and apply minimum pressure to avoid bevelling of the surface. Initial grinding should employ 100 or 180 grade abrasive to reveal the true specimen profile and to remove deformed metal. Subsequently, use Grades 240, 320, 500, and 600 without exceeding grinding times of 30 to 40 s on each paper; alter the direction of scratches by 90° for each change of paper. A final polish of 2 to 3 min on a rotating wheel charged with 4 to 8-µm diamond paste particles and lubricated with mineral spirits should suffice to remove scratches for final examination. If an especially high degree of surface finish is required, a further treatment, using diamond paste of approximately 1-µm particles, may be employed.

X1.3.3 If very soft materials are being prepared, abrasive particles may become embedded during grinding. This may be minimized by totally immersing abrasive papers in a lubricant during grinding or by using a copious flow of lubricant. If abrasive particles do become embedded, they may be removed by applying a short, light hand polish with metal polish after grinding and before diamond finishing or by one or more cycles of alternate etching and polishing.

X1.4 Etching—Etching is usually advisable to promote contrast between the metal layers, to remove traces of smeared metal, and to develop a fine line at the boundary of the coating. Some typical etchants are given in Appendix X2.

X1.5 Measurement:

X1.5.1 The measuring device may be a filar micrometer or a micrometer eyepiece. The latter has a lower precision. An image-splitting eyepiece is advantageous for thin coatings on rough substrate surfaces. Measurement of the image projected on to a ground-glass plate is usually less satisfactory because of the lack of sharpness of the image and poor legibility of the ruler when the projected image is visible.

X1.5.2 The measuring device should be calibrated at least once before and once after a measurement, unless repeated experience indicates otherwise.

X1.5.3 When making calibration and coating measurements, both should be made by the same operator, the stage micrometer and the coating should be centered in the field, and each measurement at a point should be made at least twice and averaged.

X1.5.4 For critical and referee measurements, all steps for the preparation of cross sections and measurement of coating thickness, from grinding with 600 grade or coarser abrasive, up to and including the determination, should be performed at least twice. With good techniques and equipment, and smooth coating and substrate surfaces, repeatability within 2 % or 0.5 µm, whichever is the greater, is reasonable.

X1.5.5 Some microscopes are subject to a spontaneous movement of the stage relative to the objective, possibly due to nonuniform thermal effects from the light source. Such a movement during the measurement can cause an erroneous measurement at moderate and high magnifications. This can be minimized by completing the measurement quickly and by measuring each interval twice, once from left to right and once from right to left.
X2. SOME TYPICAL ETCHANTS FOR USE AT ROOM TEMPERATURE

<table>
<thead>
<tr>
<th>Etchant</th>
<th>Use and Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitric acid (sp gr 1.42): 5 mL&lt;br&gt;Ethanol (95 %): 95 mL&lt;br&gt;&lt;strong&gt;Caution&lt;/strong&gt;—This mixture can be explosively unstable, particularly if heated.</td>
<td>For nickel or chromium coatings on steel&lt;br&gt;Etches steel&lt;br&gt;This etchant should be freshly prepared.</td>
</tr>
<tr>
<td>Iron(III)chloride hexahydrate (FeCl₃·6H₂O): 10 g&lt;br&gt;Hydrochloric acid (sp gr 1.16): 2 mL&lt;br&gt;Ethanol (95 %): 98 mL</td>
<td>For gold, lead, silver, nickel and copper coatings on steel, copper, and copper alloys.&lt;br&gt;Etches steel, copper, and copper alloys.</td>
</tr>
<tr>
<td>Nitric acid (sp gr 1.42): 50 mL&lt;br&gt;Glacial acetic acid: 50 mL</td>
<td>For determination of thickness of individual layers of multilayer coatings of nickel on steel and copper alloys; distinguishes each layer of nickel by identifying structures.&lt;br&gt;Etches nickel; excessive attack on steel and copper alloys.</td>
</tr>
<tr>
<td>Ammonium persulfate: 10 g&lt;br&gt;Ammonium hydroxide (sp gr 0.88): 2 mL&lt;br&gt;Distilled water: 90 mL</td>
<td>For tin and tin alloy coatings on copper and copper alloys.&lt;br&gt;Etches copper and copper alloys.&lt;br&gt;This etchant should be freshly prepared.</td>
</tr>
<tr>
<td>Nitric acid (sp gr 1.42): 5 mL&lt;br&gt;Hydrofluoric acid (sp gr 1.14): 2 mL&lt;br&gt;Distilled water: 93 mL</td>
<td>For nickel and copper coatings on aluminum and its alloys.&lt;br&gt;Etches aluminum and its alloys.</td>
</tr>
<tr>
<td>Chromium(VI) oxide (CrO₃): 20 g&lt;br&gt;Sodium sulfate: 1.5 g&lt;br&gt;Distilled water: 100 mL</td>
<td>For nickel and copper on zinc-based alloys. Also suitable for zinc and cadmium on steel.&lt;br&gt;Etches zinc, zinc-based alloys and cadmium.</td>
</tr>
<tr>
<td>Hydrofluoric acid (sp gr 1.14): 2 mL&lt;br&gt;Distilled water: 96 mL</td>
<td>For anodized aluminum alloys.&lt;br&gt;Etches aluminum and its alloys.</td>
</tr>
<tr>
<td>Ammonium hydroxide (sp gr 0.90): 1 part by volume&lt;br&gt;Hydrogen peroxide (3 % solution): 1 part by volume&lt;br&gt;Sodium or potassium cyanide (10 % solution): 1 part&lt;br&gt;Ammonium persulfate (10 % solution): 1 part&lt;br&gt;Make up each solution fresh each time.</td>
<td>For nickel on copper and its alloys. Swab with a fresh solution.&lt;br&gt;Etches the copper.</td>
</tr>
<tr>
<td></td>
<td>For silver and gold on copper and nickel alloys and steel.</td>
</tr>
</tbody>
</table>

X3. LIMIT OF RESOLUTION

X3.1 Resolution may be expressed as the minimum distance by which two points must be separated before they can be revealed as separate points in the image. For a microscope there is a theoretical limit of resolution determined by the numerical aperture (NA) of the objective. This theoretical limit is approached by good quality microscopes. For practical purposes better resolution cannot be obtained regardless of the quality of the optics or of the total magnification.

X3.2 Generally, the maximum useful magnification is about 1000 × the NA of an objective. That is, for practical purposes, greater magnification will not reveal additional information nor impart better definition. Such additional magnification is often referred to as “empty magnification.”
Standard Specification for Electrodeposited Coatings of Gold for Engineering Uses

This standard is issued under the fixed designation B 488; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This specification covers requirements for electrodeposited gold coatings that contain not less than 99.00 mass % gold and that are used for engineering applications.

1.2 Specifically excluded from this specification are autocatalytic, immersion, and vapor deposited gold coatings.

1.3 Gold coatings conforming to this specification are employed for their corrosion and tarnish resistance (including resistance to fretting corrosion and catalytic polymerization), bondability, low and stable contact resistance, solderability, and infrared reflectivity. Several types of coatings, differing in gold purity and hardness, are covered by this specification.

1.4 The values stated in SI units are to be regarded as the standard. Values provided in parentheses are for information only.

1.5 The following hazards caveat pertains only to the test methods section, Section 9, of this specification: This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 32 Practice for Cleaning Metals Prior to Electroplating
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 253 Guide for Preparation of Aluminum Alloys for Electroplating
B 254 Practice for Preparation of and Electroplating on Stainless Steel
B 281 Practice for Preparation of Copper and Copper Base Alloys for Electroplating and Conversion Coatings
B 322 Practice for Cleaning Metals Prior to Electroplating
B 343 Practice for Preparation of Nickel for Electroplating with Nickel
B 374 Terminology Relating to Electroplating
B 481 Practice for Preparation of Titanium and Titanium Alloys for Electroplating
B 482 Practice for Preparation of Tungsten and Tungsten Alloys for Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
B 489 Practice for Bend Test for Ductility of Electrodeposited and Autocatalytically Deposited Metal Coatings on Metals
B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
B 507 Practice for Design of Articles to Be Electroplated on Racks
B 542 Terminology Relating to Electrical Contacts and Their Use
B 558 Practice for Preparation of Nickel Alloys for Electroplating
B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
B 571 Test Methods for Adhesion of Metallic Coatings
B 578 Test Method for Microhardness of Electroplated Coatings
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 678 Test Method for Solderability of Metallic Coated Products
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 735 Test Method for Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor
B 741 Test Methods for Porosity in Gold Coatings on Metal

1 This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.08 on Engineering Coatings.


3 Annual Book of ASTM Standards, Vol 02.05.

4 Annual Book of ASTM Standards, Vol 03.04.
Substrates by Paper Electrography\textsuperscript{3}
B 748 Test Method for Measurement of Thickness of Metallic Coatings by Measurement of Cross Section with a Scanning Electron Microscope (SEM)\textsuperscript{3}
B 762 Method of Variables Sampling of Metallic and Inorganic Coatings\textsuperscript{3}
B 765 Guide for Selection of Porosity Tests for Electrodeposits and Related Metallic Coatings\textsuperscript{3}
B 799 Test Method for Porosity in Gold and Palladium Coatings by Sulfuric Acid/Sulfur-Dioxide Vapor\textsuperscript{4}
B 809 Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor (Flowers-of-Sulfur)\textsuperscript{3}
D 509 Method of Sampling and Grading Rosin\textsuperscript{5}
D 1125 Test Methods for Electrical Conductivity and Resistivity of Water\textsuperscript{6}
D 3951 Practice for Commercial Packaging\textsuperscript{7}
F 390 Test Method for Sheet Resistance of Thin Metallic Films with a Collinear Four-Probe Array\textsuperscript{8}
2.2 U.S. Government Standards:
MIL-G-45204 Gold Plating, Electrodeposited\textsuperscript{9}
MIL-STD-1916 DOD Preferred Methods for Acceptance of Product\textsuperscript{9}
2.3 ANSI/ASQC Standards
ANSI/ASQC Z1.4-1993 Sampling Procedures and Tables for Inspection by Attributes\textsuperscript{10}

3. Terminology

3.1 Definitions—For definitions of terms used in this specification refer to Terminologies B 374 or B 542.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 significant surfaces—defined as those normally visible (directly or by reflection) or essential to the serviceability or function of the article. Can be the source of corrosion products or tarnish films that interfere with the function or desirable appearance of the article. The significant surfaces shall be indicated on the drawings of the parts or by the provision of suitably marked samples.

3.2.2 underplating—a metallic coating layer between the basis metal or substrate and the topmost metallic coating. The thickness of an underplating is usually greater than 1 µm (40 µin.), in contrast to a strike or flash, which is thinner.

4. Classification

4.1 Types of Coatings—A coating shall be specified by a combination of the following:

4.1.1 Type, characterizing minimum purity in accordance with 4.2.1.

4.1.2 Code, designating Knoop hardness in accordance with 4.2.3, and

4.1.3 a numeral designating thickness in micrometres in accordance with 4.3.

4.2 Purity and Hardness:

4.2.1 Purity—The issue of this standard establishes a new ASTM Type designation, which is identical to the original standard (MIL-G-45204), established for electrodedeposited gold and is outlined in the following table.

<table>
<thead>
<tr>
<th>Mass Percent Gold, Minimum, Excluding Potassium, Carbon &amp; Nitrogen</th>
<th>New ASTM Type</th>
<th>MIL-G45204 Type</th>
<th>Old ASTM Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>99.70</td>
<td>I</td>
<td>I</td>
<td>2</td>
</tr>
<tr>
<td>99.00</td>
<td>II</td>
<td>II</td>
<td>3</td>
</tr>
<tr>
<td>99.90</td>
<td>III</td>
<td>III</td>
<td>1</td>
</tr>
</tbody>
</table>

Note: 1—It is commonly accepted that a gold purity of 99.7 % is defined as no more than 0.3 % of total codeposited metallic components, excluding potassium and sodium. Likewise, 99.9 % purity is recognized to mean no more than 0.1 % of total codeposited metal impurities, excluding potassium and sodium. Almost all gold electrodeposits will contain potassium, carbon and nitrogen that are occluded or precipitated in the deposit. In the case of Type I gold, the occluded potassium has been shown to improve durability and is desirable for that reason.\textsuperscript{11,12}

4.2.2 Gold purity is calculated by subtraction of the sum of all non-gold metals in mass %, excluding potassium and sodium, from 100 %. The presence of C, N, H, Na, and K contained in the electrodeposite are not included in the calculation.

4.2.3 Hardness—Hardness values shall be specified by ASTM code. The military standard designation of grade is included for reference.

<table>
<thead>
<tr>
<th>Knoop Hardness Range</th>
<th>ASTM Code</th>
<th>MIL-G45204 Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>90 HK\textsubscript{C} (see Note 2)</td>
<td>A</td>
<td>A</td>
</tr>
<tr>
<td>91–129 HK\textsubscript{C}</td>
<td>B</td>
<td>B</td>
</tr>
<tr>
<td>130–200 HK\textsubscript{C}</td>
<td>C</td>
<td>C</td>
</tr>
<tr>
<td>&gt;200 HK\textsubscript{C}</td>
<td>D</td>
<td>D</td>
</tr>
</tbody>
</table>

Note: 2—ASTM Code B has previously been specified for hardness grade 90–200. This hardness grade has been eliminated and replaced with hardness grade 91–129 in accordance with MIL-G-45204.

4.2.4 Relationship Between Purity and Hardness—The following combinations of purity and hardness ranges are representative of good commercial practice:

<table>
<thead>
<tr>
<th>New ASTM Type</th>
<th>Old ASTM Type</th>
<th>Code</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>2</td>
<td>A, B and C</td>
</tr>
<tr>
<td>II</td>
<td>3</td>
<td>B, C and D</td>
</tr>
<tr>
<td>III</td>
<td>1</td>
<td>A only</td>
</tr>
</tbody>
</table>

4.3 Thickness—Thickness shall be specified by an Arabic numeral that designates the minimum linear thickness in micrometres. Examples of commonly specified thicknesses are shown in Table 1.

4.3.1 See 7.4.2 for thickness tolerances.

4.3.2 Instead of specifying the thickness in micrometres, the purchaser may specify the mass of gold per unit area (coating weight) in milligrams per square centimeter. Unless otherwise specified, the density of gold is assumed to be 19.3 g/cm\textsuperscript{3} for Type III and 17.5 g/cm\textsuperscript{3} for Type I and Type II.

Note: 3—The density of Type III gold coatings will be less than or equal to 19.3 g/cm\textsuperscript{3}, but not less than 18.5 g/cm\textsuperscript{3}.

Note: 4—When significant surfaces are involved on which the specified thickness of deposit cannot readily be controlled, such as threads, holes,

\textsuperscript{3} Annual Book of ASTM Standards, Vol 06.03.
\textsuperscript{4} Annual Book of ASTM Standards, Vol 11.01.
\textsuperscript{5} Annual Book of ASTM Standards, Vol 15.09.
\textsuperscript{6} Annual Book of ASTM Standards, Vol 10.04.
\textsuperscript{7} Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.
\textsuperscript{8} Available from the American National Standards Institute, 1819 L Street NW, Washington, DC 20036.
TABLE 1 Coating Thickness

<table>
<thead>
<tr>
<th>Class</th>
<th>Minimum Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>0.50</td>
<td>0.50</td>
</tr>
<tr>
<td>0.75</td>
<td>0.75</td>
</tr>
<tr>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>1.25</td>
<td>1.25</td>
</tr>
<tr>
<td>2.5</td>
<td>2.5</td>
</tr>
<tr>
<td>5.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>

6.1 Any process that provides an electrodeposition capable of meeting the specified requirements is acceptable. However, other processes shall be specified and should meet this specification prior to the plating of the parts.

6.2 Substrate:

6.2.1 The surface condition of the basis metal should be specified and should meet this specification prior to the plating of the parts.

6.2.2 Defects in the surface of the basis metal such as scratches, porosity, pits, inclusions, roll and die marks, laps, cracks, burrs, cold shuts, and roughness may adversely affect the appearance and performance of the deposit, despite the observance of the best plating practice. Any such defects on significant surfaces shall be brought to the attention of the purchaser.

6.2.3 The basis metal shall be subject to such cleaning procedures as are necessary to ensure a satisfactory surface for subsequent electroplating (see Practices B 253, B 254, Guide B 253, Practices B 254, B 281, B 322, B 343, B 481, B 482, and B 558).

6.2.4 Proper preparatory procedures and thorough cleaning of the basis metal are essential for satisfactory adhesion and performance of these coatings. The surface must be chemically clean and continuously conductive, that is, without inclusions or other contaminants. They must be smooth and as free of scratches, gouges, nicks, and similar imperfections as possible.

NOTE 5—A metal finisher can often remove defects through special treatments such as grinding, polishing, abrasive blasting, chemical treatments, and electropolishing. However, these may not be normal in the treatment steps preceding the plating and a special agreement is indicated.

6.3 If required (see 5.1.7), steel parts with a hardness greater than 31 HRC shall be given a suitable stress relief heat treatment prior to plating. Such stress relief shall not reduce the hardness to a value below the specified minimum. Acid pickling of high strength steels shall be avoided.

6.3.1 The coating shall be applied after all basis metal heat treatments and mechanical operations on significant surfaces have been completed.

6.4 Racking—Parts should be positioned so as to allow free circulation of solution over all surfaces. The location of rack or wire marks in the coating shall be agreed upon between the producer and the supplier.

6.5 Plating Process:

6.5.1 Nickel Underplating—For thickness classes except 5.0, a nickel underplating shall be applied before the gold coating when the product is made from copper or copper alloy. Nickel underplatings are also applied for other reasons (see Appendix X6).

NOTE 6—When the thickness of the nickel underplate has a detrimental impact on the mechanical properties or bondability of the substrate, the nickel thickness may be reduced to a non-detrimental level as specified by the purchaser.

NOTE 7—In certain instances in which high-frequency analog signals are employed, such as in wave guides, the magnetic properties of nickel may attenuate the signal.

NOTE 8—In applications in which forming or flaring operations are to be applied to the plated component, a ductile nickel electrodeposition should be specified.

6.5.2 Strikes—It is recommended to apply a gold strike to the underplate or substrate, except if the latter is silver or platinum, prior to applying the gold top coating.

6.5.3 Plating—Good practice calls for parts to be electrically connected when entering the gold plating solution. A minimum of 0.5 V is suggested.

NOTE 9—For rack and barrel plating processing, residual plating salts can be removed from the articles by a clean, hot (50 to 100°C) water rinse. A minimum rinse time of 2.5 min (rack) or 5 min (barrel) is suggested. Best practice calls for a minimum of 3 dragout rinses and one running rinse with dwell times of 40 s in each station when rack plating and 80 s when barrel plating. Modern, high-velocity impingement-type rinses can reduce this time to a few seconds. This is particularly useful in automatic reel-to-reel applications in which dwell times are significantly reduced.

7. Coating Requirements

7.1 Nature of Coating—The gold deposit shall meet the appropriate purity requirements as put forth in the following:

7.1.1 Type I coatings shall contain at least 99.70 mass % gold, excluding potassium, sodium, carbon and nitrogen. For Type I Code A (“soft”) coatings only, metallic hardening agents such as nickel, cobalt or iron combined, shall be less than 0.05 mass % and none of these three elements shall be present in an amount greater than 0.03 mass %. All other metals excluding potassium and sodium are considered impurities and shall not be present in amounts greater than 0.05 mass % combined. For Type I Code B nd C coatings, metallic hardening agents such as nickel, cobalt or iron combined shall be equal to or less than

7.1.2 Type II coatings are employed, such as in wave guides, the magnetic properties of nickel may attenuate the signal.

7.1.3 Type III coatings shall contain at least 99.99 mass % gold, excluding potassium, sodium, carbon, and nitrogen. For Type I Code A (“soft”) coatings only, metallic hardening agents such as nickel, cobalt or iron combined, shall be less than 0.05 mass % and none of these three elements shall be present in an amount greater than 0.03 mass %. All other metals excluding potassium and sodium are considered impurities and shall not be present in amounts greater than 0.05 mass % combined. For Type I Code B nd C coatings, metallic hardening agents such as nickel, cobalt or iron combined shall be equal to or less than...
0.3 mass %. All other metals are considered impurities and shall not be present in amounts greater than 0.05 % mass % each, 0.1 % combined. The gold purity shall be calculated by determining the content of all non-gold metals in mass % (except potassium and sodium) and subtracting this value from 100 %.

7.1.2 Type II coatings shall contain at least 99.00 mass % gold, excluding potassium, sodium, carbon and nitrogen. Type II coatings may contain metallic hardening agents, such as nickel, cobalt, iron, which may be present in amounts greater than 0.3 mass %. All other metals, excluding potassium and nitrogen, shall be considered impurities and shall not be present in amounts greater than 0.1 mass % each. The gold purity shall be calculated by determining the content of all non-gold metals in mass % (except potassium and sodium) and subtracting this value from 100 %.

7.1.3 Type III coatings shall contain at least 99.90 mass % gold, excluding potassium, sodium, carbon and nitrogen. Individual metal impurities shall not be present in a quantity greater than 0.04 mass %. Iron, nickel and cobalt combined shall be less than 0.05 mass %, and none of these three elements shall be present in an amount greater than 0.03 mass %. All non-gold metals, excluding potassium and sodium, are considered impurities and shall not be present in amounts greater than 0.1 mass % combined. The gold purity shall be calculated by determining the content of all non-gold metals in mass % (except potassium and sodium) and subtracting this value from 100 %.

7.2 Hardness—The gold coating shall have a Knoop hardness within the specified range as shown in 4.2.3 when tested in accordance with 9.2.

7.3 Appearance—Gold coatings shall be coherent, continuous, and have a uniform appearance to the extent that the nature of the basis metal and good commercial practices permit.

7.4 Thickness:

7.4.1 The gold coating shall have a thickness on the significant surfaces in accordance with 4.3 unless otherwise specified.

Note 10—Where Type I (Code B or C) or Type II (Code B or C) gold is specified, improved protection of the underlying metals can be obtained by applying a duplex gold composite coating consisting of an underlying layer of Type III (Code A) gold. Typical thicknesses of the Type III gold underlayer are 10 to 30 % of the total specified thickness. However, care must be taken to ensure that the thickness of the Type I or Type II gold topcoat is sufficient so as not to compromise wear properties. When measuring the total gold thickness of duplex composite coatings, a density of 17.5 g/cm³ should be employed (see 9.4).

7.4.2 The thickness of the gold coating shall be equal to or exceed the specified thickness throughout the significant surface (see 3.2.1). When plated parts are used in mating applications, the maximum thickness shall not exceed the tolerance specified for the part on its engineering drawing. It is customary that allowance for plated coatings be included within the part tolerance.

Note 11—The coating thickness requirement of this specification is a minimum requirement: that is, the coating thickness is required to equal or exceed the specified thickness throughout the significant surfaces, while conforming to all maximum allowed thicknesses created by part dimensional tolerances given in the engineering drawing. Variation in the coating thickness from point to point on a coated article is an inherent characteristic of electroplating processes. Therefore, the coating thickness at any single point on the significant surface will sometimes have to exceed the specified value in order to ensure that the thickness equals or exceeds the specified value at all points. Hence, most average coating thicknesses will be greater than the specified value; how much greater is largely determined by the shape of the article (see Practice B 507) and the characteristics of the plating process. In addition, the average coating thickness on articles will vary from article to article within a production lot. Therefore, if all the articles in a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the average necessary to ensure that any single article meets the requirement (see 8.1).

7.5 Adhesion—The gold coatings shall be adherent to the substrate when tested by one of the procedures summarized in 9.5.

7.6 Integrity of the Coating:

7.6.1 Gross Defects/Mechanical Damage—The coatings shall be free of visible mechanical damage and similar gross defects when viewed at magnifications up to 10×. For some applications this requirement may be relaxed to allow for a small number of such defects (per unit area), especially if they are outside or on the periphery of the significant surfaces (see 7.6.2).

7.6.2 Porosity—Almost all as-plated electrodeposits contain some porosity. The tolerable amount of porosity in the coating depends on the severity of the environment that the article is likely to encounter during service or storage. If the pores are few in number or away from the significant surfaces, their presence can often be tolerated. Such acceptance (or pass-fail) criteria, if required, shall be part of the product specification for the particular article or coating requiring the porosity test (see 9.6 for porosity testing).

8. Sampling

8.1 The producer is urged to employ statistical process control in the coating process. Properly performed, this process will ensure coated products of satisfactory quality and will reduce the amount of acceptance inspection. The sampling plan used for the inspection of the quality of the coated articles shall be as agreed upon between the purchaser and the supplier.

Note 12—Usually, with a collection of coated articles, the inspection lot (see 8.2) is examined for compliance with the requirements placed on the articles, a relatively small number of the articles, the sample, is selected at random and is inspected. The inspection lot is then classified as complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for the sampling inspection of coatings. Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one with those that are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 identifies the plan to be used. Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and seller need to agree on the plan to be used. Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numerical value and certain statistical requirements must be met. Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Method B 762...
identifies the plan to be used.

8.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind; that have been produced to the same specifications; that have been coated by a single supplier at one time, or at approximately the same time; under essentially identical conditions; and that are submitted for acceptance or rejection as a group.

9. Test Methods

9.1 Deposit Purity—Use any recognized method to determine quantitatively the metallic impurities present. Atomic absorption spectrophotometry (or any methods with demonstrated uncertainty less than 10 %) may be used to determine the metallic impurities. Initial scanning should be carried out for all elements in order to detect any unknown or unexpected metallic impurities. Determine deposit purity by subtracting total impurities in mass from 100 %. Deposit purity should be determined on samples at plating conditions representative of actual production.

9.2 Hardness—Measure hardness in accordance with Test Method B 578. Deposit hardness should be determined on samples at plating conditions representative of actual production.

9.3 Appearance—The coating shall be examined at magnifications up to 10× for conformance to the requirements of appearance.

9.4 Thickness and Mass per Unit Area—Measure thickness by methods outlined in Test Methods B 487, B 567, B 568, or B 748. The method chosen shall be such as to give an uncertainty of less than 10 % on the particular coating being measured.

9.4.1 Thickness—The beta backscatter, X-ray fluorescence spectrometry, and coulometric methods measure the mass of gold per unit area that is present over the measured area. The cross-sectioning and magnetic methods measure the geometrical thickness of the gold present. Since many gold coatings have a density lower than the theoretical density of gold (19.3 g/cm³), instruments utilizing the measuring principles in Test Methods B 504, B 567, and B 568 will often give lower readings than the microscopical cross-section method. To convert mass of gold per unit area that is present over the measured area to a geometrical thickness, it is necessary to use standards having a density identical to that of the coatings to be measured, or to multiply the reading obtained by factor, \( f \).

\[
f = \frac{19.3}{d} \quad (1)
\]

where:
\( d \) = density of gold coating, g/cm³.

9.4.1.1 The density of gold coatings depends not only on the type of gold coating but also on the gold content and the composition and purity of the electroplating solution. The density figure, when the above methods are used for measurements of thickness, shall be considered to be 19.3 g/cm³ for Type III coatings and 17.5 g/cm³ for Type I and Type II coatings, unless:

(1) the density is supplied by the plating process vendor, or

(2) the density has been measured.

9.4.2 Mass Per Unit Area—Test Methods B 504, B 567, and B 568 measure the mass of gold per unit area (coating weight) directly and independently of the density of gold.

Note: 13—The calibration standards available from the National Institute of Standards and Technology (NIST)¹³ are certified in units of mass of gold per unit area. Other calibration standards for gold are generally certified in thickness units that can be converted to mass per unit area units by multiplying by the density of pure gold.

\[
\text{Mass per unit area (coating weight)} = \frac{19.3 \times \text{thickness (\( \mu \text{m} \))}}{10} \quad (2)
\]

This conversion should be verified by the supplier of the calibration standards.

9.5 Adhesion—Determine adhesion by one of the following procedures (see Test Methods B 571 for full details).

9.5.1 Bend Test—Bend the electroplated article repeatedly through an angle of 180° on a diameter equal to the thickness of the article until fracture of the basis metal occurs. Examine the fracture at a magnification of 10×. Cracking without separation does not indicate poor adhesion unless the coating can be peeled back with a sharp instrument.

9.5.2 Heat Test—No flaking, blistering, or peeling shall be apparent at a magnification of 10× after the gold electroplated parts are heated to 300 to 350°C (570 to 660°F) for 30 min and allowed to cool.

9.5.3 Cutting Test—Make a cut with a sharp instrument and then probe with a sharp point and examine at a magnification of 10×. No separation of the coating from the substrate shall occur.

9.6 Plating Integrity—Porosity shall be determined preferably by either Test Method B 735, B 741, B 799, or B 809 unless otherwise specified. See Guide B 765 for guidelines for selecting the appropriate test method.

Note: 14—The test to be selected will depend upon the gold thickness, the nature of the basis metal, the nature and thickness of any intermediate layers or underplate, the shape of the gold-coated part, and its intended service environment.

9.7 Ductility—When required, determine ductility in accordance with Practice B 489.

10. Special Government Requirements

10.1 The following special requirements shall apply when the ultimate purchaser is the U.S. Government or an agent of the U.S. Government.

10.1.1 Sampling—For government acceptance, the sampling plans specified in MIL-STD-1916 or ANSI/ASQC Z1.4–1993 are to be used instead of the ASTM standards specified in 8.1.

10.1.2 Thickness Testing:

10.1.2.1 In addition to the nondestructive method outlined in Test Methods B 499, B 567, and B 568, a cross-sectioning method, such as that specified by Test Method B 487 or B 748, can also be used as a referee method to confirm the precision and bias of the particular non-destructive technique when thicknesses of 0.75 µm or above are used.

¹³ Available from the National Institute of Standards and Technology, (NIST), Gaithersburg, MD 20899.
10.1.2.2 The part shall be covered with gold. The gold thickness on significant surfaces shall be at least 1.25 µm (50 µin.), unless otherwise specified in the drawings or contract. Any specific requirement for thicknesses on non-significant surfaces shall be agreed on between purchaser and supplier.

10.1.3 Packaging—Parts plated for the U.S. Government and Military, including as subcontracts, shall be packaged in accordance with Practice D 3951.

11. Keywords

11.1 engineering coatings; engineering gold; gold; gold coatings; gold electrodeposits; gold platings; underplate

APPENDIXES

(Nonmandatory Information)

X1. TYPICAL USES

X1.1 The conditions of exposure and the uses of gold coatings are so varied that it is often not possible to predict the life of a coating of a given thickness and hardness. Some typical uses of gold coatings are enumerated below, but the list is not complete and is intended for general guidance only.

X1.1.1 Type 1, Code A Gold—For semiconductor components, nuclear engineering, thermocompression bonding, thermostatic bonding, ultrasonic bonding, solderability, and high-temperature applications.

X1.1.2 Type 2—For general-purpose, high-reliability electrical contacts, and wire wrap connections.

X1.1.3 Type 3, General-Purpose Wear Resistant Gold—Will not withstand high-temperature applications because the hardening agents in the gold coating will oxidize.

X1.1.4 Solderability—Type 1 golds are preferred for solder applications. Oxidation of co-deposited metals can make soldering more difficult. It is strongly recommended that gold coating thicknesses not exceed 0.5 µm for solder applications.

There is evidence to show that when the gold content in the tin-lead solder joint exceeds about 3 % by weight, embrittlement occurs leading to potential solder joint failure.14

X1.1.5 Printed Wiring Boards—Boards that will be sheared or cropped should not be electroplated with a heavy thickness of a hard gold because of possible cracking of the gold. Coatings with purities of 99.0 to 99.7 % are used with B or C hardness and at thicknesses generally not in excess of 2.5 µm.

X1.1.6 Static Separable Connectors—Coatings at any of the listed purities and with hardnesses A, B, or C are used to any thickness up to 5 µm. Selection of the type and thickness of the gold coating is determined by the desired life in the operating environment of the electroplated component.

X2. RESISTIVITY

X2.1 When electrodeposited gold coatings are used as electrical conductor paths, the electroplating process can be expected to produce coatings having maximum resistivities as specified in Table X2.1.

X2.2 Use any reliable four-point probe method for determining resistivity of gold coatings. A suggested four-point method is detailed in Test Method F 390.

<table>
<thead>
<tr>
<th>Gold Finishes</th>
<th>Ohm per Square</th>
</tr>
</thead>
<tbody>
<tr>
<td>A and B</td>
<td>0.03</td>
</tr>
<tr>
<td>C</td>
<td>0.10</td>
</tr>
</tbody>
</table>

A When the electroplated coatings are used as electrical conductor paths, the sheet resistance in ohms per square should be specified and not the coating thickness.

X3. RESIDUAL SALTS

X3.1 Electroplated parts are placed in water of known conductivity and agitated for a specific time. The conductivity of the water extract is measured, and the increase in conductivity due to residual salts and other conducting impurities is calculated. A suggested water extract conductivity test method uses a procedure in accordance with Test Methods D 1125, Method A.

X3.2 Conductivity of water for extract test shall be 1 µS/cm or less (resistivity 1 MΩ·cm or more).

X3.2.1 A sample of the coated parts having a total surface area of 30 cm² shall ordinarily be used and extracted in 100 cm³ of equilibrated water. To prepare equilibrated water, fill a clean polyethylene bottle half-way with high-purity water (X3.1), replace the bottle cap, and shake the bottle vigorously for 2 min to equilibrate the water with the CO₂ in the air. CO₂ is a component of air. It is soluble in water and forms carbonic acid that ionizes and is at equilibrium at 0.8 µS/cm. Slowly

agitate the solution for 10 min before determining the conductivity of the extract. In a closed polyethylene bottle, the equilibrated water will remain in the range from 0.8 to 1 µS/cm for at least 1 week. Place the specimen in the bottle in accordance with X3.1.

X3.3 Inspection under a source of ultraviolet light is often employed to determine whether electroplating salts have been removed by the rinsing following gold electroplating. The presence of salts is evidenced by a characteristic fluorescence and should not be confused with fluorescing dirt or dirt particles.

X3.4 Water or purging stains, resulting from blind holes or from parts that were assembled before electroplating, as normally obtained in good commercial practice, are permissible, except where they occur on surfaces to which electrical contact is to be made or on which subsequent soldering operations are performed.

X4. SOLDERABILITY

X4.1 Where solderability requirements are specified, solderability testing shall be done in accordance with Test Method B 678.

X5. MANUFACTURE

X5.1 Substrate Preparation:

X5.1.1 Practices B 183, B 242, B 253, B 254, B 281, B 322, B 343, B 481, and B 482 should be used where appropriate for the preparation of the applicable basis metal.

X5.1.2 When electroplated materials are used for electrical contacts, cleaning alone may not properly prepare the surface for gold electroplating. If the basis metal is scratched or rough, it may be necessary to level the surface by processes such as electropolishing, chemical polishing, or the application of a self-leveling nickel or copper undercoating before gold electroplating. However, the use of these undercoatings requires the approval of the purchaser.

X5.2 Underplatings:

X5.2.1 Silver shall not be used as an underplating unless specified by the purchaser.

X5.2.2 Copper alloys containing zinc, beryllium, tellurium, or lead should be precoated to prevent adhesion problems. If copper or nickel underplatings are used, they should have a nominal thickness of at least 1.2 µm. Thickness of the underplating should be consistent with end-use requirements. With cyanide-sensitive copper alloys, such as those containing beryllium or tellurium, an acid copper strike is recommended.

X5.2.3 Gold coatings on zinc, aluminum, steel, and other metals normally require underplatings such as copper or nickel of sufficient thickness to impact adequate corrosion protection to the basis metal.

X5.2.4 To minimize diffusion, a nickel underplating with a nominal thickness of at least 1.2 µm should be applied before depositing any of these gold finishes on copper and copper alloy parts that can be subjected to elevated temperatures. The thickness of the underplating should be consistent with end-use requirements (see 6.6.1).

X5.2.5 A gold strike is often used to precede the final gold coating, to improve adhesion, and to prevent contamination of the main gold electroplating solution with metallic impurities.

X5.3 Gold Electrolytes—Most gold electroplating solutions used for producing these coatings are proprietary. Any bath that produces coatings meeting the requirements of the specification may be used.

X5.3.1 Solution Contamination—Care should be exercised to prevent contamination of gold electroplating solutions with organic or metallic impurities. Copper, zinc, cadmium (from racking materials, bus bars, etc.), iron (from insoluble anodes), lead and tin (from solder parts), and silver (from impurities in the electroplating bath makeup salts) are some of the more common contaminants, which, if present in the coating in more than trace quantities, can have harmful effects on such properties as hardness, solderability, and porosity.

X5.3.2 Activated Carbon Treatment—Periodic treatment of the solution with activated carbon is recommended to control the buildup of organic impurities. This contamination can have the effect of producing discoloration of the deposit. Thus, the appearance of test coupons may serve as an indication of an approaching need for the activated carbon treatment. (Warning—Do not store activated carbon in areas in which vapors from solvents and other chemicals may contaminate the carbon.)

X5.3.3 Control of the Gold Electroplating Bath—Under continuous operations, when control of bath composition can be readily established by scheduled additions, analyses of the electroplating bath constituents should be made on a regularly scheduled basis. When the work load is erratic, each addition should be based on bath analyses.

X5.3.4 Control of the Gold Deposit—The adhesion, hardness, and composition of the gold deposit should be determined on a regularly scheduled basis. Once it has been established that the co-deposited metallic impurity limitations are being continuously met in production, frequency of determination can be reduced. Appearance, porosity, and thickness should be determined often enough to ensure compliance with specific requirements.
X6. SOME REASONS FOR USING A NICKEL UNDERPLATE

X6.1 Diffusion Barrier:
X6.1.1 To inhibit diffusion of copper from the basis metal (and of zinc from brass) to the surface of the precious metal plating.
X6.1.2 To inhibit interdiffusion between the basis metal and the gold top coat (for example, silver and copper), which might produce a weak alloy or intermetallic compound at the interface.

X6.2 Levelling Layer—To produce a smoother surface than the basis metal in order to ensure a lower porosity gold top plate (for example, levelling nickel over a rough substrate).

X6.3 Pore Corrosion Inhibitor—A nickel underplate under the gold top coat will form passive oxides at the base of pores in humid air, provided the environment does not contain significant amounts of acidic pollutants (such as SO₂ or HCl).

X6.4 Tarnish Creepage Inhibitor for Gold—Non-copper base metals will inhibit creepage of copper tarnish films over the gold—where the tarnish originates from pores and bare copper edges.

X6.5 Load-Bearing Underlayer for Contacting Surfaces—A hard nickel underplate can serve as a load-bearing foundation for the gold top coat to prevent cracking of hard golds and reduce the wear of the precious metal during sliding of the contacting surfaces.

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Standard Practice for
Bend Test for Ductility of Electrodeposited and
Autocatalytically Deposited Metal Coatings on Metals\(^1\)

This standard is issued under the fixed designation B 489; the number immediately following the designation indicates the year of
original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A
superscript epsilon (\(\epsilon\)) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a test procedure for determining the
ductility of electrodeposited and autocatalytically deposited
coatings on sheet or strip basis metals. The purpose of the test
is to determine the resistance of metal coatings to cracking
during distortion.\(^2\)

1.2 Test Methods E 8 can be used if the coatings are too
ductile and require mandrels too small to be practical.

1.3 This standard does not purport to address all of the
safety concerns, if any, associated with its use. It is the
responsibility of the user of this standard to establish appro-
priate safety and health practices and determine the applica-
bility of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards: \(^3\)
B 177 Guide for Engineering Chromium Electroplating
D 1193 Specification for Reagent Water
E 8 Test Methods for Tension Testing of Metallic Materials

3. Summary of Practice

3.1 The practice consists of bending a narrow strip of the
electroplated or coated article over a mandrel. An elongation
measurement is obtained from the smallest diameter mandrel
that does not cause the coating to fracture.

4. Significance and Use

4.1 The routine measurement of the ductility of electrode-
posited and autocatalytically deposited metal coatings can be
useful in process control, especially when the electroplating
process is used for decorative and engineering purposes.

5. Apparatus

5.1 Series of Mandrels, with diameters from 6 to 50 mm, in
3-mm steps with lengths of 100 to 150 mm so they can be held
in a vise.

5.2 Micrometer, to measure the thickness of the test speci-
mens.

5.3 Guillotine Shears or other device to cut the specimens to
size.

5.4 File or Grinder to remove burrs and to round or chamfer
edges.

5.5 Vise, to hold mandrels.

5.6 Magnifier, 10\(^\times\).

6. Test Specimen

6.1 Flat specimens, 10 mm wide, and not less than 150 mm
long, shall be cut from the electroplated or coated article if the
shape permits, no closer than 25 mm from the edges. Guillotine
sheets are preferred, but any convenient method may be used.
Basis metal thickness and temper shall be suitable to permit
bending around the smallest diameter mandrel, if necessary.
Low-carbon AISI 1010 to 1025 steel strip or sheet, 0.25 to 1.0
mm thick is usually suitable. Basis metals that have low
ductility can initiate cracks that can propagate through the
coatings. The procedure indicated in 6.2 shall then be followed.

6.2 When the shape is such that a test specimen cannot be
obtained from the part, a test panel may be prepared of
appropriate basis metal, such as low-carbon steel (see 6.1),
with the same coating system in the same baths. The panel shall
be sufficiently large to obtain several pieces after trimming 25
mm from the edges. The specimens shall be prepared in
accordance with 6.1. Brass or copper panels may be used
instead of copper-electroplated zinc alloy panels.

6.3 The long edges of the test pieces shall be rounded or
chamfered by filing or grinding.

7. Procedure

7.1 Place the largest mandrel in the vise. Bend the test
specimen, with the coating outward, over the mandrel so that as
the bend progresses the test specimen will remain in contact
with the top of the mandrel. Continue bending with slow,
steadily applied pressure until the two legs are parallel. If there
are no cracks visible under a 10× magnifier, repeat the test, using new specimens, on progressively smaller-diameter mandrels, until cracks appear across or through the coating. Take the preceding mandrel diameter as the value for the ductility determination. If the coating is electrodeposited chromium, the specimens may require heating or aging to overcome temporary hydrogen embrittlement. A procedure to overcome hydrogen embrittlement is covered in Guide B 177.

7.1 Small cracks not greater than 1.5 mm long, confined to the edges of the test specimen do not signify failure.

7.1.1 At times, no single crack may develop over the convex surface. If jagged cracks, or a series of shorter cracks develop (excluding edges), they signify failure.

7.2 In multiple coatings, cracking may occur in the outer coatings only. In the case of nickel, cracks may extend through the nickel to an intermediate copper layer or to the basis metal. Methods for determining this are provided in the Appendix. A positive test for copper or iron signifies failure.

7.3 Except for very ductile coatings, the apparent ductility is an inverse function of the thickness. If the test is to be used to evaluate the electroplating or autocatalytic process by periodically testing the ductility of coatings produced by the process, all specimens used must have approximately the same coating and total thickness.

8. Calculation

8.1 Determine the elongation as follows:

\[ E = 100 \frac{T}{D + T} \]

where:

- \( E \) = percent elongation,
- \( T \) = total thickness of the basis metal and deposit, and
- \( D \) = diameter of the mandrel.

Note 1—To calculate percent elongation, \( E \), the dimensions of \( T \) and \( D \) must be identical.

9. Precision and Bias

9.1 This practice is a useful one for routine control of the ductility of metallic coatings. The largest source of error is in the detection of crack initiation. The precision and bias for this practice have not been statistically determined.

10. Keywords

10.1 autocatalytic deposits; ductility tests; electrodeposits

APPENDIX

(Nonmandatory Information)

X1. COPPER AND IRON DETECTION—SPOT TEST TECHNIQUE

X1.1 Apparatus

X1.1.1 Spot Test Plate.
X1.1.2 Dropping Pipets (Medicine Droppers).
X1.1.3 Wash Bottle.
X1.1.4 Glass Stirring Rod.

X1.2 Reagents

X1.2.1 The solutions shall be made with water conforming to Specification D 1193, Type IV.
X1.2.2 Acetic Acid Mixture—Prepare the mixture by adding to 45 mL of glacial acetic acid 5 mL of wetting agent, such as 1 % solution of sodium lauryl sulfate.
X1.2.3 Hydrogen Peroxide—Use 30 volume % solution.
X1.2.4 Zinc Acetate Solution—Prepare the zinc acetate solution by using 1 mass % of ASC reagent grade material.
X1.2.5 Ammonium Mercuric Thiocyanate Solution—Add 8 g of ASC reagent grade mercuric chloride and 9 g of ASC reagent grade ammonium thiocyanate to 100 mL of water.
X1.2.6 Nitric Acid (1+4)—Add 1 part of concentrated nitric acid (HNO₃, sp gr 1.42) to 4 parts of water by volume.
X1.2.7 Potassium Thiocyanate Solution—Use a 10 mass % solution.

X1.3 Copper Detection in the Presence of Nickel and Chromium

X1.3.1 Using a dropping pipet, apply 1 drop of the acetic acid mixture and 1 drop of hydrogen peroxide to the bent and cracked surface. Permit the solution to remain for about 1 to 2 min, avoiding its contact with the cut edges.
X1.3.2 Transfer the drop of reagent from the test part with a pipet to a cavity in the spot test plate. Wash the test spot with a drop of water, and add the wash solution to the liquid in the spot plate.
X1.3.3 Add 1 drop of zinc acetate solution and 1 drop of ammonium mercuric thiocyanate solution to the solution in the spot plate cavity.
X1.3.4 Gently agitate the spot plate or stir the solution. A violet colored precipitate indicates the presence of copper.

X1.4 Iron Detection in the Presence of Chromium, Nickel, or Copper

X1.4.1 Apply 1 drop of HNO₃ (1+4) to the bent surface to be tested.
X1.4.2 Allow the acid to remain in contact with the surface for about 1 min.
X1.4.3 Transfer the drop of HNO₃ with a dropping pipet to a cavity in the spot plate. At no time should the acid come in contact with the cut edges.
X1.4.4 Add 1 drop of potassium thiocyanate solution to the solution in the spot plate cavity.
X1.4.5 Gently agitate the spot or stir the solution. The appearance of a blood red color indicates iron.
Standard Practice for Micrometer Bend Test for Ductility of Electrodeposits

1. Scope

1.1 This practice describes a procedure for measuring the ductility of electrodeposited foils.2

1.2 This practice is suitable only for the evaluation of electrodeposits having low ductility.

1.3 The obtained ductility values must only be considered semi-quantitative because this test has a significant operator dependence.

1.4 This practice is best used for in-house process control where measurements are always made by the same operator. A change in ductility value can be used as an indication of possible changes in the electroplating solution.

1.5 This standard does not purport to address the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

3 B 177 Guide for Engineering Chromium Electroplating

3. Summary of Practice

3.1 This practice consists of measuring the bend of a foil held between the jaws of a micrometer; these are closed until fracture or cracks appear.

4. Significance and Use

4.1 This practice is useful as one method of controlling some electroplating solutions. It serves to indicate the presence of contamination or some other adverse condition.

4.2 Ductility measurements are of particular value when electroplated parts are to be subjected to moderate stress such as that involved in bolting an electroplated bumper to an automobile.

Note 1—The foils used in this practice are typically 25 to 40 µm thick. Foils in this thickness range do not have the same properties as bulk metal. For example, a nickel electrodeposit 0.5 mm thick, prepared in purified bright nickel electroplating solutions for which this test is being used, had less than 3 % elongation in a tension test, and could not be bent to a 90° angle without complete fracture. However, foils 25 to 40 µm thick, electroplated at the same time, had micrometer ductility values in the 10 to 25 % range.

5. Apparatus

5.1 Micrometer, 25-mm with flat jaws to measure the thickness and to compress the foil.

5.2 Hand or Power Shear, grinding wheel, or hack saw, to trim the edges of the electroplated panel and to separate the foil from the basis metal.

5.3 Pair of Sharp Scissors to cut the test specimens.

6. Test Specimens

6.1 An electrodeposit shall be prepared using a basis metal with a smooth surface from which the electrodeposit can be readily separated. A stainless steel or nickel electroplated steel panel may be used for this purpose, prepared as in 6.2.

6.2 A piece of cold-rolled steel, of any convenient size, such as 100 by 150 mm, shall be properly cleaned, acid dipped, and electroplated with approximately 7.5 µm of nickel. After rinsing, the specimen shall be cleaned anodically for 15 s in a hot alkaline cleaner, rinsed, acid dipped in about 1 N sulfuric acid (about 27 mL of concentrated sulfuric acid added to about 900 mL of cold water, mixed, and diluted with cold water to 1 L), and immediately placed in the electroplating solution of the metal to be tested. An electrodeposit 25 to 40 µm thick shall be electroplated on the prepared surface. The deposit shall be plated at an average current density and under conditions (agitation, temperature, etc.) approximating those used on parts plated in the solution being tested.
6.3 The volume of the plating solution used to produce the test specimen shall be sufficient so that the concentration of the additives do not drop below 90% of original additive concentration. Additions to the test solution shall not be made since they can alter the original composition.

NOTE 2—The ductility of nickel, with or without chromium, will vary depending upon plating conditions, contamination, and the additive system used. If electroplated with chromium, the foil may require heating or aging to overcome temporary hydrogen embrittlement. A procedure to overcome hydrogen embrittlement is covered in 8.2 of Guide B 177.

6.4 Proper preparation of the surface from which the foil must be separated undamaged is critical. Buffed nickel or buffed stainless steel, which may require anodic treatment, in a hot alkaline cleaner as defined in 6.2, can be used as the basis metal. Steel or nickel dipped in a chromating solution, such as used for chromating zinc, can be used as the basis metal. Copper or brass, masked on one side, can be used as the basis metal, and can be subsequently dissolved from the coating to be tested. Because the dissolving step can embrittle the test deposit, the test deposit must be aged as stipulated in Note 2.

6.5 Cut off the edges of the panel with a power or hand shear, or by any convenient method that permits ready separation of the foil from the basis metal.

6.6 Using a pair of sharp scissors, cut two or more test specimens, about 5 to 75 mm from the center of the foil.

7. Procedure

7.1 Measure the thickness of the test foil with the micrometer at the point of bending. Bend the test foil in the shape of a “U” with the side of the foil that was against the basis metal facing inward in the “U”. Place the bent foil between the jaws of the micrometer so that as the jaws are closed, the bend remains between the jaws. Close the micrometer jaws slowly until the foil cracks (Note 3). Use an average of two or more foil tests. Record the micrometer reading as 2R and the thickness of the foil as determined by the micrometer as T.

7.2 At times, no single crack may develop over the convex surface. If jagged cracks or a series of shorter cracks develop (excluding edges), they signify failure. If no cracks develop, the maximum ductility values are obtained.

8. Calculation

8.1 Two standard formulas are used to compute ductility:

Ductility, percent \[= \frac{100T}{(2R−T)} \] \hspace{1cm} (1)

Maximum value is 100 %. \hspace{1cm} (1)

Ductility, ratio \[= \frac{T}{2R} \] \hspace{1cm} (2)

Maximum value is 0.5. \hspace{1cm} (2)

8.1.1 Either formula can be used but they give different values for the same ductility. It is important that the formula be consistently used for purpose of comparison. When reporting ductility values, the formula must be indicated.

8.2 It should be understood that this value bears no simple relation to the percentage elongation obtained through tension or other tests. The ductility of this type of low-ductility electrodeposited varies with the thickness. Usually the greater the thickness, the lower is the percentage ductility for these foils. (Note 2).

NOTE 3—With foils of a ductility of 70 % or greater using formula \[100T/(2R−T)\] or 0.4, using formula \[T/2R\] (see 8.1). It is helpful to examine the foil at low magnification (10×) while it is still in the micrometer.

This standard is issued under the fixed designation B 499; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the use of magnetic instruments for the nondestructive measurement of the thickness of nonmagnetic coatings over ferrous or other magnetic base metals.

NOTE 1—Autocatalytically deposited nickel-phosphorus alloys containing more than 8% phosphorus are sufficiently nonmagnetic to be measured by this test method, as long as the measurement is made prior to any heat treatment.

1.2 These instruments measure either the magnetic attraction between a magnet and the basis metal, as influenced by the presence of the coating, or the reluctance of a magnetic-flux path passing through the coating and the basis metal.

1.3 Measurements made in accordance with this test method will be in compliance with the requirements of ISO International Standard 2178 as printed in 1982.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 International Standard:

3. Significance and Use

3.1 The thickness of a coating is often critical to its performance. For most nonferrous coatings on steel, the magnetic method is reliable for measuring coating thickness nondestructively and is suitable for specification acceptance testing and SPC/SQC applications. The test method requires that the magnetic properties of the substrate used during the calibration be the same as that of the test specimen.

4. Factors Affecting the Measuring Accuracy

4.1 The following factors affect the accuracy of a coating thickness measurement:

4.1.1 Coating Thickness—Inherent to the test method is a measurement uncertainty that, for thin coatings, is constant and independent of the coating thickness. The magnitude of this measurement uncertainty is primarily a function of test piece surface finish (see 4.1.6 on surface roughness). For thicknesses greater than about 25µm, this uncertainty is proportional to the coating thickness.

4.1.2 Magnetic Properties of the Basis Metal—Magnetic thickness measurements are affected by variations in the magnetic properties of the basis metal. (For practical purposes, magnetic variations in low-carbon steel can often be considered to be insignificant. To avoid the influences of severe or localized heat treatments and cold working, the instrument should be calibrated using a calibration standard having a basis metal with the same magnetic properties as that of the test specimen or, preferably and if available, with a sample of the part to be tested before application of the coating.)

4.1.3 Basis Metal Thickness—For each instrument, there is a critical thickness of the basis metal above which the measurements will not be affected by an increase in the thickness of the basis metal. Since it depends on the instrument probe (Note 2) and the nature of the basis metal, its value should be determined experimentally if not supplied by the manufacturer.

NOTE 2—In this method “instrument probe” will also include the term “magnet.”

4.1.4 Edge Effects—The method is sensitive to abrupt changes in the surface contour of the test specimen. Therefore, measurements made too near an edge or inside corner will not be valid unless the instrument is specifically calibrated for such a measurement. The effect may extend to about 20 mm from the discontinuity, depending on the instrument.

4.1.5 Curvature—The measurements are affected by the curvature of the test specimen. The influence of curvature varies considerably with the make and type of instrument but always becomes more pronounced as the radius of curvature decreases. Instruments with two-pole probes may also produce different readings if the poles are aligned in planes parallel or
perpendicular to the axis of a cylindrical surface. A similar effect can occur with a single-pole probe if the tip is unevenly worn.

4.1.6 Surface Roughness—Measurements are influenced by the surface topography of the basis metal and coating. Surface roughness becomes significant when the degree of roughness is greater than 10% of the coating thickness, causing increased scatter in measurements. Therefore, it is necessary, on a rough or scratched surface, to make a greater number of measurements at different positions to obtain an average value that is representative of the mean coating thickness. If the basis metal is rough, it may also be necessary to check the zero of the instrument at several positions on a portion of the uncoated, rough, basis metal.

4.1.7 Direction of Mechanical Working of the Basis Metal—Measurements made by an instrument having a two-pole probe or an unevenly worn single-pole probe may be influenced by the direction in which the magnetic basis metal has been subjected to mechanical working (such as rolling), the reading changing with the orientation of the probe on the surface.

4.1.8 Residual Magnetism—Residual magnetism in the basis metal affects the measurements made by instruments which employ a stationary magnetic field. Its influence on measurements made by reluctance instruments employing an alternating magnetic field is much smaller.

4.1.9 Stray Magnetic Fields—Strong stray magnetic fields, such as are produced by various types of electrical equipment, can seriously interfere with the operation of magnetic thickness instruments.

4.1.10 Foreign Particles—Magnetic instruments of all types must make physical contact with the test surface and are, therefore, sensitive to foreign material that prevents intimate contact between probe and coating surface. Both the test surface and instrument probe should be kept free of foreign material.

4.1.11 Conductivity of Coating—Some magnetic instruments work at frequencies between 200 and 2000 Hz. At these frequencies, eddy currents produced in thick, highly conductive coatings may interfere with the reading.

4.1.12 Pressure—Instrument readings are sensitive to the pressure with which the probe is applied to the test specimen. Application of the probe should not be allowed to deform the coating.

4.1.13 Probe Orientation—Instruments using the principle of magnetic attraction may be sensitive to the orientation of the magnet in relation to the field of gravity of the earth. Thus, the operation of an instrument in a horizontal or upside-down position may require a different calibration, or may be impossible.

5. Calibration of Instruments

5.1 Before use, each instrument shall be calibrated in accordance with the instructions of the manufacturer, employing suitable thickness standards.

5.2 During use, the calibration shall be checked at frequent intervals, at least once a day. Attention shall be given to the factors listed in Section 4 and to the procedures described in Section 5.

5.3 Calibration standards of known thickness are available either as shims or foils or as coated specimens.

5.3.1 Calibration Foils:

Note: In the following paragraphs, the use of the word “foil” will imply a nonmagnetic metallic or nonmetallic foil or shim.

5.3.1.1 Because of the difficulty of ensuring adequate contact, foils are generally not recommended for the calibration of instruments based on the principle of magnetic attraction but they are suitable in some circumstances provided the necessary precautions are taken. They can normally be used for the calibration of other types of instruments.

5.3.1.2 Foils are advantageous for calibration on curved surfaces and are more readily available than coated standards. To prevent measurement errors it is necessary to ensure that intimate contact is established between foil and substrate. Resilient foils should be avoided to prevent indentation errors. Only nonferrous metal foils should be used for thicknesses less than 15 μm. Calibration foils are subject to wear and indentation and, therefore, should be replaced frequently. Worn foils shall not be used to calibrate the instrument.

5.3.2 Coated Standards—These calibration standards consist of coatings of known, uniform thickness permanently bonded to the substrate material.

5.4 The basis metal of the calibration standards shall have magnetic properties similar to those of the basis metal of the coated test specimen. To confirm their suitability, a comparison of the readings obtained with the basis metal of the bare standard and that of the test specimen is recommended.

5.5 In some cases the calibration of the instrument should be checked by rotating the probe in increments of 90° (see 4.1.7 and 4.1.8).

5.6 The basis-metal thickness for the test and the calibration shall be the same if the critical thickness, defined in 4.1.3, is not exceeded. It is often possible to back up the basis metal of the standard or of the test specimen with a sufficient thickness of similar material to make the readings independent of the basis-metal thickness.

5.7 If the curvature of the coating to be measured is such as to preclude calibration on a flat surface, the curvature of the coated standard, or of the substrate on which the calibration foil is placed, shall be the same.

6. Measuring Procedure

6.1 Operate each instrument in accordance with the instructions of the manufacturer giving appropriate attention to the factors listed in Section 4.

6.2 Check the calibration of the instrument at the test site each time the instrument is put into service and at frequent intervals during use to assure proper performance.

6.3 Observe the following precautions:

6.3.1 Basis-Metal Thickness—Check whether the basis-metal thickness exceeds the critical thickness. If not, either use the back-up method mentioned in 5.6 or make sure that the calibration has been made on a standard having the same thickness and magnetic properties as the test specimen.

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3 Coated standards suitable for many applications of the test method may be purchased from the office of Standard Reference Materials, National Institute of Standards and Technology, Gaithersburg, MD 20899.
6.3.2 Edge Effects—Do not make readings close to an edge, hole, inside corner, etc., of a specimen unless the validity of the calibration for such a measurement has been demonstrated.

6.3.3 Curvature—Do not make readings on a curved surface of a specimen unless the validity of the calibration for such a measurement has been demonstrated.

6.3.4 Number of Readings—Because of normal instrument variability and in order to minimize surface roughness effects, a measurement shall be the mean value of several readings.

6.3.4.1 For each measurement, make at least 3 readings, removing the probe after each reading, and average the readings. If any 2 of the readings differ from each other by more than 5 % of the average reading or 2 µm, whichever is the greater, then the measurement shall be discarded and repeated.

6.3.4.2 The substrate or coating, or both may be too rough to meet this criterion. In such a case it may be possible to obtain a valid measurement by averaging a number of readings. To be valid under this test method, the validity of such a procedure must be demonstrated (see Appendix X1).

6.3.4.3 Instruments of the attractive force type are sensitive to vibrations, and readings that are obviously erroneous should be rejected.

6.3.5 Direction of Mechanical Working—If the direction of mechanical working has a pronounced effect on the reading, make the measurement on the test specimen with the probe in the same orientation as that used during calibration. If this is impossible, make four measurements in various orientations by rotating the probe in increments of 90°.

6.3.6 Residual Magnetism—When residual magnetism is present in the basis metal, when using two-pole instruments employing a stationary magnetic field make measurements in two orientations differing by 180°. With single-pole instruments employing a stationary magnetic field, it may be necessary to demagnetize the test specimen to get valid results, and this may also be advisable with two-pole instruments.

6.3.7 Surface Cleanliness—Before making measurements, clean any foreign matter such as dirt, grease, and corrosion products from the surface without removing any coating material. Avoid any areas having visible defects, such as welding or soldering flux, acid spots, dross, or oxide when making measurements.

6.3.8 Lead Coatings—The magnet of an instrument of the attractive force type may stick to lead and lead alloy coatings. Apply a very thin film of oil to improve the reproducibility of readings and correct the measurement for the thickness of the oil film. Excess oil shall be wiped off so that the surface is virtually dry. The correction may be determined by measuring the coating thickness of a nonsticking coating of appropriate thickness with and without the oil film and taking the difference between the two measurements. Do not use this procedure with other coatings.

6.3.9 Techniques—The readings obtained may depend on the technique of the operator. For example, the pressure applied to a probe, or the rate of applying a balancing force to a magnet, will vary from one individual to another. Reduce or minimize such effects either by having the instrument calibrated by the same operator who will make the measurement or by using constant-pressure probes. In appropriate cases when a constant pressure probe is not being used, the use of a measuring stand is strongly recommended.

6.3.10 Positioning of Probe—In general, place the instrument probe perpendicular to the specimen surface at the point of measurement. For some instruments of the attractive force type, this is essential. With some instruments, however, it is desirable to tilt the probe slightly and select the angle of inclination giving the minimum reading. If, on a smooth surface, the readings obtained vary substantially with the angle of inclination, it is probable that the probe is worn and needs to be replaced. If a magnetic instrument is to be used in a horizontal or upside-down position, calibrate it for that position.

7. Report

7.1 The report should include the following information:
7.1.1 Type of instrument used,
7.1.2 Size and description of test specimen,
7.1.3 Whether special jigs were used,
7.1.4 Type of calibration standard and the method used,
7.1.5 Thickness of the coating as determined from the measurements,
7.1.6 Operator identification, and
7.1.7 Date.

8. Precision and Bias

8.1 The equipment, its calibration, and its operation shall be such that the coating thickness can be determined with an uncertainty of less than 10 % at 95 % confidence level.

8.2 Although an uncertainty of less than 10 % may be achieved consistently for a great number of applications, the uncertainty may be greater when coating thickness is less than 25 µm.

8.3 Instruments suitable for compliance with 6.1 are available commercially. For many coating systems, the instruments are capable of making measurements with an uncertainty of less than 5 % at 95 % confidence level.

8.4 The measurement bias is the discrepancy remaining between the measured thickness and the true thickness if all random errors are eliminated. It is, therefore, no greater than and attributable to (1) the calibration error of the instrument and (2) the quality of the calibration standard used to calibrate the instrument.

8.5 The precision is being determined by round-robin testing.

9. Keywords

9.1 coating thickness; coatings; magnetic method; nonmagnetic coatings; thickness; thickness testing
X1. MEASUREMENTS ON ROUGH SURFACES

X1.1 Measurements on rough surfaces are subject to random errors associated with the position of the instrument probe relative to the peaks and valleys of the rough surface. These random errors increase with surface roughness, but can be reduced by averaging 10 or more readings.

X1.2 Roughness can also introduce a bias (systematic error) because the probe seldom, if ever, rests at the bottom of a valley; and the magnetic field in the neighborhood of the probe differs from that at a smooth surface. In the case of a rough substrate, the valleys are filled with coating material but when the instrument is calibrated with a foil, the foil rests on the peaks of the substrate. A bias can be corrected for if the magnitude of the bias can be determined by microscopical or other measurements.
Standard Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method\(^1\)

This standard is issued under the fixed designation B 504; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (\(\epsilon\)) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of the thickness of metallic coatings by the coulometric method, also known as the anodic solution or electrochemical stripping method.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ISO Standard:

ISO 2177 Metallic Coatings—Measurement of Coating Thickness—Coulometric Method by Anodic Dissolution\(^2\)

3. Summary of Test Method

3.1 The thickness of the coating is determined by measuring the quantity of electricity (coulombs) required to dissolve the coating anodically from a known and accurately defined area.

3.2 As commonly practiced, the method employs a small metal cell which is filled with an appropriate electrolyte. The test specimen serves as the bottom of the cell and an insulating gasket between the cell and the specimen defines the test area. The test specimen as anode and the cell gasket may be presumed to meet these requirements; others must be evaluated before use by testing standards having known thicknesses. Appendix X1 lists some electrolytes and coating-substrate combinations that have been used with some instruments.

3.3 Commercial instruments using this principle are available. The method is rapid and versatile, but destructive to the coating. In general, its range is considered to be between 0.75 and 50 \(\mu\)m. Chromium, gold, tin, and other coatings can be measured down to 0.075 \(\mu\)m.

4. Significance and Use

4.1 Measurement of the thickness of a coating is essential to assessing its utility and cost.

4.2 The coulometric method destroys the coating over a very small (about 0.1 \(\text{cm}^2\)) test area. Therefore its use is limited to applications where a bare spot at the test area is acceptable or the test piece may be destroyed.

5. Factors Affecting the Accuracy of the Method

5.1 Composition of Electrolytes—Electrolytes used for coulometric thickness measurements must permit the coating metal to dissolve at a constant anodic-current efficiency (preferably 100 %); they must have a negligible spontaneous chemical effect on the coating metal and must so differentiate electrochemically between the coating and the substrate that a suitably sharp and large voltage change occurs at the end point of the test.

5.1.1 Electrolytes furnished with commercial instruments may be presumed to meet these requirements; others must be evaluated before use by testing standards having known thicknesses. Appendix X1 lists some electrolytes and coating-substrate combinations that have been used with some instruments.

5.2 Current Variation—For coulometric instruments employing the constant-current technique, variation of the current during a test will result in errors. For instruments using a current-time integrator, variation of the current during a test will not result in error unless the current change is such as to displace the anodic current density beyond the range of constant or 100 % anodic-current efficiency.

5.3 Area Variation—The accuracy of the thickness measurement will not be better than the accuracy with which the test area is defined or known. Typically, this test area is defined by a flexible, insulating gasket. Area variation is usually minimized by using as large an area as practical and by using a constant pressure device. If excessive pressure is applied to such a gasket, the test area may be altered undesirably.
5.4 Agitation—In most, but not all, coulometric thickness measurements, a relatively high anodic-current density is employed to shorten the test time. It is then necessary to agitate the electrolyte to maintain a constant anodic-current efficiency. Where agitation is required, insufficient agitation may result in polarization of the specimen, thereby causing a premature and false endpoint.

5.5 Alloying Between Coatings and Metallic Substrates—The measurement of a coating thickness by the coulometric method implicitly assumes that a sharply defined interface exists between the coating and the substrate. If an alloy layer exists between the coating and the substrate as, for example, in the case of coatings applied by hot dipping, the coulometric end-point may occur at some point within the alloy layer, thus giving a high value of the thickness of the unalloyed coating.

5.6 Purity of Coating—Impurities or additives that codeposit with the coating may change the effective electrochemical equivalent of the coating and also change the anodic current efficiency.

5.6.1 Alloy Coating—Variations in the composition of alloy coatings will change the effective electrochemical equivalent of the coating.

5.7 Cleanliness of Test Surface—The surface to be tested must be clean. Oil, grease, and organic coatings such as lacquer must be removed with suitable solvents. Oxides, conversion coatings, and corrosion products are preferably removed by carefully burnishing the test surface with a clean, soft pencil eraser. Tin and nickel surfaces, in particular, should be so burnished prior to testing to remove passive oxide films.

5.8 Density of Coating—The coulometric method intrinsically measures coating mass per unit area, the equivalent linear thickness being a function of the density of the coating. If the density of the coating tested is different from the value of the density used for the calibration, the linear thickness obtained coulometrically will be different from the actual linear thickness of the coating tested.

5.8.1 Density of Alloy Coatings—Variation in the composition of alloy coatings will change the density of the coating.

5.9 Number and Location of Tests—Since the coulometric test method measures, essentially, a local coating thickness, a single test may not be representative of the coating thickness over the entire significant surface.

6. Calibration of Equipment

6.1 The equipment shall be calibrated by means of standards having known coating thicknesses. If commercial equipment is used, the manufacturer's instructions shall be followed insofar as they are compatible with this test method.

6.2 Calibration of Direct-Reading Instruments—Direct-reading instruments shall be calibrated against standards having known coating thicknesses, and adjusted to produce correct readings corresponding to the coating thicknesses of the standard.

6.3 Calibration of Nondirect-Reading Instruments:

6.3.1 Nondirect-reading instruments shall be calibrated against standards having a known coating thickness by using a calibration constant, $C$, calculated as follows:

$$C = \text{coating thickness of the standards/instrument reading} \quad (1)$$

6.3.2 The instrument shall be adjusted so that where standards having known coating thicknesses are tested, the correct thickness is obtained by multiplying the instrument reading by the calibration constant, $C$.

6.4 Thickness Standards—The thickness standards shall consist of the same type of coating and substrate as the specimens to be measured, and they shall have an accuracy of ±5 % or better.

7. Procedure for Making Measurements

7.1 If commercial equipment is used, the manufacturer's instructions shall be followed insofar as they are compatible with this test method.

7.2 The test surface shall be cleaned of all foreign material that might affect the measurement.

Note 1—Certain nickel deposits, frequently dull nickel, may exhibit passivity. When such coatings are tested coulometrically, the voltage across the specimen and test cell is markedly higher (approximately 1 V) than normal, and the coating does not dissolve. Oxygen is evolved at the specimen and the test may continue indefinitely.

Note 2—Removal of the passivity may be accomplished in some cases by mildly abrading (as with a pencil eraser) the nickel surface prior to testing. Alternatively, the specimen may be made cathodic in the coulometric electrolyte for 10 to 20 s by applying current from an external source. Allowing the nickel to be in contact with 10 % volume hydrochloric acid for approximately 1 min prior to the test may also be used effectively.

7.3 After completion of the measurement, the test surface shall be examined visually, and if the dissolution of the coating is not virtually complete, the measurement shall be discarded and repeated.

8. Precision and Bias

8.1 The equipment, its calibration, and its operation shall be such that the coating thickness can be determined with an uncertainty of less than 10 %.

8.2 Instruments suitable for compliance with 8.1 are available commercially. For many coating systems the instruments are capable of making measurements with an uncertainty of less than 4 % (95 % confidence).

8.3 Although an uncertainty of less than 10 % may be achieved consistently for a great number of coating-substrate combinations, the uncertainty may be greater when the coating thickness is less than 1 µm or exceeds 50 µm.

8.4 The bias of a coulometric measurement is the discrepancy remaining between the measured thickness and the true thickness if all random errors are eliminated. It is, therefore, no greater than, and attributable to (1), the calibration error of the instrument and (2) the quality of the calibration standard used to calibrate the instrument.
X1. ELECTROLYTES

X1.1 Table X1.1 lists electrolytes that have been used for coulometric thickness measurements; however, they are not necessarily suitable for use with all types of coulometric instruments.

X1.2 Use of these electrolytes is not mandatory for compliance with this method, and when commercial coulometric instruments are used, the manufacturer’s recommendations shall be followed.

X1.3 Table X1.2 lists other coating-substrate combinations that have been used successfully with commercially available electrolytes.

### TABLE X1.1 Typical Electrolytes for Electrodeposited Coatings

<table>
<thead>
<tr>
<th>Coating</th>
<th>Steel</th>
<th>Copper and Alloys (such as brass)</th>
<th>Nickel</th>
<th>Aluminum</th>
<th>Zinc</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cadmium</td>
<td>1, 10</td>
<td>1, 10</td>
<td>1</td>
<td>1</td>
<td>...</td>
</tr>
<tr>
<td>Chromium</td>
<td>2, 11</td>
<td>3, 4, 12</td>
<td>2, 13</td>
<td>2, 13</td>
<td>...</td>
</tr>
<tr>
<td>Copper</td>
<td>5, 14</td>
<td>...</td>
<td>5, 15</td>
<td>5</td>
<td>16</td>
</tr>
<tr>
<td>Lead</td>
<td>17</td>
<td>17</td>
<td>17</td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>Nickel</td>
<td>6, 18</td>
<td>6, 19</td>
<td>...</td>
<td>6, 18</td>
<td>...</td>
</tr>
<tr>
<td>Silver</td>
<td>7</td>
<td>8</td>
<td>7</td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>Tin</td>
<td>3, 4, 20</td>
<td>3, 4, 20</td>
<td>3, 4</td>
<td>2, 21</td>
<td>...</td>
</tr>
<tr>
<td>Zinc</td>
<td>9</td>
<td>9</td>
<td>9</td>
<td>9</td>
<td>...</td>
</tr>
</tbody>
</table>

A The numbers in the table refer to the following aqueous solutions:

(1) 100 g KI/L, with traces of I₂
(2) 100 g Na₂SO₄/L
(3) 175 mL HCl (sp gr 1.18)/L
(4) 150 g NaOH/L
(5) 80 g Na₂C₂H₃O₇ (sodium potassium tartrate) + 100 g NH₄NO₃/L
(6) 30 g NH₄NO₃ + 30 g NaSCN/L
(7) 100 g NaNO₃ + 3 mL HNO₃ (sp gr 1.42)/L
(8) 180 g KSCN/L
(9) 100 g NaCl or KCl/L
(10) 30 g KCl + 30 g NH₄Cl/L
(11) 100 mL H₂PO₄ (sp gr 1.75) + 10 g CrO₃/L
(12) 100 g Na₂CO₃ [for coatings up to 5 µm (0.2 mil)]/L
(13) 84 mL H₂PO₄ (sp gr 1.75)/L
(14) 800 g NH₄NO₃ + 10 mL NH₄OH (sp gr 0.88)/L
(15) 100 g K₂SO₄ + 20 mL H₂PO₄ (sp gr 1.75)/L
(16) Pure H₂SiF₆ solution containing not less than 30 % H₂SiF₆ (Slightly weaker acid may be used, if some MgSiF₆ is added to the solution.)
(17) 200 g CH₃COONa + 200 g CH₃COOH/L
(18) 800 g NH₄NO₃ + 3.8 g CSI(NH₃)₂ (thiourea)/L
(19) 100 mL HOI (sp gr 1.18)/L
(20) 100 g KNO₃ + 100 g KCl/L
(21) 50 mL H₂SO₄ (sp gr 1.84) + 5 g KF/L
### TABLE X1.2 Measurable Coating-Substrate Combinations

<table>
<thead>
<tr>
<th>Substrate Coating</th>
<th>Aluminiun</th>
<th>Copper and Copper Alloys</th>
<th>Nickel</th>
<th>Steel</th>
<th>Magnetic Stainless Steel</th>
<th>Nonmetallic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brass^A</td>
<td>X</td>
<td>X</td>
<td></td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Cobalt</td>
<td></td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Gold</td>
<td>X</td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Indium</td>
<td></td>
<td>X</td>
<td></td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Iron</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nickel (Electrodes)^A</td>
<td>X</td>
<td>X</td>
<td></td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nickel-Iron^B</td>
<td></td>
<td></td>
<td></td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tin-Lead^A</td>
<td></td>
<td>X</td>
<td></td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tin-Nickel^A</td>
<td></td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tin-Zinc^A</td>
<td></td>
<td>X</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

^A The measurement accuracy of these alloy coatings is dependent on the composition of the coating.
Standard Practice for Design of Articles to Be Electroplated on Racks

This standard is issued under the fixed designation B 507; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers design information for parts to be electroplated on racks. The recommendations contained herein are not mandatory, but are intended to give guidance toward good practice.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Significance and Use

2.1 When an article is to be electroplated, it is necessary to consider not only the characteristics of the electroplating process, but also the design of the part to minimize electroplating and finishing costs and solution dragout as well as to improve appearance and functionality. It is often possible during the design and engineering stages to make small adjustments in shape that will result in considerable benefit toward a better quality part at a lower cost.

2.2 The specific property of an electroplating process that would require some attention to the details of optional designs, is the throwing power of the electroplating solution. Throwing power is the improvement of the coating distribution over the primary current distribution on an electrode (usually cathode) in a given solution, under specified conditions.

3. Current Distribution and Throwing Power

3.1 The apparent current during practical electroplating is never uniform over the surface of the product. Even parallel plates have a nonuniform distribution of current when freely suspended in a bath as shown in Fig. 1. In this example, the current lines tend to concentrate as corners, and edges (high-current density) of the part. Consequently more metal is deposited at the high-current density areas than at the low-current density areas.

4. Relative Throwing Powers of Different Electrolytes

4.1 Throwing power is not the same for all metals and all electroplating baths. Table 1 lists the commonly used electroplating processes. They are arranged according to decreasing throwing power.

4.2 A Rochelle-type copper electroplating solution has excellent throwing power compared to the poor throwing power of a chromic acid solution used to deposit chromium. The widely used Watts-type nickel bath has fair throwing power.

5. Geometric Factors Determining Deposit Distribution

5.1 Since a metal deposits preferentially at protuberances, such as sharp corners, edges, fins, and ribs, these should be rounded to a radius of at least 0.4 and preferably 0.8 mm to avoid excessive buildup. Contouring a base corner in a depression is also recommended to avoid thickness deficiency at the location.

5.2 The width-to-depth ratio of a depression or recess should be held to more than three as shown in Fig. 2. Otherwise, a special auxiliary anode must be employed inside the recess to promote more uniform current distribution. An auxiliary anode is usually made of the depositing metal and is placed close to the low-current density areas to enhance metal deposition at those regions.

5.3 All sharp edges and base angles of a recess should be rounded to a radius of 0.25 times or more the depth of the recess as shown in Fig. 3. When sharp recess angles are required for a functional purpose, the electroplater cannot be expected to meet a minimum thickness at those locations unless it is specifically required.

6. Examples of Distribution of Electrodeposited Nickel on Various Shapes

6.1 Fig. 4 through Fig. 5 show the kind of nickel distribution that was obtained on several different cathode configurations as deposited from a Watts-type bath at normal operating current densities. The thicknesses illustrated are exaggerated to emphasize the variations that were obtained. The data are

1 This practice is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.01 on Ancillary Activities.


measurements taken from metallographic cross sections. Reference to the figures enables similar conclusions to be drawn with most other metals, excluding chromium. The ranges will be smaller for metals above nickel in Table 1 and larger for metals below nickel.

6.2 Improvement in nickel distribution can be gained inside an angle by increasing the angle size, as shown in Fig. 4. Two surfaces meeting at an angle of $60^\circ$ show an average-to-minimum thickness ratio of 3.3, and increasing the angle to $90^\circ$ or $120^\circ$ the ratio can be reduced to 2.7 or 1.9, respectively.

6.3 Sharp corners should be given as large a radius as practical to improve metal distribution in a recess and avoid excessive buildup on protuberances. Fig. 6(a) illustrates a part with a sharp angled recess. Nickel distribution is not very uniform with practically no deposit down in the corners of the recess. Rounding the corners of the recess on the part, as shown in Fig. 6(b), yields a more uniform nickel thickness in the recess. The average-to-minimum thickness ratio in these examples was 9.2 for the part with sharp corners and 5.6 for the part with the rounded corners.

6.4 Deep recesses will always have a thinner deposit than the surrounding external areas, as shown in the cross section of a concave part in Fig. 7(a). The average-to-minimum nickel thickness ratio for this example was 6.6. A more uniform deposit thickness can be obtained on a convex-shaped part, as shown in the example of Fig. 7(b). In this case the average-to-minimum nickel thickness ratio was 2.

6.5 Another example of an elongated curved surface (convex) is illustrated in Fig. 5(a). The nickel deposit was fairly uniform with an indicated average-to-minimum thickness ratio of 2. However, when this shape is joined to another like a flat plate, metal distribution is considerably different as illustrated by Fig. 5(b).

7. Racking and Rinsing

7.1 Other factors besides metal distribution should also be taken into account when designing a part that will be rack electroplated. The parts must be attached firmly to the rack, so that all significant surfaces come in contact with the electrolyte.

7.2 The parts must be attached to a rack firmly enough to prevent falling off during electroplating, and the attachment should be with enough force to provide a continual low-resistance electrical contact. In many cases, parts are rigidly fastened to racks through spring clips, prongs or bolts. Little or

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**TABLE 1 Relative Throwing Powers of Common Electroplating Baths**

<table>
<thead>
<tr>
<th>Bath/Metal</th>
<th>Ranking</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rochell copper (cyanide based)</td>
<td>Excellent</td>
</tr>
<tr>
<td>Cyanide cadmium</td>
<td>Excellent</td>
</tr>
<tr>
<td>Cyanide gold</td>
<td>Good</td>
</tr>
<tr>
<td>Cyanide silver</td>
<td>Good</td>
</tr>
<tr>
<td>Alkaline tin</td>
<td>Good</td>
</tr>
<tr>
<td>Cyanide zinc</td>
<td>Good</td>
</tr>
<tr>
<td>Alkaline non cyanide zinc</td>
<td>Good</td>
</tr>
<tr>
<td>Fluoborate lead</td>
<td>Good</td>
</tr>
<tr>
<td>All chloride nickel</td>
<td>Fair</td>
</tr>
<tr>
<td>Tin nickel</td>
<td>Fair</td>
</tr>
<tr>
<td>Sulfamate nickel</td>
<td>Fair</td>
</tr>
<tr>
<td>Watts nickel&lt;sup&gt;A&lt;/sup&gt;</td>
<td>Fair</td>
</tr>
<tr>
<td>Bright nickel</td>
<td>Fair</td>
</tr>
<tr>
<td>Acid chloride zinc</td>
<td>Fair</td>
</tr>
<tr>
<td>Nickel-iron</td>
<td>Fair</td>
</tr>
<tr>
<td>Chloride iron</td>
<td>Fair</td>
</tr>
<tr>
<td>Pyrophosphate copper</td>
<td>Fair</td>
</tr>
<tr>
<td>Acid copper</td>
<td>Fair</td>
</tr>
<tr>
<td>Trivalent chromium</td>
<td>Poor</td>
</tr>
<tr>
<td>Hexavalent chromium</td>
<td>Poor</td>
</tr>
</tbody>
</table>

<sup>A</sup> Used for examples illustrated by Figs. 4-5.
no metal will deposit at the points of contact; therefore, it is important to select noncritical areas for attaching parts to racks.

7.3 Articles attached to racks should be oriented to permit electroplating to be free of roughness on significant surfaces. Roughness comes from insoluble debris suspended in an electroplating bath that becomes incorporated into the depositing metal, especially on upward facing surfaces. Thus, in many circumstances, it is advisable to have the significant surfaces in a vertical position, or even be inverted during electroplating.

7.4 Orientation of a part on a rack is also important to reduce opportunities for air entrapment in cupped areas. Air pockets will prevent metal deposition on the exposed surface. Adequate drainage of parts on racks is also desirable to reduce dragout of the electrolyte to the rinses. Engineering design can incorporate holes at strategic locations to allow satisfactory runoff of solution.
FIG. 5 Nickel Distribution on a Convex Surface (a) alone Compared to the Same Configuration as Part of a Larger Composite (b)

Maximum - 69 μm - Minimum - 18 μm

Average - 36 μm - Ratio Average Minimum - 2.0

Maximum - 56 μm - Minimum - 2.5 μm

Average - 20 μm - Ratio Average Minimum - 8.0

FIG. 6 Improving Nickel Thickness Distribution (Average/Minimum Thickness Ratio) by Rounding Corners

Maximum - 65 μm

Minimum - 2.5 μm

Average - 23 μm

Ratio Average Minimum - 9.2

Maximum - 81 μm

Minimum - 5 μm

Average - 28 μm

Ratio Average Minimum - 5.6
FIG. 7 Comparing Nickel Distribution on Concave (a) and Convex (b) Surfaces

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Standard Test Method for Determination of Tap Density of Metallic Powders and Compounds

This standard is issued under the fixed designation B 527; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method specifies a method for the determination of tap density (packed density) of metallic powders and compounds, that is, the density of a powder that has been tapped, to settle contents, in a container under specified conditions.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   B 215 Practice for Sampling Finished Lots of Metal Powders

3. Significance and Use

3.1 This test method covers the evaluation of the tapped density physical characteristic of metallic powders and compounds. The degree of correlation between the results of this test method and the quality of powders in use will vary with each particular application and has not been fully determined.

4. Apparatus

4.1 Balance, of appropriate capacity and accuracy to satisfy the requirements shown in Table 1.

4.2 Graduated Glass Cylinder,3 calibrated to contain 100 cm³ at 20°C, the height of the graduated portion being approximately 175 mm. The graduations shall be at 1 cm³ intervals, thus allowing a measuring accuracy of ± 0.5 cm³. For apparent densities over 4.0 g/cm³, do not use the 100 cm³ cylinder.

4.2.1 Alternatively, the following may be used:

4.2.1.1 Graduated Glass Cylinder, calibrated to contain 25 cm³ at 20°C, the height of the graduated portion being approximately 135 mm. The graduations shall be at 0.2 cm³ intervals.

4.2.1.2 A 25-cm³ cylinder shall be used for powders of apparent density higher than 4 g/cm³, in particular for refractory metal powders, but may also be used for powder of lower apparent density.

4.3 Tapping Apparatus,4 which permits the tapping of the graduated cylinder against a firm base. The tapping shall be such that a densification of the powder can take place without any loosening of its surface layers. The stroke shall be 3 mm (0.118 in.) and the tapping frequency shall be between 100 and 300 taps/min. An example of a tapping apparatus is shown in Fig. 1.

5. Test Specimen

5.1 For the quantities of powder required for each test, see Table 1. Obtain test powder samples according to Practices B 215.

5.2 In general, the powder should be tested in the as-received condition. In certain instances the powder may be dried. However, if the powder is susceptible to oxidation, the drying shall take place in a vacuum or in inert gas. If the powder contains volatile substances, it shall not be dried.

5.3 The test shall be carried out on three test samples.

6. Procedure

6.1 Clean the inside wall of the graduated cylinder (5.2) with a suitable clean brush or, if necessary, by rinsing with a

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1 This specification is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.03 on Refractory Metal Powders.

2 Annual Book of ASTM Standards, Vol 02.05.

3 Corning No. 3046, Pyrex brand has been found suitable.

4 The following have been found suitable: Shandon Southern Instruments, Inc., Tap-Pak Volumeter Model JEL-ST2 (Manufactured by J. Engelmann A.G. or Ludwigshafen 9. Rh. West Germany), 515 Broad Street, Sewickly, PA 15143; Vankel Industries, Vanderkamp Tap Density Tester, 36 Meridian Road, Edison, NJ 08820; Quantachrome Corp., Dual Autotap, 6 Aerial Way, Syosset, NY 11791.
6.2 Weigh, to the nearest 0.1 g, the mass of the test portion as indicated in Table 1, using a balance (4.1).

6.3 Pour the test portion into the graduated cylinder. Take care that a level surface of the powder is formed. Place the cylinder in the tapping apparatus (4.3). Tap the cylinder until no further decrease in the volume of the powder takes place (see Note 1).

**NOTE 1**—In practice, the minimum number of taps, \( N \), such that no further change in volume takes place would be determined. For all further tests on the same type of powder, the cylinder would be subjected to \( 2N \) taps, except where general experience and acceptance have established a specific number of taps (no less than \( N \) taps) as being satisfactory. For fine refractory metal powders, 3000 taps has been found to be satisfactory for all sizes.

6.4 If the tapped surface is level, read the volume directly. If the tapped surface is not level, determine the tape volume by calculating the mean value between the highest and the lowest reading of the tapped surface. Read the final volume to the nearest 0.5 cm\(^3\) when using a 100 cm\(^3\) cylinder and to the nearest 0.2 cm\(^3\) when using a 25 cm\(^3\) cylinder.

7. Calculation

7.1 The tap density is given in the following equation

\[
PT = \frac{M}{V}
\]

where:

- \( PT \) = tap density, g/cm\(^3\),
- \( M \) = mass of powder, g, and
- \( V \) = volume of tapped powder, cm\(^3\).

8. Report

8.1 Report the following information:

8.1.1 Reference to this test method,
8.1.2 All details necessary for identification of the test sample,
8.1.3 The drying procedure, if the powder has been dried,
8.1.4 Cylinder capacity, mass of test portion and method used,
8.1.5 The result obtained,
8.1.6 All operations not specified in this test method or regarded as optional,
8.1.7 Details of any occurrence that may have affected the result.

9. Precision and Bias

9.1 Precision and bias cannot be stated at this time because this test method covers a broad range of powders and associated densities.

10. Keywords

10.1 apparent density; bulk density; density; density ratio; metal powders; packed density; powder metallurgy; tap density
1. Scope

1.1 This test method covers determination of the transverse rupture strength of sintered, including post-treated, metal powder test specimens by subjecting them to a uniformly increasing transverse force under controlled conditions. The term “transverse rupture strength” as used herein, defines the stress, calculated from the flexure formula required to break a specimen as a simple beam supported near the ends and applying the force midway between the fixed line center of the supports.

1.2 Limitations — The transverse rupture test is only applicable to relatively brittle materials. In cases where a ductile specimen is being tested and the permanent deflection as a result of testing exceeds 0.020 in. (0.50 mm), the test results may be questionable.

1.3 The values stated in inch-pound units are to be regarded as the standard. The SI values are provided for information only.

1.4 Test Method B 406 should be consulted for determining the transverse rupture strength of cemented carbides.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 243 Terminology of Powder Metallurgy

B 312 Test Method for Green Strength for Compacted Metal Powder Specimens

B 406 Test Method for Transverse Rupture Strength of Cemented Carbides

3. Terminology

3.1 Definitions — For definitions of terms in this test method, see Terminology B 243.

4. Significance and Use

4.1 This test method is used to measure the strength of sintered, including post-treated, specimens. Transverse rupture strength is not a design value. For many sintered materials, transverse rupture strength is approximately twice the ultimate tensile strength.

5. Apparatus

5.1 Punches and Die (Fig. 1), for producing a test specimen in accordance with 6.1

5.2 Equipment capable of applying the required pressure to produce the standard test specimen.

5.3 Balance, suitable for weighing accurately to 0.01 g.

5.4 Micrometer, with a resolution of 0.0001 in. (0.002 mm).

5.5 Transverse Rupture Strength Fixture, as shown in Fig. 2.

5.6 Compression Testing Machine, capable of applying a breaking force on the test specimen with an accuracy of 1.0 % and readable to the nearest 10 lbf (50 N).

6. Test Specimen

6.1 The test specimen shall have the shape and dimensions shown in Fig. 3.

7. Procedure

7.1 Prepare test specimens by pressing and sintering using tooling described in Test Method B 312, or machine from finished parts. Measure the width and thickness of the specimens to the nearest 0.001 in. (0.03 mm) with a micrometer.

7.2 Locate the specimen in the transverse rupture test fixture perpendicular to the supporting rods. Apply compressive force parallel to the pressing direction of the specimen at a no-load crosshead speed of not greater than 0.2 in. (5.0 mm)/min until rupture occurs. Record this breaking force to the nearest 10 lbf (50 N).

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1 This test method is under the jurisdiction of ASTM Committee B-9 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.05 on Structural Parts.


2 Annual Book of ASTM Standards, Vol 02.05.
NOTE 1—The dimensions for the cavity shall be 0.500 ± 0.004 in. wide (12.70 ± 0.10 mm) by 1.250 ± 0.004 in. long (31.75 ± 0.10 mm). The mating parts shall fit freely and should be finished to a 4-µin. (N3) or better, to dimensions of 0.0005 in. (0.013 mm) to 0.0010 in. (0.025 mm) smaller than the die cavity in each dimension. The dimensions given in the drawing typify the die cavity and punch within the stated tolerance at the normal width and length.

FIG. 1 Example of Tooling to Produce the Test Specimen
8. Calculation

8.1 Calculate the transverse rupture strength of the compact as follows:

\[ TRS = \frac{3 \times P \times L}{(2 \times t^2 \times w)} \]  

where:

- \( TRS \) is the transverse rupture strength.
- \( P \) is the load applied.
- \( L \) is the length of the specimen.
- \( t \) is the thickness of the specimen.
- \( w \) is the width of the specimen.
TRS = transverse rupture strength of the compact, psi (MPa),
P = force required to rupture the specimen, lbf (N),
L = length of specimen span relative to fixture 1.000 in. (25.4 mm),
w = width of the specimen, in. (mm), and
t = thickness of specimen, in. (mm).

9. Report

9.1 Report the transverse rupture strength in pounds-force per square inch (megapascals) to the nearest 1000 psi (10 MPa). When reporting the results, state the following supplementary data to clarify the results:

9.1.1 Identification of the material being tested, that is, composition, density, state of heat treatment, and compacted or machined specimen.

10. Precision

10.1 On the basis of test error alone, the difference in the absolute value of two test results, \( x_1 \) and \( x_2 \), obtained in the same laboratory on the same material, will be expected to exceed \( r \), the repeatability interval, only about 5% of the time. If such a difference is found to be larger than \( r \) there is reason to question one or both results. The value of \( r \) varies proportionally with the magnitude of the transverse rupture strength and is calculated from the following equation:

\[
r = 6700 + (0.064) (x - 41000)
\]

where the units of \( r \) and \( x \) are psi. This applies over the range from \( x \) from 41 500 to 145 000 psi. The unit \( x \) is the average of the two points being checked.

\[
\frac{x_1 + x_2}{2} = x
\]

10.2 On the basis of test error alone, the difference in absolute value of two test results, \( x_1 \) and \( x_2 \), obtained in different laboratories on the same material, will be expected to exceed \( R \), the reproducibility interval, only about 5% of the time. If such a difference is found to be larger than \( R \) there is reason to question one or both results. The value of \( R \) varies proportionally with the magnitude of the transverse rupture strength and is calculated from the following equation:

\[
R = 8900 + (0.122) (x - 41000)
\]

where units of \( R \) and \( x \) are psi.

10.3 The value of \( r \) and \( R \) are assumed to vary linearly over the transverse rupture strength range from 41 500 to 145 000 psi. Transverse rupture strength was measured only at the two end points.

11. Keywords

11.1 flexural strength; 3 Point Bend Test; transverse rupture; transverse rupture strength; TRS

1. Scope

1.1 This test method covers the use of magnetic instruments for the nondestructive measurement of the thickness of an electrodeposited nickel coating on either a magnetic or nonmagnetic substrate.

1.2 These instruments measure either the magnetic attraction between a magnet and the coating-substrate combination, or the reluctance of a magnetic flux path passing through the coating and the basis metal.

1.3 For this test method, there are two types of coating-substrate combinations that can be encountered: Type A, nickel coatings on a magnetic substrate, and Type B, nickel coatings on a nonmagnetic substrate.

1.4 The effective measuring ranges of instruments using the principle of magnetic attraction are up to 50 µm for Type A coatings, and up to 25 µm for Type B coatings. For reluctance gages, the effective ranges are much greater, and measurements up to 1 mm or more, can be made on both types of coatings.

1.5 Measurements made in accordance with this test method will be in compliance with the requirements of ISO Standard 2361 as printed in 1982.

1.6 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ISO International Standard:
ISO 2361 Electrodeposited Nickel Coatings on Magnetic and Nonmagnetic Substrates—Measurement of Coating Thickness—Magnetic Method

3. Significance and Use

3.1 The thickness of a coating is often critical to its performance. This magnetic method is suitable for measuring nondestructively the thickness of some nickel coatings and for specification acceptance.

3.2 This method requires that the magnetic properties of the coating and its substrate be the same as those of the calibration standards.

4. Factors Affecting the Measuring Accuracy

4.1 Coating Thickness—Inherent in the method is a measuring uncertainty that, for thin coatings, is constant and independent of the coating thickness; for thicknesses greater than about 50 µm, this uncertainty is proportional to the coating thickness.

4.2 Magnetic Properties of the Basis Metal (Type A coatings only)—Magnetic thickness measurements are affected by variations in the magnetic properties of the basis metal. For practical purposes, magnetic variations in low-carbon steel can often be considered to be insignificant. To avoid the influences of severe or localized heat treatments and cold working, the instrument should be calibrated using a calibration standard having a basis metal with the same magnetic properties as that of the test specimen or, preferably and if available, with a sample of the part to be tested before application of the coating.

4.3 Basis Metal Thickness (Type A coatings only)—For each instrument, there is a critical thickness of the basis metal above which the measurements will not be affected by an increase in that thickness. Since it depends on the instrument probe (Note 1) and the nature of the basis metal, its value should be determined experimentally, if it is not supplied by the manufacturer.

Note 1—The term “instrument probe” also includes the term “magnet.”

4.4 Edge Effect—The method is sensitive to abrupt changes in the surface contour of the test specimen. Therefore, measurements made too near an edge or inside corner will not be valid, unless the instrument is specially calibrated for such a measurement. This also applies to measurements made on geometrically limited areas, such as narrow conductors on...
4.5 Curvature—Measurements are affected by the curvature of the test specimen. The influence of curvature varies considerably with the make and type of instrument, but always becomes more pronounced as the radius of curvature decreases. Instruments with two-pole probes may also produce different readings, depending on whether the poles are aligned in planes parallel or perpendicular to the axis of a cylindrical surface. A similar effect can occur with a single-pole probe, if the tip is unevenly worn. Measurements made on curved test specimens may not, therefore, be valid unless the instrument is specifically calibrated for such measurements.

4.6 Surface Roughness:

4.6.1 Measurements are influenced by the surface topography of the substrate and the coating, and a rough or scratched surface will give individual instrument readings that all vary from point to point. In this case, it is necessary to make many readings at different positions to obtain an average value that is representative of the mean coating thickness.

4.6.2 If the basis metal is magnetic and rough, it may also be necessary to check the zero of the instrument at several positions on a sample of the uncoated rough substrate.

4.6.3 If the roughness of the substrate surface is small, relative to the coating thickness, its effect will probably be negligible.

4.7 Direction of Mechanical Working of the Basis Metal (Type A coatings only)—Measurements made by an instrument having a two-pole probe or an unevenly worn single-pole probe may be influenced by the direction in which the basis metal has been subjected to mechanical working, such as rolling. The reading may change with the orientation of the probe on the surface.

4.8 Residual Magnetism (Type A coatings only)—Residual magnetism in the basis metal affects the measurements made by instruments that employ a stationary magnetic field. Its influence on measurements made by reluctance instruments employing an alternating magnetic field is much smaller.

4.9 Stray Magnetic Field—Strong stray magnetic fields, such as those produced by various types of electrical equipment, can seriously interfere with the operation of magnetic thickness instruments.

4.10 Foreign Particles—The probes of magnetic instruments of all types must make physical contact with the test surface and are, therefore, sensitive to foreign material that prevents intimate contact between the probe and the coating surface. Both the test surface and the instrument probe should be kept free of foreign material.

4.11 Magnetic Properties of the Coating—Magnetic thickness measurements are affected by variations in the magnetic properties to the coating. These properties depend on the conditions under which the deposit is produced, the type and composition of the coating, and its stress. The magnetic properties of multiple-layer nickel coatings will also depend on the relative thickness of each of the layers.

4.11.1 A heat treatment at 400°C for 30 min will equalize the magnetic permeability of dull (Watts) nickel coatings of the same composition. Bright nickel deposits may or may not have the same magnetic properties after heat treatment.

4.12 Nickel Coatings on the Back of the Substrate (Type B coatings only)—Nickel coatings on the back of the substrate can affect the measurements, depending on the thickness of the substrate.

4.13 Pressure—Instrument readings are sensitive to the pressure with which the probe is applied to the test specimen. No deformation of the coating or probe should be allowed. Errors that sometimes are encountered with the use of manual probes can be avoided by employing spring-loaded probes that exert a relatively constant pressure.

4.14 Probe Orientation—Instrument readings may be sensitive to the orientation of the magnet in relation to the field of gravity of the earth. Thus, the operation of an instrument in a horizontal or upside-down position may require a different calibration, or may be impossible.

5. Calibration of Instruments

5.1 Before use, each instrument shall be calibrated in accordance with the instructions of the manufacturer, employing suitable thickness standards. During use, the calibration shall be checked at regular intervals, at least once a day. Attention shall be given to the factors listed in Section 4 and to the procedures in Section 6.

5.2 The calibration standards shall be coated standards obtained by electroplating nickel adherently onto a substrate. The coating thickness of the calibration standards shall bracket the user’s highest and lowest coating thickness measurement requirement.

5.3 The substrate and the coating of the standard shall have the same magnetic properties as those of the test specimen (see 4.2, 4.3, 4.12 and section 4.12.1).

5.3.1 To assure the similarity of the magnetic properties of the nickel deposit and for Type A coatings the steel substrate, calibration standards shall be produced by another suitable test method, such as cross sectioning or the coulometric test method from a specimen produced under identical conditions as the test specimen to be measured. To confirm the similarity of the magnetic properties of the substrate to those of the standards, a comparison of the readings obtained with the bare basis metal of the standard to that of the test specimen is recommended.

5.3.2 In the same manner, the similarity of the magnetic properties of the coating of the test specimen to that of the standard can be established by verifying with the coulometric or microscopical method that the thickness reading obtained on the test specimen by means of the properly calibrated instrument corresponds to the actual thickness determined by one or both of the above methods.

5.4 Where indicated, the calibration of the instrument should be checked by rotating the probe in increments of 90° (see 4.7 and 4.8).

5.5 For Type A coatings, the basis metal thickness for the test and the calibration shall be the same if the critical thickness, defined in 4.3, is not exceeded. When possible, back up the basis metal of the standard, or the test specimen, with a sufficient thickness of similar material to make the readings independent of the basis metal thickness.

5.6 If the curvature of the coating to be measured is such as to preclude calibration on a flat surface, the curvature of the
coated standard shall be the same as that of the test specimen.

6. Procedure

6.1 Operate each instrument in accordance with the instructions of the manufacturer. Give appropriate attention to the factors listed in Section 4.

6.2 Check the calibration of the instrument at the test site each time the instrument is put into service, and at regular intervals during use, to assure proper performance.

6.3 Observe the following precautions:

6.3.1 Basis Metal Thickness (Type A coatings only)—Check whether the basis metal thickness exceeds the critical thickness. If not, either use the back-up method of 5.5, or make sure that the calibration has been made on a standard having the same thickness and magnetic properties as the test specimen.

6.3.2 Edge Effects—Do not make readings close to an edge, hole, inside corner, etc., of a specimen, unless the validity of the calibration for such a measurement has been demonstrated.

6.3.3 Curvature—Do not make readings on a curved surface of a specimen, unless the validity of the calibration for such a measurement has been demonstrated.

6.3.4 Number of Readings:

6.3.4.1 For each measurement, make at least 3 readings, removing the probe after each reading, and average the readings. If any 2 of the readings differ from each other by more than 5\% of the average reading or 2 \( \mu \)m, whichever is the greater, then the measurement shall be discarded and repeated.

6.3.4.2 The substrate or coating, or both, may be too rough to meet this criterion. In such a case it may be possible to obtain a valid measurement by averaging a number of readings. To be valid under this test method, the validity of such a procedure must be demonstrated (see Appendix X1).

6.3.4.3 Instruments of the attractive force type are sensitive to vibrations, and readings that are obviously erroneous should be rejected.

6.3.5 Direction of Mechanical Working (Type A coatings only)—If the direction of mechanical working has a pronounced effect on the reading, make the measurement on the test specimen with the probe in the same orientation as that used during calibration. If this is impossible, make four measurements at various orientations by rotating the probe in increments of 90°.

6.3.6 Residual Magnetism (Type A coatings only)—When residual magnetism is present in the basis metal, it is necessary, when using instruments employing a stationary magnetic field, to make measurements in two orientations differing by 180°. It may also be necessary to demagnetize the test specimen to get valid results (see 4.7 and 4.8).

6.3.7 Surface Cleanliness—Before making measurements, clean any foreign matter such as dirt, grease, and corrosion products from the surface without removing any coating material. When making measurements avoid any areas having visible defects that are difficult to remove, such as welding or soldering flux, acid spots, dross, or oxide.

6.3.8 Techniques—The readings obtained may depend on the technique of the operator. For example, the pressure applied to a probe or the rate of applying a balancing force to a magnet will vary from one individual to another. Such effects can be reduced or minimized either by having the instrument calibrated by the same operator who will make the measurement, or by using constant pressure probes.

6.3.9 Positioning of the Probe:

6.3.9.1 In general, place the instrument probe perpendicular to the specimen surface at the point of measurement. For some instruments of the attractive force type, this is essential. With some instruments, however, it is desirable to tilt the probe slightly and select the angle of inclination giving the minimum reading.

6.3.9.2 If, on a smooth surface, the readings obtained vary substantially with the angle of inclination, it is probable that the probe is worn and needs to be replaced. If a magnetic instrument is to be used in horizontal or upside-down position, calibrate it for that position.

7. Report

7.1 The report should include the following information:

7.1.1 A reference to this standard,

7.1.2 Type of instrument used,

7.1.3 Size and description of test specimen,

7.1.4 Whether special jigs were used,

7.1.5 Type of calibration standard and the method used,

7.1.6 Thickness of the coating as determined from the measurements,

7.1.7 Operator identification, and

7.1.8 Date.

8. Precision and Bias

8.1 The equipment, its calibration, and its operation shall be such that the coating thickness can be determined with an uncertainty of less than 10\% at 95\% confidence level.

8.2 Although an uncertainty of less than 10\% may be achieved consistently for a great number of applications, the uncertainty may be greater when the coating thickness is less than 25 \( \mu \)m.

8.3 Instruments suitable for compliance with 6.1 are available commercially. For many coating systems, the instruments are capable of making measurements with an uncertainty of less than 5\% at 95\% confidence level.

8.4 The measurement bias is the discrepancy remaining between the measured thickness and the true thickness if all random errors are eliminated. It is, therefore, no greater than, and attributable to (1) the calibration error of the instrument and (2) the quality of the calibration standard used to calibrate the instrument.

8.5 The precision is being determined by round-robin testing.

9. Keywords

9.1 coating thickness; magnetic method; nickel coatings; thickness; thickness testing
X1. MEASUREMENTS ON ROUGH SURFACES

X1.1 Measurements on rough surfaces are subject to random errors associated with the position of the instrument probe relative to the peaks and valleys of the rough surface. These random errors increase with surface roughness, but can be reduced by averaging 10 or more readings.

X1.2 Roughness can also introduce a bias (systematic error) because the probe seldom, if ever, rests at the bottom of a valley; and the magnetic field in the neighborhood of the probe differs from that at a smooth surface. In the case of a rough substrate, the valleys are filled with coating material, but when the instrument is calibrated with a foil, the foil rests on the peaks of the substrate. A bias can be corrected for if the magnitude of the bias can be determined by microscopical or other measurements.
Standard Specification for Appearance of Electroplated Plastic Surfaces

This standard is issued under the fixed designation B 532; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope
1.1 This specification limits the amounts and types of visual defects that can be tolerated on decoratively electroplated surfaces of plastic products. The quantity of defects permitted shall be specified by the purchaser.

2. Referenced Documents
2.1 ASTM Standards:
   B 374 Terminology Relating to Electroplating
   D 883 Terminology Relating to Plastics

3. Terminology
3.1 Definitions—See Terminology B 374 for terms relating to electroplating. For terms not relating to electroplating, see Terminology D 883.

4. Classification of Surfaces
4.1 The appearance of surfaces will be separately evaluated depending on the prominence of the surface under conditions of ordinary use.
   4.1.1 Surface A—The surface that appears most prominent to the observer, or most likely to be noticed at first glance.
   4.1.2 Surface B—The surface (usually adjacent to Surface A) which is somewhat less prominent to the observer.
   4.1.3 Surface C—The surface not readily seen by the observer except at an angle, or by special observation.
   4.1.4 Surface D—The surface not visible to the observer under conditions of normal use.

5. Significance and Use
5.1 Where electroplated plastics are used for decorative purposes, the appearance after plating is important. This standard provides a means of evaluating the appearance of the substrate and the coating.

6. Method of Evaluation
6.1 The part under test shall be evaluated in the same relative position as it will normally be viewed under usual conditions of use.
6.2 After proper positioning, the part under test shall be evaluated at a distance of not less than 600 mm nor more than 900 mm from the observer, using diffused daylight-type fluorescent lighting or 1500 to 1600 lm/m² at the surface of the part being inspected.
6.3 The observer shall have 20/20 vision at a distance of 600 to 900 mm, corrected with eyeglasses if necessary.

7. Appearance Guide
7.1 Table 1 limits the extent to which various defects are acceptable on each of the four surface classifications described in Section 4.
7.2 In Table 1 the term “some” assumes that the defects cited are not outstanding, too numerous, or located too closely together. The specific number of defects permitted, and their spacing, shall be specified by the purchaser. The term “no” indicates that the defect cited is not normally acceptable. The term “yes” indicates that the defect cited is normally acceptable.

8. Keywords
8.1 appearance; electroplated plastic
### TABLE 1 Evaluation of Appearance of Electroplated Plastic Surfaces

<table>
<thead>
<tr>
<th>Defect</th>
<th>Surface</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cold shot*</td>
<td>no</td>
<td>no</td>
<td>some</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Ejection marks*</td>
<td>no</td>
<td>no</td>
<td>yes</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Flash*</td>
<td>no</td>
<td>no</td>
<td>no</td>
<td>no</td>
<td>no</td>
</tr>
<tr>
<td>Gate mark*</td>
<td>no</td>
<td>some</td>
<td>yes</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Parting time*</td>
<td>no</td>
<td>some</td>
<td>yes</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Sink marks*</td>
<td>no</td>
<td>no</td>
<td>yes</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Splay*</td>
<td>no</td>
<td>no</td>
<td>some</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Weld line*</td>
<td>no</td>
<td>some</td>
<td>yes</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Burnt deposit*</td>
<td>no</td>
<td>no</td>
<td>some</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Cracks*</td>
<td>no</td>
<td>no</td>
<td>no</td>
<td>no</td>
<td>no</td>
</tr>
<tr>
<td>Peeling*</td>
<td>no</td>
<td>no</td>
<td>no</td>
<td>no</td>
<td>no</td>
</tr>
<tr>
<td>Pits*</td>
<td>no</td>
<td>no</td>
<td>some</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Roughness*</td>
<td>no</td>
<td>no</td>
<td>some</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Scratches, nicks*</td>
<td>no</td>
<td>some</td>
<td>some</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Staining*</td>
<td>no</td>
<td>no</td>
<td>some</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Voids*</td>
<td>no</td>
<td>no</td>
<td>some</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Blisters†</td>
<td>no</td>
<td>no</td>
<td>no</td>
<td>no</td>
<td>no</td>
</tr>
<tr>
<td>Visible intermediate deposits‡</td>
<td>no</td>
<td>no</td>
<td>no</td>
<td>some</td>
<td>yes</td>
</tr>
<tr>
<td>Off color‡</td>
<td>no</td>
<td>no</td>
<td>some</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Rack mark‡</td>
<td>no</td>
<td>no</td>
<td>no</td>
<td>yes</td>
<td>yes</td>
</tr>
<tr>
<td>Stardusting‡</td>
<td>no</td>
<td>no</td>
<td>some</td>
<td>yes</td>
<td>yes</td>
</tr>
</tbody>
</table>

* These defects arise primarily in the plastic-molding operation.
† Existence of these defects should be noted and resolved prior to tool manufacture. A change of design may be required.
‡ These defects arise in either the plastic-molding or electroplating operation.
§ Blistering, or peeling, or cracks in the gate area or in the electroplating-rack contact area may be acceptable if the given area is covered or otherwise hidden after assembly.
¶ Not removable by wiping or cleaning.
∥ These defects arise primarily in the electroplating operation.
Wifi May occur at rack contacts or low current-density areas.

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Standard Test Method for
Peel Strength of Metal Electroplated Plastics

1. Scope

1.1 This test method gives two procedures for measuring the force required to peel a metallic coating from a plastic substrate. One procedure (Procedure A) utilizes a universal testing machine and yields reproducible measurements that can be used in research and development, in quality control and product acceptance, in the description of material and process characteristics, and in communications. The other procedure (Procedure B) utilizes an indicating force instrument that is less accurate and that is sensitive to operator technique. It is suitable for process control use.

1.2 The tests are performed on standard molded plaques. This method does not cover the testing of production electroplated parts.

1.3 The tests do not necessarily measure the adhesion of a metallic coating to a plastic substrate because in properly prepared test specimens, separation usually occurs in the plastic just beneath the coating-substrate interface rather than at the interface. It does, however, reflect the degree that the process is controlled.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Document

2.1 ASTM Standards:

E 4 Practices for Force Verification of Testing Machines

3. Summary of Test Method

3.1 A properly prepared standard test specimen, called a plaque, is copper electroplated, with no additional metal coating. The coated plaque is either tested as is, or it is conditioned by a low-temperature bake and then tested. The coating is cut through to the plastic substrate in a way that forms two strips of coating (see Fig. 1). Each strip is peeled from the substrate at a right angle using an instrument that indicates the force required to separate it from its substrate.

4. Significance and Use

4.1 The force required to separate a metallic coating from its plastic substrate is determined by the interaction of several factors: the generic type and quality of the plastic molding compound, the molding process, the process used to prepare the substrate for electroplating, and the thickness and mechanical properties of the metallic coating. By holding all others constant, the effect on the peel strength by a change in any one of the above listed factors may be noted. Routine use of the test in a production operation can detect changes in any of the above listed factors.

4.2 The peel test values do not directly correlate to the adhesion of metallic coatings on the actual product.

4.3 When the peel test is used to monitor the coating process, a large number of plaques should be molded at one time from a same batch of molding compound used in the production moldings to minimize the effects on the measurements of variations in the plastic and the molding process.

5. Apparatus

5.1 Procedure A—A tension testing machine that has self-aligning grips and that has a loading range that includes the forces to be measured, 5 to 200 N, shall be used to separate the coating from the substrate, and measure the force required. The machine shall meet the verification requirements of Practices E 4. The speed of separation of the crossarms of the machine shall be adjusted to give a separation rate of 25 ± 3 mm/min. The machine shall automatically and continuously record on a chart the load on one coordinate and the amount of peel on the other coordinate. The amount of peel may be obtained from calculation, using a known chart speed.

5.2 Procedure B—A spring-loaded, force-indicating instrument with a measurement range that includes the force to be

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1 This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.08.03 on Decorative Coatings.


2 This test is also known as the Jacquet Test. A detailed treatment of the test has been published by Saubestre et al in Plateing, Vol 52, 1965, p. 982.

3 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard’s Document Summary page on the ASTM website.

4 The sole source of supply of the Instron universal testing machine known to the committee at this time is Instron Corp., Canton, MA.

5 If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.
measured, 5 to 200 N, shall be used to measure the force required to separate the coating. The indicated force shall be accurate to ±10%.

6. Sampling

6.1 A sampling procedure is not applicable to this test method.

7. Test Specimen

7.1 Perform the test using a flat, molded plastic plaque such as shown in Fig. 1. Plaque dimensions may vary up to ±10% provided that the edges of slit peel strips are not closer than 11 mm from any plaque edge.

8. Conditioning

8.1 Condition the electroplated plaques requiring conditioning by baking for 1 h in an air-circulating oven operated at 70 ± 3°C. Place the plaques in the oven in a way that permits free circulation of air around all surfaces of the plaques. Allow the conditioned plaques to cool to room temperature and then test them within 1 h. Test the plaques that do not require conditioning within 30 to 60 min following the coating process.

Note 1—The separation strength of an unconditioned plaque will change with time. The separation strength of a conditioned plaque may change with time after conditioning.

9. Procedure

9.1 Mold the required number of plaques using the specified molding compound and molding procedure.

Note 2—When the test is used as a control of the coating process, commercially prepared plaques can be used.

9.2 Clean, activate, and electroplate the plaques as specified. The thickness of the metallic coating in the test area (see Fig. 1) shall be 40 ± 4 µm.

9.3 Cut through the coating along the dashed lines shown in Fig. 1, and also along the centerline so as to produce two parallel strips of coating that are 25.00 ± 0.25 mm wide and approximately 75 mm long.

Note 4—Following are methods that can be used to cut the coating:

(a) Slit the coating with a milling machine equipped with a slitting saw. Operate the saw at a peripheral speed of 300 mm/s and a feed of 5 mm/s.

(b) Slit with a device such as shown in Fig. 2.

(c) Cut the coating with a sharp blade guided by a straightedge.

9.4 Using a sharp chisel or knife, peel back an approximately 15-mm tab of one of the strips at the end adjacent to the mold gate. Fiberglas-reinforced adhesive-backed tape may be applied to the two surfaces of the tab to improve the grip on the tab between the jaws of the testing machine.

9.5 Procedure A:

9.5.1 Install a holding fixture on the fixed arm of the testing machine. The fixture shall support the plaque at a right angle to the pull direction of the machine. A suitable fixture is illustrated in Fig. 3.

Note 3—It is necessary to have a ductile copper coating to perform this test. The thickness and uniformity of thickness of the metallic coating directly influence the peel strength; therefore, in order to standardize this test, the coating thickness is specified. If a different coating thickness is used, the results will not be comparable to other test results. It may be necessary to use shielding during plating to obtain the required coating thickness uniformity.


7 The sole source of supply of a Union HSG #14-0.064 slitting saw known to the committee at this time is Union Twist Drill Co., Athol, MA.

8 The sole source of supply of the Specimen Scriber known to the committee at this time is Plateq Corp., New Haven, CT.
9.5.2 Place the plaque in the holding fixture with the tab end facing out. Locate the plaque so that the strip to be tested is centered over the centerline of the fixture.

9.5.3 Clamp the tab in the jaws of the movable crossarm of the testing machine.

9.5.4 Separate the strip from the plaque at a speed of 25 ± 3 mm/min, and continuously record the force.

9.5.5 Repeat the test using the other strip on the plaque.

9.5.6 If a strip tears during separation, discontinue the test. The readings obtained up to the point of tearing may be used if at least 25 mm of the strip were separated before the tear.

9.6 Procedure B:

9.6.1 Restrain the plaque in a way that permits separating the strip.

9.6.2 Attach the tab to the spring-loaded tester.

9.6.3 Separate the strip from the plaque at an angle of 90 ± 5° to the plaque and at a rate of 25 ± 3 mm/min.

9.6.4 During the separation the force on the indicator will rise to peak values and fall back. Record the peak values.

9.6.5 Repeat the test using the other strip on the plaque.

9.6.6 If a strip tears during separation, discontinue the test. The readings obtained up to the point of tearing may be used if at least 25 mm of the strip were peeled before the tear.

9.7 Repeat the peel test with a second plaque.

9.8 Examine the underside of the peeled strip and the bared area of the plaque and determine whether the separation occurred in the surface layers of the plaque (cohesive failure) or at the interface (adhesive failure). Plastic adhering to the underside of the strip is not always apparent to the naked eye, and therefore should be verified microscopically.

9.9 Remove any plastic from the strip by means of a suitable solvent. Acetone, for example, will remove ABS. Measure the thickness of the strip in several locations using a ball micrometer that can be read to the nearest 1 µm. (A micrometer that reads in inches may be used if it can be read to the nearest 0.0001 in., 0.0001 in. = 2.5 µm.)

10. Calculation

10.1 Peel, Procedure A—Calculate the arithmetic mean of the highest and the lowest forces that were recorded for each strip. Calculate the arithmetic mean of the four pull tests (two strips on two plaques). If the testing machine indicates the force in units other than newtons, convert the values to newtons.

10.2 Peel, Procedure B—Calculate the arithmetic mean of the recorded peak values. If the tester indicates the force in units other than newtons, convert the values to newtons.

10.3 Thickness—Calculate the arithmetic mean thickness of each strip. Calculate the arithmetic mean thickness of the four strips (two strips on two plaques).

11. Report

11.1 Report the following information:

11.1.1 Identification of the plastic in the plaque by generic type, manufacturer, and by manufacturer’s grade and lot designations.

11.1.2 The molding conditions used.

11.1.3 The cleaning, activating, and plating processes used.

11.1.4 The procedure (A or B) used.
11.1.5 The mean peel strength of the four tests and the highest and lowest peel values obtained. Report the values in newtons per 25-mm width. Report separately, identifying each, the results obtained with the unconditioned and the conditioned plaques.

11.1.6 The arithmetic mean coating thickness and the maximum and minimum thicknesses. Report the values in micrometres. Report separately, identifying each, the thicknesses for the unconditioned and the conditioned plaques.

11.1.7 Whether the failure was cohesive or adhesive or mixed. Report separately, identifying each, the findings for the unconditioned and the conditioned plaques.

12. Precision and Bias

12.1 The precision and bias of this test method have not yet been determined, but the test is useful in controlling the quality of electroplated plastics.

13. Keywords

13.1 electroplated plastic; peel strength test
Standard Practice for
Rating of Electroplated Panels Subjected to
Atmospheric Exposure

This practice covers a preferred method for evaluating the condition of electroplated test panels that have been exposed to corrosive environments for test purposes. It is based on experience in use of the method with standard 10- by 15-cm (4- by 6-in.) panels exposed on standard ASTM racks at outdoor test sites in natural atmospheres. It has been used also for rating similar panels that have been subjected to accelerated tests such as those covered by Practice B 117, Method B 287, Method B 368, and Method B 380. Any modifications needed to adapt the method to rating actual production parts are not considered in this practice.

1. Scope

1.1 This practice covers a preferred method for evaluating the condition of electroplated test panels that have been exposed to corrosive environments for test purposes. It is based on experience in use of the method with standard 10- by 15-cm (4- by 6-in.) panels exposed on standard ASTM racks at outdoor test sites in natural atmospheres. It has been used also for rating similar panels that have been subjected to accelerated tests such as those covered by Practice B 117, Method B 287, Method B 368, and Method B 380. Any modifications needed to adapt the method to rating actual production parts are not considered in this practice.

1.2 This practice refers only to decorative-protective coatings that are cathodic to the substrate, typified by nickel/chromium or copper/nickel/chromium on steel or zinc die castings. It is not intended for use with anodic sacrificial coatings such as zinc and cadmium on steel.

2. Referenced Documents

2.1 ASTM Standards:
B 117 Practice for Operating Salt Spray (Fog) Apparatus
B 287 Method of Acetic Acid-Salt Spray (Fog) Testing
B 368 Method for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test)
B 380 Method of Corrosion Testing of Decorative Electrodeposited Coatings by the Corrodkote Procedure

3. Basis of Procedure

3.1 The rating method described in this recommended practice is based on the recognition that typical decorative-protective deposits such as nickel/chromium, with or without a copper undercoat, have two functions: (1) to protect the substrate from corrosion and thus prevent degradation of appearance caused by basis metal corrosion products (for example, rust and rust stain); and (2) to itself maintain a satisfactory appearance. Although these functions overlap, they can be evaluated separately and it is frequently desirable to do so. Accordingly, this practice assigns separate ratings to (1) appearance as affected by corrosion of the substrate and (2) appearance as affected by deterioration of the coating itself.

3.2 The rating number assigned to the ability of the coating to protect the substrate from corrosion is called the “protection” number or rating.

3.3 The rating number assigned to the inspector’s judgment of the overall appearance of the panel, including all defects caused by the exposure (Note 1), is called the “appearance” number or rating.

Note 1—Panels that are not “perfect” even before being exposed should normally be rejected (see Note 4).

3.4 The result of inspecting a panel is recorded as two numbers separated by a slash (/), the protection number being given first.

3.5 In addition to recording the numerical rating of a panel, the inspector should note the type(s) and severity of defect(s) contributing to the rating. This may be done by the use of agreed symbols for the most common defects (Appendix X1) and abbreviations for degree or severity of these defects.

4. Types of Defects

4.1 “Protection” defects include crater rusting (Note 2), pinhole rusting, rust stain, blisters (Note 3), and any other defects that involve basis metal corrosion.

Note 2—“Rusting” or “rust” as used in this document includes corrosion products of the substrate and is not confined to iron or steel; the white corrosion products of zinc die castings and aluminum, for example, are included in this term.

Note 3—Blisters on plated zinc die castings usually connote basis metal corrosion; but the inspector’s judgment may be required to decide whether a blister does or does not arise at the substrate-coating interface.

4.2 “Appearance” defects include, the addition to those caused by basis metal corrosion, all defects that detract from the appearance (that is, the commercial acceptability) of the panel. Typical are: surface pits, “crow’s feet,” crack patterns, surface stain, and tarnish.

4.3 Defects developing on exposure that reflect improper preparation or plating should be noted but no attempt should be made to rate panels showing major amounts of such defects. Peeling of the coating from the substrate, or of one coat from
another, is the principal such defect.

5. Preparation for and Manner of Inspection

NOTE 4—It may be desirable to expose panels for test even though they are defective in certain respects before exposure. In that case, an inspection should be made and recorded before the panels are exposed.

5.1 Panels may be inspected on the exposure racks or may be removed to a more suitable location if necessary. Lighting during inspection should be as nearly uniform as possible; direct reflection from sun or clouds should be avoided, and various angles of inspection should be tried to ensure that defects show up.

5.2 If the condition of the panels allows, inspection should be made in the “as-is” condition. If dirt, salt deposits, and so forth, make it impractical to inspect them, panels may be sponged with a mild soap solution followed by water rinse; but no pressure should be exerted in this procedure such as would tend to upgrade the rating by, for example, cleaning off rust or rust stain. Panels should be allowed to dry before inspecting them.

5.3 Defects to be noted and taken into account in rating panels include only those that can be seen with the unaided eye (Note 5) at normal reading distance.

NOTE 5—“Unaided eye” includes wearing of correctional glasses if the inspector normally wears them.

5.3.1 Optical aids may be used to identify or study defects once they are found by unaided eye inspection.

5.4 Edge defects, occurring within 6.5 mm (¼ in.) of the edges of a panel, may be noted in the description but are not counted in arriving at the numerical rating. Similarly contact and rack marks, mounting holes, and so forth, should be disregarded.

5.5 Rubbing, polishing, and so forth, of the surface of the panel may be desirable to study one or another aspect of its condition. Such procedure shall be confined to the minimum area absolutely necessary for the purpose, preferably not more than 1 cm² of a 10- by 15-cm panel.

6. Assignment of Protection Rating

6.1 The numerical rating system is based on the area covered by protection defects, by the following equation:

\[
R = 3 (2 - \log A)
\]

(1)

where \(R\) = rating and \(A\) = percentage of the total area covered by defects. \(R\) is rounded off to the nearest whole number, leading to the tabulation given in Table 1.

<table>
<thead>
<tr>
<th>Area of Defect (in percent)</th>
<th>Rating</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>10</td>
</tr>
<tr>
<td>To 0.1</td>
<td>9</td>
</tr>
<tr>
<td>0.1 to 0.25</td>
<td>8</td>
</tr>
<tr>
<td>0.25 to 0.5</td>
<td>7</td>
</tr>
<tr>
<td>0.5 to 1.0</td>
<td>6</td>
</tr>
<tr>
<td>1.0 to 2.5</td>
<td>5</td>
</tr>
<tr>
<td>2.5 to 5</td>
<td>4</td>
</tr>
<tr>
<td>5 to 10</td>
<td>3</td>
</tr>
<tr>
<td>10 to 25</td>
<td>2</td>
</tr>
<tr>
<td>25 to 50</td>
<td>1</td>
</tr>
<tr>
<td>&gt;50</td>
<td>0</td>
</tr>
</tbody>
</table>

6.1.1 Strict application of the equation given in 6.1 would lead to ratings greater than 10 for panels with extremely small defective areas. Rating 10, accordingly, is arbitrarily assigned to a panel with no defects, and the equation operates at ratings 9 and below.

6.1.2 If desired, fractional ratings between 9 and 10 may be assigned to panels judged better than 9 but not perfect. Fractional ratings below 9, although normally not especially useful, may be assigned if desired.

6.2 As an aid in judging the defective area, standards of comparison, consisting of photographs of panels or of dot charts are made part of this practice. See Appendix X2. These photographs and charts⁶ are 10 by 15 cm (4 by 6 in.) to facilitate comparison with the panel being inspected. The standards represent as nearly as possible the maximum amount of corrosion permissible for a given rating; there is a standard for each rating 1 through 9. A panel worse than the standard for rating 1 would rate 0.

6.2.1 The types of corrosion defects normally encountered differ according to the type of atmospheric exposure. Typical decorative deposits exposed to marine atmospheres often tend to fail by crater rusting, whereas in industrial atmospheres, they are more likely to exhibit pinpoint rusting; and the latter atmosphere also tends to be more severe with regard to degradation of the coating system but somewhat less severe with regard to basis metal corrosion. For this reason, the same standard comparison photographs or charts are not suitable for use at both types of locations; photographs are more helpful in assessing panels exposed to marine atmospheres, whereas dot charts can be used for industrial locations (Appendix X2).

6.3 In rating any given panel, it is recommended that the appropriate series of standards be placed beside it and the basis metal corrosion defects in the panel be matched as nearly as possible with one of the standards. If the panel is somewhat better than standard (X) but not as good as standard (X + 1) it is rated (X); if somewhat worse than standard (X) but not as bad as standard (X − 1) it is rated (X − 1). At the inspector’s option, decimal fractional ratings may be assigned.

6.3.1 If a large group of panels is being inspected at one time, it is recommended that the panels be assessed individually as in 6.3; but when the entire group has been rated, the ratings should be reviewed to make sure that ratings assigned actually reflect the relative merits of the panels. This acts as a check on individual ratings and aids in ensuring that the inspector’s judgment or frame of reference has not changed during the course of the inspection, owing to fatigue, change in lighting conditions, haste to finish the job, or other causes. One method of facilitating this comparison is to remove individual panels from their racks and place them beside other panels. It may be advisable to physically arrange all of the panels in order to merit.

7. Assignment of Appearance Rating

7.1 This recommended practice recognizes that whereas the degree of protection afforded the substrate can be assessed
fairly objectively in accordance with Section 6, the assessment of appearance depends on many subjective factors. Therefore, the appearance rating cannot be assigned with the same degree of precision as can the protection rating.

7.1.1 There are many modes of deterioration in appearance mentioned in 4.2 but this list is not exhaustive, and as new plating systems are developed and introduced to industry, they may well exhibit new types and modes of deterioration.

7.1.2 Unlike the protection rating, the appearance rating is based not only on the area of the defects but also on their severity: the degree to which they would detract from the commercial acceptability of an article of appearance similar to that of the panel.

7.2 The appearance rating is based, in the first instance, on the protection rating. Since corrosion of the substrate also detracts from appearance, the appearance rating can be no higher than the protection rating.

7.2.1 If basis metal corrosion is the only defect, there being no additional defects affecting only the coating, the appearance rating is the same as the protection rating. If there are surface defects not accounted for in the protection rating, the appearance rating will be one or more units lower than the protection rating. This lowering of the appearance rating is referred to in what follows as the “penalty.”

7.3 The inspector must decide, on the basis of best current practice and opinion, whether a surface coating defect is (1) very slightly, (2) slightly (3) moderately, or (4) severely damaging to the acceptability of the appearance. Guidelines are given below, but judgment factors inevitably enter into the decision.

7.3.1 Defects only slightly damaging may include very light surface pitting that detracts little from the reflectivity, light tarnish or stain easily removed by mild cleaning (such as would be given, for instance, in normal car-washing practice), superficial crack patterns typical of some kinds of chromium plate, and so forth. Such defects, to be categorized as “slight,” must not render the finish commercially unacceptable.

7.3.1.1 A penalty of 1 or 2 points (rating numbers) is assessed for appearance defects classified as slight. One point is assessed if the defects can be classified as very slight, two if slight.

7.3.2 Defects moderately damaging include the same types as in 7.3.1 but more severe, so as to render the appearance questionably acceptable from a commercial standpoint. For example, surface pits that begin to detract from reflectivity; tarnish or stain that, although removable, requires more drastic treatment than routine washing.

7.3.2.1 A penalty of 3 or 4 points (rating numbers) is assessed for appearance defects classified as moderate.

7.3.3 Surface defects that render the panel definitely unacceptable in appearance are classified as severe.

7.3.3.1 A penalty of 5 or more points, up to the maximum available, is assessed for severe surface defects.

7.4 The procedure for checking the ratings described in 6.3.1 is of particular importance in assigning appearance ratings, and is strongly recommended.

8. Low-Rated Panels

8.1 The system described in the foregoing should be satisfactory for assessing relatively good panels. Difficulties may be encountered in attempting to rate severely corroded panels. For example, if a panel rates as low as 4 for protection, it may be difficult to assess any additional appearance defects. At the option of the inspector, this difficulty may be handled as follows:

8.1.1 A cutoff point may be chosen below which appearance ratings are deemed to be of no significance. For example, it may be agreed that any panel with a protection rating of 5 is so unacceptable that an appearance rating has no meaning. Such panels may arbitrarily be assigned one of two appearance ratings: (1) if there are no obvious additional surface defects, appearance rating is set equal to protection rating; (2) if there are any surface defects at all, no attempt is made to assess their severity and the appearance rating is 0.

8.1.2 Alternatively, after setting the cutoff point as in 8.1.1, the appearance rating may be disregarded and only a protection rating assigned.

9. Keywords

9.1 atmospheric exposure; corrosion rating; electroplated deposits

Note 6—If fractional rating was used for the protection number, this would result in a fractional appearance rating; in that case the fractional appearance rating may be retained, or rounded off to the nearest whole number, provided, however, that the appearance rating may not be higher than the protection rating.
X1. ABBREVIATIONS DESCRIBING DEFECTS

X1.1 Types of Failure

R = corrosion (rusting) of the basis metal. (Permanent or massive type of basis metal corrosion such as that in pinholes, bare, or flaked areas, or in craters of broken blisters.)
Rs = stain as a result of basis metal corrosion products, such as rust stain, which can be removed readily with a damp cloth or chamois and mild abrasive revealing a sound bright surface.
S = stains or spots other than that of obvious basis metal corrosion products.
Sp = surface pits. Corrosion pits probably not extending through to the basis metal—that is absence of obvious basis metal corrosion products bleeding therefrom.
F = flaking or peeling of deposit.
B = blistering.
C = cracking.
Z = crazing.
W = crow’s feet.

X1.2 Degree or Extent of Pinhole Rusting, Staining, Surface Pitting, Flaking, and So Forth

s = slight amount.
i = intermediate or moderate amount.
x = excessive amount.

X1.3 Description of Blisters

s = less than about 0.5 mm in diameter.
i = about 0.5 to 2.0 mm in diameter.
x = greater than about 2.0 mm in diameter.
vl = 5 or fewer.
f = 5 to 10.
i = 10 to 25.
m = 25 to 50.
ym = over 50.

X1.4 Description of Location of Defects

e = edge.
g = general.

X2. DOT CHARTS AND PHOTOGRAPHS

X2.1 The dot chart standards (Fig. X2.1)\textsuperscript{5,7} are most\textsuperscript{7} useful when assessing the degree of corrosion in industrial locations. The photographs (Fig. X2.2)\textsuperscript{6} are usually more helpful when determining the extent of corrosion in marine atmospheres.

\textsuperscript{7} Permission for reproduction is granted by the Chrysler Corp. for use of these charts, which are a part of the Laboratory Procedure 461-H-79.
FIG. X2.1 Example of Dot Charts

RATING 1  50%

RATING 2  25%

RATING 3  10%

RATING 4  50%

RATING 5  25%

RATING 6  10%

FIG. X2.1 (continued)
FIG. X2.1 (continued)

FIG. X2.2 Example of Corrosion Appearance
FIG. X2.2 (continued)

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.
Standard Specification for Electrodeposited Coatings of Tin

This standard is issued under the fixed designation B 545; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

e1 Note—The warning note in S4.1 was editorially updated in April 2004.

1. Scope

1.1 This specification covers the requirements for electrodeposited (electroplated) coatings of tin applied to metallic articles. Tin coatings are used to provide a low contact-resistance surface, to protect against corrosion (see 1.2), to facilitate soldering, to provide anti-galling properties, and to be a stopoff coating in the nitriding of high-strength steels.

1.2 Some corrosion can be expected from tin coatings exposed outdoors. In normal indoor exposure, tin is protective on iron, steel, nickel, copper, and their alloys. Corrosion can be expected at discontinuities in the coating (such as pores) due to galvanic couples formed between the tin and the underlying metal through the discontinuities, especially in humid atmospheres. Porosity increases as the coating thickness decreases, so that minimum thicknesses must be specified for each application. Parts coated with tin can be assembled safely in contact with iron and steel, tin-coated aluminum, yellow chromated zinc, cadmium, and solder coatings. (See X5.2 for oxidation and corrosion properties.)

1.3 This specification applies to electroplated coatings of not less than 99 % tin (except where deliberately alloyed for special purposes, as stated in X6.3) obtained from any of the available tin electroplating processes (see 4.3).

1.4 This specification does not apply to hot-dipped tin or other non-electrodeposited coating; it also does not apply to mill products. For mill products, refer to Specifications A 623 or A 623M.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards: 2

A 623 Specification for Tin Mill Products, General Requirements
A 623M Specification for Tin Mill Products, General Requirements [Metric]
B 32 Specification for Solder Metal
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 246 Specification for Tinned Hard-Drawn and Medium-Hard-Drawn Copper Wire for Electrical Purposes
B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings
B 320 Practice for Preparation of Iron Castings for Electroplating
B 322 Practice for Cleaning Metals Prior to Electroplating
B 374 Terminology Relating to Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
B 507 Practice for Design of Articles to be Electroplated on Racks
B 542 Terminology Relating to Electrical Contacts and Their Use
B 558 Practice for Preparation of Nickel Alloys for Electroplating
B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
B 568 Test Method for Measurement of Coating Thickness

2 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard’s Document Summary page on the ASTM website.
3. Terminology

3.1 Definitions—Many of the terms used in this specification are defined in Terminology B 374 or B 542.

3.1.1 rack-plating—an electrodeposition process in which articles to be coated are mounted on racks or other fixtures during the process.

3.1.2 significant surface—that portion of the surface of a coated article at which the coating is required to meet all of the requirements of the coating specification for that article; significant surfaces are usually those that are essential to the serviceability or function of the article, or that can be a source of corrosion products or tarnish films that interfere with the function or desirable appearance of the article; significant surfaces shall be indicated on the drawings of the parts or by the provision of suitably marked samples.

3.1.3 undercoating (see 3.1.4)—also called an underplate in the electronics industry.

3.1.4 underplating—application of a metallic coating layer between the basis metal or substrate and the topmost metallic coating or coatings. The thickness of such an undercoating is usually greater than 0.8 µm (30 µin.). This is in contrast to strikes or flashes, whose thicknesses are generally much smaller.

4. Classification

4.1 General—Orders for articles to be plated in accordance with this specification shall specify the service class (4.2) (and underplating, if required), indicating the severity of service required for the coating. Other coatings variations, such as surface appearance type (4.3) or alloy composition (Appendix X6), are optional.

4.2 Service Class:

<table>
<thead>
<tr>
<th>Class</th>
<th>Minimum Thickness</th>
<th>Typical Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>2.5 µm (100 µin.)</td>
<td>Mild service conditions, particularly where the significant surface is shielded from the atmosphere (as in electronic connector housings). To provide corrosion and tarnish resistance where greater thicknesses may be detrimental to the mechanical operation of the product (for example, small electrical spring contacts and relays). Class A is often used for tin coatings that are not to be soldered, but must function as low-resistance electrical contact surfaces.</td>
</tr>
<tr>
<td>B</td>
<td>5 µm (200 µin.)</td>
<td>Mild service conditions with less severe requirements than Class C (below). Applications are as follows: precleaning of solderable basis metals to facilitate the soldering of electrical components; as a surface preparation for protective painting; for antigalling purposes; and as a stop-off in nitriding. Also found on baking pans after refiring.</td>
</tr>
<tr>
<td>C</td>
<td>8 µm (320 µin.), (10 µm (400 µin.) for steel substrates)</td>
<td>Moderate exposure conditions, usually indoors, but more severe than Class B. Examples are electrical hardware (such as cases for relays and coils, transformer cans, screened cages, chassis, frames, and fittings) and for retention of the solderability of solderable articles during storage.</td>
</tr>
<tr>
<td>D</td>
<td>15 µm (600 µin.), (20 µm (800 µin.) for steel substrates)</td>
<td>Severe service, including exposure to dampness and mild corrosion from moderate industrial environments. Examples are fittings for gas meters, automotive accessories (such as air cleaners and oil filters), and in some electronic applications. Very severe service conditions, including elevated temperatures, where underlyng metal diffusion and intermetallic formation processes are accelerated. Thicknesses of 30 to 125 µm (0.001 to 0.005 in.) may be required if the coating is subjected to abrasion or is exposed to slowly corrosive liquids or corrosive atmospheres or gases. Thicker coatings are used for water containers, threaded steel couplings of oil drilling strings, and seacoast atmospheres. Coatings subject to mild etchants are included. Similar to Class A, but for shorter-term contact applications and short shelf-life requirements, subject to purchaser approval.</td>
</tr>
<tr>
<td>E</td>
<td>30 µm (0.0012 in.)</td>
<td>Bright coatings are obtained by heating the matte coating above the melting point of tin for a few seconds, followed by quenching; palm oil and hydrogenated oils and fats are used as heat-transfer medium at a temperature of 260 ± 8°C (500 ± 14°F), but other heating methods also are in use, such as hot air. The maximum thickness for flow-brightening is, in most cases, approximately 8 µm (300 µin.); thicker coatings tend to dewet. The shape of the part is also a factor; flat surfaces dewet more readily than wires or rounded shapes.</td>
</tr>
<tr>
<td>F</td>
<td>1.5 µm (60 µin.)</td>
<td>Examples are fittings for gas meters, automotive accessories (such as air cleaners and oil filters), and in some electronic applications. Very severe service conditions, including elevated temperatures, where underlyng metal diffusion and intermetallic formation processes are accelerated. Thicknesses of 30 to 125 µm (0.001 to 0.005 in.) may be required if the coating is subjected to abrasion or is exposed to slowly corrosive liquids or corrosive atmospheres or gases. Thicker coatings are used for water containers, threaded steel couplings of oil drilling strings, and seacoast atmospheres. Coatings subject to mild etchants are included. Similar to Class A, but for shorter-term contact applications and short shelf-life requirements, subject to purchaser approval.</td>
</tr>
</tbody>
</table>

4.3 Surface Appearance Type (Electroplating Process):

4.3.1 Matte Tin Electrodeposits—Coatings with a matte appearance are obtained from tin plating baths (stannate, sulfate, methylsulfonate, and fluoborate) used without the addition of any brightening agents. However, all matte baths (except for stannate baths) do require the addition of grain refiners, and often of other additives in order to produce the desired matte finish.

4.3.2 Bright Tin Electrodeposits—Bright coatings are obtained when proprietary brightening agents are used in specific bright tin plating baths.

4.3.3 Flow-Brightened Electrodeposits—Flow-brightened coatings are obtained by heating the matte coating above the melting point of tin for a few seconds, followed by quenching; palm oil and hydrogenated oils and fats are used as heat-transfer medium at a temperature of 260 ± 8°C (500 ± 14°F), but other heating methods also are in use, such as hot air. The maximum thickness for flow-brightening is, in most cases, approximately 8 µm (300 µin.); thicker coatings tend to dewet. The shape of the part is also a factor; flat surfaces dewet more readily than wires or rounded shapes.
5. Ordering Information

5.1 In order to make the application of this specification complete, the purchaser must supply the following information to the seller in the purchase order and drawings:

5.1.1 Title, ASTM designation number, and year of issue of this specification;

5.1.2 Deposit by classification (4.1), including thickness or service class (4.2);

5.1.3 Composition and metallurgical condition of the substrate to be coated (6.1);

5.1.4 Additional underplating, if required (6.8);

5.1.5 Surface-appearance type (for example, matte, flow-brightened, or bright), if required (4.3 and 6.2);

5.1.6 Location of significant surfaces (3.1.2);

5.1.7 Hydrogen embrittlement relief, if required (Supplementary Requirement S2); and

5.1.8 Any other items needing agreement (for example, 6.5.2 and 8.5).

6. Coating Requirements

6.1 Substrate—The metal substrate shall be subjected to such surface preparation, cleaning, and electroplating procedures as are necessary to yield deposits with the desired quality.

Note 2—Careful preparation of metal surfaces is necessary in order to assure good adhesion and quality. For suitable methods, see Practices B 183, B 242, B 281, B 320, B 322, and B 558. Also see 6.6.

6.2 Electroplating shall be applied after all basis metal heat treatments and mechanical operations have been completed.

6.3 Appearance—Tin coatings shall have the characteristic appearance, including surface texture (4.3), for the process used. The appearance shall be uniform throughout, insofar as the basis metal will permit. They shall be adherent and visually free of blisters, pits, peeled areas, cracks, nodules, and unplated areas. They shall not be stained or discolored. Flow-brightened coatings shall be free of dewetted areas and beads. All surfaces shall be substantially free of grease or oil used in the flow-brightening process.

6.4 All tin-coated articles shall be clean and undamaged. When necessary, preliminary samples showing the finish shall be supplied to and approved by the purchaser. Where a contact mark is inevitable, its location shall be subject to agreement between the supplier and the purchaser.

6.5 Thickness of Coatings—Tin coatings on articles shall conform to the thickness requirements specified in 4.2 as to the minimum thickness on significant surfaces.

6.5.1 Local Thickness—The thickness values specified in 4.2 are the minimum local thicknesses measured by one or more of the methods given in Practice B 659 at any number of desired spots on the significant surface.

6.5.2 Mean Thickness—When specified by the purchaser, instead of being a local minimum requirement, the thickness requirement can be a minimum (arithmetic) mean thickness.

Note 3—Specification of the coating thickness in terms of the mean is normally made when the coated articles are small and relatively simple, such as connector pins and terminals.

Note 4—Thickness of electrodeposited coatings varies from point to point on the surfaces of a product (see Practice B 807). The thickness is less in interior corners and holes. Such surfaces are normally exempt from the thickness requirement. If the full thickness is required on these surfaces, the electroplater will have to use special techniques that probably will increase the cost of the process.

Note 5—When articles are plated by mass plating techniques (such as barrel plating), such measurement methods as “strip and weigh” or “weigh before and after plating” may be used to determine the mean thickness.

6.6 Adhesion—Adhesion of the coating shall be tested by one of the methods given in Appendix X2. The coating should adhere to the basis metal when subjected to the agreed test; flaking or blistering of the coating is to be taken as evidence of unsatisfactory adhesion.

6.7 Integrity of the Coating:

6.7.1 Gross Defects/Mechanical Damage—Coatings shall be free of visible mechanical damage and similar gross defects when viewed at up to 4× magnification. For some applications, this requirement may be relaxed to allow for a small number of such defects (per unit area), especially if they are outside of or on the periphery of significant surfaces (also see 6.7.2).

6.7.2 Porosity—Almost all as-plated electrodeposits contain some porosity. The amount of porosity in the coating that may be tolerable depends on the severity of the environment that the article is likely to encounter during service or storage. If the pores are few in number, or away from significant surfaces, their presence can often be tolerated. Such acceptance (or pass-fail) criteria, if required, should be part of the product specification for the particular article or coating requiring the porosity test. See 8.5 for porosity testing.

6.8 Underplating:

6.8.1 For tin coatings in Class A and Class F (4.2) that will not be exposed to solder temperatures (especially those that must function as electrically conductive surfaces), a nickel underplate or undercoating of at least 1.3 µm (50 µin.) shall be applied before tin plating.

6.8.2 To prevent zinc migration and impairment of solderability during service or storage, substrates of brass or other copper alloys containing more than 5% zinc must have a copper undercoating of at least 2.5 µm (100 µin.), or a nickel undercoating of at least 1.3 µm (50 µin.), prior to tin plating. A thicker coating of nickel may be required in some situations for additional retardation.

6.9 Hydrogen Embrittlement Relief—High-tensile strength steels and severely cold-worked steels are susceptible to embrittlement by hydrogen in both cleaning and electroplating operations. See Supplementary Requirements S1 and S2 for details.

7. Sampling

7.1 The sampling plan used for inspection of a quantity of the coated articles shall be as agreed upon between the purchaser and the supplier.
NOTE 6—The procedure for sampling is accomplished by selecting a relatively small number of the finished articles at random. These articles (the inspection lots) are inspected and classified as complying or not complying with the requirements of the specification. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Three standards, Test Method B 602, Guide B 697, and Test Method B 762, contain sampling plans that are designed for the sampling inspection of coatings. Test Method B 602 contains four sampling plans, three for use with tests that are non-destructive and one for use when they are destructive. The buyer and seller may agree on the plan to be used. If they do not, Test Method B 602 identifies the plan to be used.

Guide B 697 provides a large number of plans and also provides guidance in the selection of a plan. When Guide B 697 is specified, the buyer and seller must agree on the plan to be used.

Test Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numerical value, and certain statistical requirements must be met. Test Method B 762 contains several plans and also provides instructions for calculating plans to meet special needs. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 762 identifies the plan to be used.

7.2 An inspection lot shall be defined as a collection of coated articles that meet the following requirements: they are of the same kind; have been produced to the same specifications; have been coated by a single supplier at one time, or at approximately the same time, under essentially identical conditions; and are submitted for acceptance or rejection as a group.

7.3 Special Test Specimens—It may be preferable to use special test specimens to represent product in process control or in acceptance inspection when, for example, destructive tests are used and it is desirable not to destroy product or if the test specimen is better adapted to the test. The use of special test specimens, their number, the material from which they are made, their size and shape, and the conditions of their coating shall be as agreed upon by the purchaser and the seller.

8. Test Methods

8.1 Deposit Purity—Atomic absorption or energy dispersion spectrophotometry, or any other methods with a demonstrated uncertainty of less than 10 % of the component measured, may be used to determine impurities. Initial scanning should be conducted for all elements in order to detect any unknown or unexpected impurities. Determine deposit purity by subtracting the total impurities from 100 %.

NOTE 7—Deposit purity is best determined on samples of the actual product (see Section 7). If special test specimens are used (7.3), care must be taken to arrange the specimens so as to electroplate them under the same conditions as typical production pieces.

8.2 Thickness:

8.2.1 Standard Thickness—The coating thickness shall be measured at locations on significant surfaces by one of the following test methods: Test Methods B 487, B 499 (magnetic substrates only), B 504, B 567, and B 568. Practice B 659 may be consulted to determine the most appropriate test method.

8.2.1.1 When Methods B 504 and B 568 are used with tin platings that have been alloyed with more than 0.5 % of lead, bismuth, or antimony to reduce whiskering (see X6.3.2.5), the standard shall have the same composition as the coating. When Test Method B 567 is used to measure these types of coatings, the measuring instrument shall be calibrated with thickness standards that have the same substrate and same composition of coating as the product.

8.2.2 Mean Thickness—If the entire surface of the article is a significant surface, the mean thickness can also be determined by the method described in Appendix X1. Also see Note 5.

8.3 Adhesion—Adhesion of the coating shall be tested by one of the recommended methods of Practice B 571 (see Appendix X2).

8.4 Solderability—For coatings that must be solderable, the method by which solderability is tested shall be specified. Three test methods are described in Appendix X3, while a simple dip test is given in Test Method B 678. The purchaser shall specify whether the tested articles are to receive an artificial aging treatment, such as that given in Test Method B 678, so as to demonstrate whether the articles may be expected to retain their solderability during long storage periods.

8.5 Porosity and Gross Defects Testing:

8.5.1 Coatings on articles of steel (or iron) having a local thickness of 10 µ (0.4 mil) or greater should be subjected to the test given in Appendix X5.

8.5.2 For coatings on articles made from copper or copper alloy as the basis metal, the following tests should be used:

8.5.2.1 To determine mechanical damage or gross defects only, subject samples to the sodium polysulfide immersion test outlined in Specification B 246. Black spots or lines are evidence of mechanical damage or gross defects.

8.5.2.2 To determine all porosity that penetrates down to the copper substrate, especially for coatings in Service Class A, the humid sulfur vapor test (Test Method B 809) shall be used. Blackening at pores will be evidence of porosity.

9. Rejection and Rehearing

9.1 Articles that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the seller promptly and in writing. In cases of rejection, the seller may make a claim for a rehearing. Product that shows coating imperfections in subsequent manufacturing operations may be rejected.

10. Keywords

10.1 electrodeposited tin; electroplated tin; tin; tin coatings
SUPPLEMENTARY REQUIREMENTS

The following supplementary requirements shall apply only when specified by the purchaser in the contract or order.

**S1. Pretreatment of Iron and Steel for Reducing the Risk of Hydrogen Embrittlement**

S1.1 Parts for critical applications that are made of steels with ultimate tensile strengths of 1000 MPa, hardness of 31 HRC or greater, that have been machined, ground, cold formed, or cold straightened subsequent to heat treatment, shall require stress relief treatment when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specifications B 849 (heat treatment) and B851 (shot peening) may be consulted for a list of pretreatments that are used widely.

**S2. Post Coating Treatments of Iron and Steel for Reducing the Risk of Hydrogen Embrittlement**

S2.1 Parts for critical applications that are made of steels with ultimate tensile strengths of 1000 MPa, hardness of 31 HRC or greater, as well as surface hardened parts, shall require post coating hydrogen embrittlement relief baking when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specifications B 850 may be consulted for a list of post treatments that are used widely.

**S3. Solderability Requirements**

S3.1 If the coating must be solderable, refer to 8.4 for test methods for determining solderability.

**S4. Government Packaging Requirements**

S4.1 Parts plated for the U.S. government and military, including subcontractors, shall be packaged according to Practice D 3951. (Warning—Some contemporary packaging materials may emit fumes that are deleterious to the surface of the coating.)

**APPENDIXES**

(Nonmandatory Information)

**X1. DETERMINATION OF MEAN COATING THICKNESS OF SMALL ARTICLES BY THE WEIGHT LOSS METHOD**

X1.1 Select at random from the production lot the number of coated articles specified by the purchaser. The number of articles shall be sufficient to have a total weight of coating of at least 0.2 g. Calculate the coated area in mm² to an accuracy of at least 1 part in 100.

X1.2 Clean the articles by, for example, washing in a solvent or water-detergent followed by a clean water rinse. Dry and weigh with an accuracy of at least 1 part in 1000.

X1.3 Immerse the articles in a solution of 20 g antimony trioxide in 1 L of concentrated hydrochloric acid (sp gr 1.19) and allow them to remain for 1 min after gas evolution stops. Remove the articles, rinse them in clean water, wipe and brush as necessary to remove smut (antimony residues), and dry. Reweigh.

X1.4 Calculate the mean thickness in µm by dividing the loss in weight of the articles by the area and multiplying the quotient by 1.37 \times 10^5.

**X2. ADHESION TESTS**

X2.1 *Burnishing Test*—Rub an area of not more than 630 mm² (1 in.²) of the electroplated surface, selected at the discretion of the inspector, rapidly and firmly for 15 s with a smooth metal implement. A suitable burnishing implement is a copper or steel disk used edgewise and broadside. Maintain pressure sufficient to burnish the film at every stroke, but not so great as to cut the deposit. Poor adhesion will be shown by the appearance of a loose blister that grows as rubbing is continued. If the quality of the deposit is also poor, the blister may crack, and the plating will peel away from the basis metal.

X2.2 *Quenching Test*—Heat the electroplated article in an oven for a sufficient time to reach 150 ± 10°C (300 ± 20°F), and then quench in water at room temperature. Poor adhesion will be shown if the coating blisters, cracks, or peels.

X2.3 *Reflow Test*—Parts may be evaluated by reflowing in a bath of palm oil at a temperature of 235 to 260°C (455 to 500°F) until the plating melts. A bright coating completely covering the significant surfaces will indicate acceptable quality.

X2.4 *Bend Test*—A sample shall be bent, with the coated surface away, over a mandrel until its two ends are parallel. The mandrel shall have a diameter equal to the thickness of the sample. Examination of 4× magnification should show no evidence of peeling or cracking. Some tin coatings, especially of bright tin, may show fine cracks during this test, but if these do not separate from the surface, it should not be considered an adhesional failure.
X.3. SOLDERABILITY TESTS

X3.1 General:

X3.1.1 Methods for testing the solderability of tin-coated articles are based on measurement of the extent of wetting by molten solder or determination of the minimum time required to produce full or perfect wetting by the solder (1, 2).

X3.1.2 The extent of wetting can be observed by simple immersion in solder under controlled conditions, or by performing dip tests with automated equipment. Visual examination, time of wetting, measurement of area of spread, and calculation of spread values are used to assess solderability (1, 2).

X3.1.3 The minimum wetting time is determined by carrying a specimen in a fixture through a standing wave of solder at a controlled speed and measuring the time of immersion required to achieve complete wetting.

X3.2 Simple Dip Test—The test in Test Method B 678 is recommended.

X3.3 Wetting Balance (1, 3, 4):

X3.3.1 The wetting balance provides the most objective, operator-independent, and reproducible measure of the solderability of a surface of currently known methods. Numerous models are available under different names such as the surface-tension balance, and various trade names, such as “meniscograph,” but all share certain common features.

X3.3.2 The base structure holds a heated solder bath that can be raised or lowered at a specific rate. The test piece is held in a clamp that extends from an arm directly over the solder bath. The clamp is attached to a load cell and to a transducer, which transmits a signal to a converter that adjusts the signal to a useful mode (analog or digital) for the recording instrument, computer, or strip-chart recorder.

X3.3.3 The parameters of the test are set on the instrument, according to the manufacturer’s instructions, to reveal the most information concerning the solderability of the test piece.

X3.3.4 The bath temperature must be held within precise limits at a suitable temperature with respect to the solder alloy. The immersion rate, which may vary from 1 to 25 mm/s, must be constant from sample to sample.

X3.3.5 The immersed surface area of the standards and samples should be similar, as well as the depth of immersion and the type and weight of flux on the test piece.

X3.3.6 The opposing forces of buoyancy and wetting, versus time, which is transmitted from the transducer to the strip-chart recorder or the computer during the test, is plotted.

X3.3.7 The most significant information obtained from the graph are the wetting time, rate of wetting, total wetting force, and whether the wetting force remains constant over the time of the test.

X3.3.8 Test samples are to be run against a series of standards that have been run to establish averages and to define precision windows. The goal is to define minimum acceptable solderability in terms of a maximum wetting time, minimum wetting rate, minimum force, and stable wetting at a set dwell time.

X4. SOME DEFINITIONS OF WETTING TERMINOLOGY

X4.1 dewetting—a condition that results when molten metal has coated a surface and then receded, leaving irregularly shaped mounds of metal separated by areas covered with a thin metal film; basis metal is not exposed.

X4.2 nonwetting—a condition whereby a surface has contacted molten metal, but the metal has not adhered to all of the surfaces; basis metal remains exposed.

X4.3 wetting—the formation of a relatively uniform, smooth, unbroken, and adherent film of the metal coating to a basis metal.

X5. SULFUR DIOXIDE POROSITY TEST (STEEL SUBSTRATES)

X5.1 Principle—Exposure to a moist atmosphere containing a low concentration of sulfur dioxide causes spots of substrate corrosion product to appear at discontinuities in the coating. If the sulfur dioxide concentration in the atmosphere is too high, the corrosion product formed is too fluid to permit easy observation of pore sites. The method provided, which depends on the production of sulfur dioxide from the reaction between sodium thiosulfate and sulfuric acid within the test chamber, ensures suitable conditions for the development of immobile corrosion products at discontinuities.

X5.2 Apparatus:

X5.2.1 The test cabinet shall be a chamber fitted with a lid or door, and should preferably be made of glass or a transparent plastic material. The size should be sufficient to accommodate the test specimens with their lowest part at least 75 mm (3 in.) above the surface of a solution occupying at least 1/30 of the total capacity.

X5.2.2 The closure of the vessel and other joints should be gas tight but need not be capable of resisting pressure. A glass plate makes an adequate joint on the lubricated ground edges of a glass tank.
X5.2.3 The cabinet should be of uniform cross section, and the solution placed in it should cover the base completely.

X5.2.4 The specimens under testing should be supported by a glass or plastic stand inside the cabinet. The significant surfaces may be inclined at any angle, but it may be desirable to choose the same method of support for similar articles.

X5.3 Corrosive Medium—The corrosive medium should be moist air containing sulfur dioxide prepared by adding 1 part by volume of 0.1 N sulfuric acid to 4 parts of a solution containing 10 g of sodium thiosulfate crystals in 1 L of water.

X5.4 Temperature of Test—Conduct the test at 20 ± 5°C (60 ± 9°F), taking precautions against rapid temperature fluctuation in the course of the test.

X5.5 Procedure:

X5.5.1 Before the test, clean the specimens with an organic solvent (for example, trichloroethane), wipe with a lint-free cloth, and allow to attain room temperature. Introduce into the test cabinet a volume of aqueous sodium thiosulfate solution equal to 1/50 of the volume of the cabinet. Suspend the test specimens above this solution on nonmetallic supports, with the surfaces of the specimens not less than 25 mm (1 in.) apart, not less than 25 mm (1 in.) from any wall of the cabinet, and not less than 75 mm (3 in.) from the surface of the sodium thiosulfate solution. Add to the sodium thiosulfate solution a volume of 0.1 N sulfuric acid equal to a quarter of the volume of the thiosulfate solution and seal the cabinet, keeping it shielded from draughts or other causes of rapid temperature fall. Addition of the sulfuric acid may be made before the test specimens are placed in position, provided that the cabinet is closed within 5 min of addition of the acid.

X5.5.2 Leave the specimens in the closed vessel for 24 h. After removing the specimens from the corrosive atmosphere, allow them to dry without wiping or cleaning in any way, and then examine them using the options outlined in Guide B 765, Sections 6 and 7.

X6. DESIGN CONSIDERATIONS

X6.1 General—Properties of electrodeposited tin coatings are affected to various degrees by their service and storage environments (4.2). These should be taken into account when designing for special applications.

X6.2 Temperature and Other Environmental Effects:

X6.2.1 Diffusion and Intermetallic Formation—Interdiffusion between tin coatings and copper or copper alloys does occur. The diffusion is slow at room temperature and rapid at elevated temperatures. Evidence of diffusion is the formation of a layer of copper-tin intermetallic at the interface and diffusion of a layer of zinc to the surface if the substrate is brass. Diffusion may lead to darkening of a thin coating and impairment of its solderability characteristics, particularly after long storage. With such thin coatings, a diffusion barrier of nickel may be advantageous, although users should also consider the use of thicker coatings when solderability must be maintained over a period of years (6.8). An underplating of nickel or copper must be used as a diffusion barrier on brass.

X6.2.2 Tin Phase Transformation—Tin coatings, like pure metal, may be subject to allotropic transformation at low temperatures (also called tin pest or disease). Where electrodeposited tin coatings are subject to long-term storage or use at very low temperatures, it may be advisable, when specified by the purchaser, to codeposit small amounts (<1 %) of bismuth, antimony, or lead with the tin. These alloying additions, particularly the first, have been shown to inhibit the transformation.

X6.2.3 Tin Oxidation (5, 6)—In clean, dry atmospheres at room temperature, tin is covered quickly with a very thin, but continuous, air-formed oxide film. Although the tin surface will remain bright for long periods in the absence of moisture, this invisible surface oxide layer is not truly protective. It gradually thickens, and it may eventually produce a dull gray appearance.

X6.2.4 Oxides and Solderability—Tin oxide films are not as easily reduced as copper oxide. However, the non-hydrated films are not an impediment to soldering, because as soon as a break is made in the oxide, the molten solder can interact with the layer of free tin below the oxide film. The mechanical forces of the molten solder and tin flowing together break up the oxide layer. There needs to be sufficient free tin below the oxide layer in order for this to occur, because the intermetallic compounds formed from tin and the basis metal will not react in this way (7).

X6.3 Whisker Growth:

X6.3.1 Sometimes metal filaments, usually called whiskers, grow spontaneously from the surface of electrodeposited metals (for example, tin, cadmium, and zinc) within a period after plating that may vary from weeks to months to years. These whiskers frequently are approximately 2.5 μm (0.0001 in.) in diameter, but they can grow to 10 mm (⅜ in.) or longer.
(25-mm whiskers have been recorded) and can have a current carrying capacity of as much as 10 mA. In many applications for tin coatings, whisker growth has not been a problem, but there are critical applications (X6.3.2) in which it may create a problem.

X6.3.2 Whiskers are particularly undesirable when associated with miniaturized low-voltage apparatus where narrow spacing exists between adjacent components that must remain electrically isolated to avoid short circuits. In this specialized application, the occurrence of whisker growth is sufficiently frequent to be an obstacle to the use of tin. Therefore, in designing for low-voltage electronic equipment where components are closely spaced, tin coatings should either not be used, or whisker formation should be inhibited or prevented by the application of prevention methods. Practically all of the following methods take into account the chief cause of tin whiskering, which is the presence of stresses in the tin coating (8):

- X6.3.2.1 Flow brightening (1) or annealing the tin plating to reduce the internal stresses.
- X6.3.2.2 Plating the tin to sufficient thickness to minimize stress-producing epitaxial effects.
- X6.3.2.3 Use of compatible underplatings, especially nickel.
- X6.3.2.4 Avoiding high compressive loads and stresses while making joints or connections.
- X6.3.2.5 Codepositing lead, bismuth, antimony, copper, or nickel with the tin reduces the risk of whisker growth (9). A tin-lead plating that is used extensively for the prevention of whiskering has a nominal lead composition of 7 % (with allowed variations of ±5 %) by mass, the balance being tin. However, some of these platings cannot be used in contact with food.

REFERENCES

(1) For example, Wassink, R. J., Klein, Soldering in Electronics, Electrochemical Publications, Ltd., Ayr, Scotland, 1989.
1. Scope

1.1 This guide covers the use of the dropping test to measure the thickness of electrodeposited zinc, cadmium, copper, and tin coatings.

Note 1—Under most circumstances this method of measuring coating thicknesses is not as reliable or as convenient to use as an appropriate coating thickness gage (see Test Methods B 499, B 504, and B 568).

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section


B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method

B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry

D 1193 Specification for Reagent Water

3. Summary of Guide

3.1 A dropping test consists of applying dropwise a corrosive solution, at a constant rate, to the electroplated surface, and measuring the time required to penetrate the coating and expose the substrate; this time is proportional to the coating thickness.

4. Significance and Use

4.1 The thickness of a metal coating is often critical to its performance.

4.2 This procedure is useful for an approximate determination when the best possible accuracy is not required. For more reliable determinations, the following methods are available: Test Methods B 487, B 499, B 504, and B 568.

4.3 This test assumes that the rate of dissolution of the coating by the corrosive reagent under the specified conditions is always the same.

5. Factors Affecting the Accuracy

5.1 The following factors will affect the accuracy of a coating thickness measurement made by this method:

5.1.1 Cleanliness of Surface—Any foreign material on the surface to be tested, including lacquer, grease, corrosion products, and conversion coatings, will interfere with the test and must be removed. Tarnish and conversion coatings can often be removed by mild burnishing with a soft, clean pencil eraser.

5.1.2 Concentration of Test Solution—Variation from the stated concentrations will introduce an error unless the thickness factor is adjusted accordingly. Test solutions cannot be reused.

5.1.3 Temperature—The thickness factor for a given solution, being a function of the temperature, must be known for the temperature at which the test is made. Specimens to be tested should be allowed to reach room temperature before testing.

5.1.4 Dropping Rate—The thickness factor is also a function of the dropping rate. The thickness factors given in Section 10 are for a dropping rate of 100 drops/min and will remain valid for the range of 95 to 105 drops/min.

5.1.5 Solution Drainage—The thickness factors given in Section 10 are valid only if the test solution is rapidly drained off, as from a flat surface inclined 45° from the horizontal. The test cannot be made on a horizontal surface or at a location that does not permit rapid drainage.

5.1.6 Drop Size—Variation in drop size may alter the penetration rate. The tip of the dropping apparatus should conform to the dimensions given in Fig. 1 and should be kept clean.

5.1.7 Alloy Layer—The presence of an alloy layer at the
coating-substrate interface may obscure the end point and introduce an uncertainty as to the amount of alloy included in the thickness measurement.

5.1.8 Detection of End Point—The end point (the time at which the coating has been penetrated and the substrate exposed) is characterized by a change in the appearance of the surface on which drops are falling. This is a matter of personal judgment and may not always be clearly defined, depending upon the coating-substrate combination and the extent of alloying, if any. Such errors may be minimized by standardizing the test method with identical specimens with a known coating thickness determined by other means.

5.1.9 Composition of Coating—The dropping tests described in this document are intended for use on nominally “pure” coatings. The thickness factors listed in Section 10 are expected to vary with gross variations in the composition of the coating, as might result from codeposition of an alloying component. The specific effects, on the thickness factors, of impurities or of inclusions from brighteners or other addition agents can be appreciable. Uncertainty from these sources may be minimized by standardizing the test method against standards, prepared from the same type of plating solution, the thicknesses of which have been determined by other means.

6. Apparatus

6.1 Fig. 1 illustrates one form of apparatus used for dropping tests. Equivalent apparatus are commercially available, as is a solenoid operated unit with a digital readout.

7. Reagents and Materials

7.1 Purity of Reagents—Reagent grade chemicals shall be used in the preparation of all test solutions.

7.2 Purity of Water—Water used in the preparation of test solutions shall be reagent water as defined in Specification D 1193.

8. Test Solutions

8.1 Solution for Cadmium and Zinc:

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chromic acid (CrO₃)</td>
<td>200 g/L</td>
</tr>
<tr>
<td>Sulfuric acid (96 % H₂SO₄, sp gr 1.84)</td>
<td>50 g/L or 27 mL/L</td>
</tr>
</tbody>
</table>

**Note:** Sulfuric acid is added slowly with stirring to at least 20 times its volume of water.

8.2 Solution for Cadmium only:

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonium nitrate (NH₄NO₃)</td>
<td>110 g/L</td>
</tr>
<tr>
<td>Hydrochloric acid (37 % HCl, sp gr 1.19)</td>
<td>10 mL/L</td>
</tr>
</tbody>
</table>

8.3 Solution for Zinc only:

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonium nitrate (NH₄NO₃)</td>
<td>100 g/L</td>
</tr>
<tr>
<td>Nitric acid (70 % HNO₃, sp gr 1.42)</td>
<td>55 mL/L</td>
</tr>
</tbody>
</table>

8.4 Solution for Tin:

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trichloroacetic acid (CHCl₃COOH)</td>
<td>100 g/L</td>
</tr>
</tbody>
</table>

8.5 Solution for Copper:

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrochloric acid (37 % HCl sp gr 1.19)</td>
<td>200 mL/L</td>
</tr>
<tr>
<td>Antimony trioxide (Sb₂O₃)</td>
<td>20 g/L</td>
</tr>
<tr>
<td>Glacial acetic acid (99.7 % CH₃COOH)</td>
<td>250 mL/L</td>
</tr>
<tr>
<td>Ferric chloride (FeCl₃·6H₂O)</td>
<td>450 g/L</td>
</tr>
</tbody>
</table>

**Note:** For ease of preparation, dissolve the antimony trioxide in the hydrochloric acid, add 150 mL of water and the glacial acetic acid, and then add the ferric chloride in small portions. Finally dilute to 1.00 L with water.

8.6 Variations of ±2 % in the composition of a test solution are permissible.

9. Procedure

9.1 Clean the specimen and allow it and the test solution to reach room temperature, which should be recorded. Support the specimen so that the surface to be tested makes a 45° angle with the horizontal, about 13 mm below the tip of the dropping apparatus. Drop the test solution onto the specimen at a rate of 100 drops/min. Note and record the time required for the
solution to penetrate the coating at the point on which the drops fall.

10. Computation of Thickness

10.1 Multiply the time required to penetrate the coating by an appropriate thickness factor to obtain the coating thickness.

10.2 Below are the thickness factors, applicable to nominally "pure" coatings (5.1.9), for the solutions given in Section 8. They are valid for a dropping rate of 100 drops/min from a capillary tip, the dimensions of which are given in Fig. 1.

<table>
<thead>
<tr>
<th>Material</th>
<th>Factor (µm/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cadmium (chromic acid)</td>
<td>0.35</td>
</tr>
<tr>
<td>Zinc (chromic acid)</td>
<td>0.26</td>
</tr>
<tr>
<td>Cadmium (nitrate)</td>
<td>0.25</td>
</tr>
<tr>
<td>Zinc (nitrate)</td>
<td>0.25</td>
</tr>
<tr>
<td>Tin</td>
<td>0.25</td>
</tr>
<tr>
<td>Copper</td>
<td>0.13</td>
</tr>
</tbody>
</table>

10.2.1 The first two thickness factors for cadmium and zinc and the chromic acid solution are only valid for a temperature of 24°C; the factors for these metals and this test solution at other temperatures are given in Fig. 2 and Fig. 3.

FIG. 2 Chart for Determining Thickness of Zinc Coating by the Chromic Acid Dropping Test
10.2.2 The last four thickness factors are valid for temperatures in a range from 20 to 30°C.

11. Reproducibility and Accuracy

11.1 The reproducibility for a single operator is estimated to be ±2 drops (equivalent to 1.2 s) or ±5 %, whichever is greater.

11.2 The over-all accuracy may be substantially poorer than the reproducibility, because of the uncertainties discussed in Section 5, and will depend upon the control exercised over the variables. For maximum accuracy, the test method should be standardized with specimens identical to those being tested, having known coating thicknesses determined by other methods. Sometimes a chemical can be added to the dropping solution to aid in determining the end point, for example, potassium ferricyanide (K₃Fe(CN)₆) has been added to the test solution for zinc in 8.3.
Standard Guide for Measurement of Thin Chromium Coatings by Spot Test

This standard is issued under the fixed designation B 556; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers the use of the spot test for the measurement of thicknesses of electrodeposited chromium coatings over nickel and stainless steel with an accuracy of about ±20% (Section 9). It is applicable to thicknesses up to 1.2 µm.\(^2\)

NOTE 1—Although this test can be used for coating thicknesses up to 1.2 µm, there is evidence that the results obtained by this method are high at thicknesses greater than 0.5 µm.\(^3\) In addition, for coating thicknesses above 0.5 µm, it is advisable to use a double drop of acid to prevent depletion of the test solution before completion of the test.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method\(^4\)
- B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry\(^4\)
- B 588 Test Method for Measurement of Thickness of Transparent or Opaque Coatings by Double-Beam Interference Microscope Technique\(^4\)

3. Summary of Guide

3.1 A drop of hydrochloric acid (test solution) is deposited on the surface of the test specimen, and the time required for the hydrochloric acid to penetrate through the chromium coating (penetration time) is measured. The coating thickness is proportional to this time.

4. Significance and Use

4.1 The thickness of a decorative chromium coating is often critical to its performance.

4.2 This procedure is useful for an approximate determination when the best possible accuracy is not required. For more reliable determinations, the following methods are available: Methods B 504, B 568, and B 588.

4.3 This test assumes that the rate of dissolution of the chromium by the hydrochloric acid under the specified conditions is always the same.

5. Test Solutions

5.1 For chromium on nickel the test solution is reagent grade hydrochloric acid having a specific gravity at 16°C of 1.180 ± 0.002. (This corresponds to 11.5 N ± 0.2 N, which may be checked by titration.) For chromium on stainless steel the test solution is 20 g/L of antimony trioxide dissolved in reagent grade hydrochloric acid having a specific gravity at 16°C of 1.160.

NOTE 2—As received, reagent grade hydrochloric acid is normally more concentrated than 11.5 N.

6. Preparation of Test Area

6.1 The test area must be free of foreign material. Clean by rubbing the test area with a paste of magnesium oxide, rinse it, and dry it with a clean cloth or filter paper. Draw a ring with a diameter of about 6 mm on the test area with melted paraffin or with a wax pencil.

7. Procedure

7.1 Let the test specimen, the test solution, and the dropper stand long enough to reach room temperature, which should be between 16 and 25°C. Temperatures up to 30°C are permissible, but the measurements become less reliable at the higher temperatures because of increasing sensitivity to temperature. Thin test specimens should be set on a heavy metal plate to avoid a rapid change in temperature which could be produced in such specimens by the heat of reaction.

7.2 To determine the penetration time, \(t\), deposit a drop (0.03 to 0.05 mL) of the test solution inside the ring of wax, and measure the time between the beginning of the gas formation and the first appearance of nickel. Make this...
measurement to the nearest \( \frac{1}{2} \) s with a stop watch. If the reaction, that is, the formation of gas bubbles, does not start immediately, it can be started by touching the test surface within the ring with a fine nickel wire. The end point of the penetration is characterized by the cessation of the gassing and the appearance of the yellow color of the nickel surface. There may be an uncertainty of 2 or 3 s in determining when the end point has been reached.

7.3 If the basis metal is 18-8 stainless steel, gassing will stop at the end point; if it is 17% chromium stainless steel, the rate of gas liberation will decrease when the end point is reached.

7.4 When using the antimony trioxide-hydrochloric acid solution, the absence of a black film will indicate that there is no chromium deposit; if a black film forms, but if there is little or no gassing, the chromium is estimated to be less than 0.1 \( \mu \text{m} \) thick.

7.5 Measure the room temperature near the test area to an accuracy of 0.2°C.

8. Calculation of Thickness

8.1 Calculate the thickness of the chromium coating by the equation:

\[
\text{Thickness} = ut
\]

where:

- \( t \) = penetration time, and
- \( u \) = speed of dissolution of the chromium coating as a function of the test temperature (see Fig. 1).

**Note 3**—Fig. 1 is applicable when chromium plate is deposited from the conventional sulfate chromic acid solution under conventional operating conditions. If these procedures are varied, it may be necessary to restandardize the test.

---

**FIG. 1** Temperature Factor for Hydrochloric Acid (sp gr 1.180) and Antimony Trioxide (20 g/L) in Hydrochloric Acid (sp gr 1.160)
8.2 In case of duplex chromium coatings, the thickness will correspond to the combined thickness of the two coatings.

9. Precision and Bias

9.1 This thickness determination has an uncertainty of about ±20%, and is, therefore, much less accurate than the coulo-
1. Scope

1.1 This practice is intended to serve as a guide for producing adherent electrodeposits of nickel on nickel alloys. Only those methods that are well known and generally practiced are included. Methods that have been used successfully but not on a broad scale are not included. Once nickel is applied, other metals may be electroplated on the product.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Cleaning

2.1 The following cleaning treatments may be used. The choice of the procedure will be governed largely by the condition of the surface.

2.1.1 Degreasing is used to remove the bulk of grease, oil, and finishing compounds that may be present on the surface. The cleaning may be vapor degreasing, solvent wash, emulsion and the type of alloy. A mild etching treatment should be used on polished surfaces if a highly finished surface is required.

2.1.2 Electrolytic Alkaline Cleaning—Removal of final traces of dirt, grease, and oil is accomplished best with electrolytic alkaline cleaning. The solution may be either a proprietary cleaner or a formulated one.

3. Activation

3.1 The procedure used for activating the nickel alloy surface usually determines the soundness of the adhesion. The choice of treatment is governed by the condition of the surface and the type of alloy. A mild etching treatment should be used on polished surfaces if a highly finished surface is required. Too mild a treatment may result in a sacrifice of maximum adhesion.

3.2 Anodic-Cathodic Sulfuric Acid—A 25 mass % sulfuric acid solution, containing 166 mL of concentrated, 93 mass % sulfuric acid (density 1.83 g/mL) diluted to 1 L is used for this etching treatment in which the alloy is first etched anodically at a low-current density of 2 A/dm² for 10 min and then made passive at 20 A/dm² for 2 min and finally cathodic for 2 or 3 s at 20 A/dm². (Warning—Slowly add the sulfuric acid with rapid stirring to the approximate amount of water required.) When the initial mixture cools, dilute to exact volume. The temperature of the solution should be in the range from 20 to 25°C (70 to 80°F). Chemical lead may be used for the electrodes. Rinsing should be used before electroplating.

3.3 Acid-Nickel Chloride Treatment—This procedure uses an anodic treatment followed by a cathodic treatment in a low-pH nickel chloride solution. The composition of the solution is 240 g/L of nickel chloride (NiCl₂·6H₂O) and 31 mL of concentrated 31.45 mass % hydrochloric acid (density 1.16 g/mL). The normal procedure is to make the alloy anodic for 2 min at 3 A/dm² and then cathodic for 6 min at the same current density. The temperature of the solution should be in the range from 20 to 25°C. Nickel may be used for the electrodes. Separate tanks are recommended for the anodic and cathodic steps to avoid contamination of solution but a single tank may be used. Rinsing should be used before electroplating except where indicated in Table X1.1.

4. Alloys

4.1 Recommended activating treatments for specific nickel alloys are listed in the Appendix, Table X1.1.
# APPENDIX

(Nonmandatory Information)

## X1. RECOMMENDED ACTIVATION TREATMENTS FOR NICKEL ALLOYS

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Activation</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Permanickel 300</td>
<td>3.2</td>
<td>...</td>
</tr>
<tr>
<td>Duranickel 301</td>
<td>3.2</td>
<td>...</td>
</tr>
<tr>
<td>Incoloy 801</td>
<td>3.3</td>
<td>...</td>
</tr>
<tr>
<td>Incoloy 901</td>
<td>3.5</td>
<td>no rinsing before electroplating</td>
</tr>
<tr>
<td>Inconel 600</td>
<td>3.3</td>
<td>...</td>
</tr>
<tr>
<td>Inconel 722</td>
<td>3.3</td>
<td>anodic step only 15 s</td>
</tr>
<tr>
<td>Inconel 750</td>
<td>3.3</td>
<td>...</td>
</tr>
<tr>
<td>Invar Regular</td>
<td>3.2</td>
<td>3 min after onset of passivity</td>
</tr>
<tr>
<td>Invar 36</td>
<td>3.3</td>
<td>...</td>
</tr>
<tr>
<td>42 % nickel-iron</td>
<td>3.2</td>
<td>3 min after onset of passivity</td>
</tr>
<tr>
<td>46 to 50 % nickel-iron</td>
<td>3.2</td>
<td>3 min after onset of passivity</td>
</tr>
<tr>
<td>Monel 400</td>
<td>3.2</td>
<td>5 min after onset of passivity</td>
</tr>
<tr>
<td>Monel K-500</td>
<td>3.2</td>
<td>5 min after onset of passivity</td>
</tr>
<tr>
<td>Monel R-405</td>
<td>3.2</td>
<td>5 min after onset of passivity</td>
</tr>
<tr>
<td>Monel 501</td>
<td>3.2</td>
<td>5 min after onset of passivity</td>
</tr>
<tr>
<td>Nickel 205</td>
<td>3.4</td>
<td>...</td>
</tr>
<tr>
<td>Nickel 211</td>
<td>3.4</td>
<td>...</td>
</tr>
<tr>
<td>Nimonic 75</td>
<td>3.3</td>
<td>...</td>
</tr>
<tr>
<td>Nimonic 80A</td>
<td>3.3</td>
<td>...</td>
</tr>
<tr>
<td>Ni-Span C902</td>
<td>3.2</td>
<td>...</td>
</tr>
<tr>
<td>Ni-Resist Type 1</td>
<td>3.2</td>
<td>...</td>
</tr>
<tr>
<td>Ni-Resist Type 2–5</td>
<td>3.2</td>
<td>...</td>
</tr>
</tbody>
</table>

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Standard Test Method for Measurement of Coating Thickness by the Beta Backscatter Method

This standard is issued under the fixed designation B 567; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the beta backscatter gages for the nondestructive measurement of metallic and nonmetallic coatings on both metallic and nonmetallic substrate materials.

1.2 The test method measures the mass of coating per unit area, which can also be expressed in linear thickness units provided that the density of the coating is known.

1.3 The test method is applicable only if the atomic numbers or equivalent atomic numbers of the coating and substrate differ by an appropriate amount (see 7.2).

1.4 Beta backscatter instruments employ a number of different radioactive isotopes. Although the activities of these isotopes are normally very low, they can present a hazard if handled incorrectly. This standard does not purport to address the safety issues and the proper handling of radioactive materials. It is the responsibility of the user to comply with applicable State and Federal regulations concerning the handling and use of radioactive material. Some States require licensing and registration of the radioactive isotopes.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 International standard:
ISO 3543: Metallic and Nonmetallic Coatings—Measurement of Thickness—Beta Backscatter Method

3. Terminology

3.1 Descriptions of Terms:

3.1.1 activity—the nuclei of all radioisotopes are unstable and tend to change into a stable condition by spontaneously emitting energy or particles, or both. This process is known as radioactive decay. The total number of disintegrations during a suitably small interval of time divided by that interval of time is called “activity.” Therefore, in beta backscatter measurements, a higher activity corresponds to a greater emission of beta particles. The activity of a radioactive element used in beta backscatter gages is generally expressed in microcuries (1 µCi = 3.7 × 10⁻¹⁰ disintegrations per second).

3.1.2 aperture—the opening of the mask abutting the test specimen. It determines the size of the area on which the coating thickness is measured. This mask is also referred to as a platen, an aperture plate, a specimen support, or a specimen mask.

3.1.3 backscatter—when beta particles pass through matter, they collide with atoms. Among other things, this interaction will change their direction and reduce their speed. If the deflections are such that the beta particle leaves the body of matter from the same surface at which it entered, the beta particle is said to be backscattered.

3.1.4 backscatter coefficient—the backscatter coefficient of a body, is the ratio of the number of beta particles backscattered to that entering the body. is independent of the activity of the isotope and of the measuring time.

3.1.5 backscatter count:

3.1.5.1 absolute backscatter count—the absolute backscatter count, , is the number of beta particles that are backscattered during a finite interval of time and displayed by the instrument. will, therefore, depend on the activity of the source, the measuring time, the geometric configuration of the measuring system, and the properties of the detector, as well as the coating thickness and the atomic numbers of the coating and substrate materials. , is the count produced by the uncoated substrate, and , that of the coating material. To obtain these values, it is necessary that both these materials are available with a thickness greater than the saturation thickness (see 3.1.12).

3.1.5.2 normalized backscatter—the normalized backscatter, , is a quantity that is independent of the activity of the source, the measuring time, and the properties of the detector. The normalized backscatter is defined by the equation:

\[ x_n = \frac{X - X_o}{X_s - X_o} \]
where:

\[ X_0 = \text{count from the substrate}, \]
\[ X_s = \text{count from the coating material}, \]
\[ X = \text{count from the coated specimen}, \]

and each count is for the same interval of time.

Because \( X \) is always \( X_0 \) and \( \leq X_s \), \( x_n \) can only take values between 0 and 1. (For reasons of simplicity, it is often advantageous to express the normalized count as a percentage by multiplying \( x_n \) by 100.)

3.1.5.3 normalized backscatter curve—the curve obtained by plotting the coating thickness as a function of \( x_n \).

3.1.6 beta particles—beta particles or beta rays are high-speed electrons that are emitted from the nuclei of materials undergoing a nuclear transformation. These materials are called beta-emitting isotopes, beta-emitting sources, or beta emitters.

3.1.7 coating thickness—in this test method, coating thickness refers to mass per unit area as well as geometrical thickness.

3.1.8 dead time or resolving time—Geiger-Müller tubes used for counting beta particles have characteristic recovery times that depend on their construction and the count rate. After reading a pulse, the counter is unresponsive to successive pulses until a time interval equal to or greater than its dead time has elapsed.

3.1.9 energy—it is possible to classify beta emitters by the maximum energy of the particles that they release during their disintegration. This energy is generally given in megarontons, MeV.

3.1.10 equivalent (or apparent) atomic number—the equivalent atomic number of an alloy or compound is the atomic number of an element that has the same backscatter coefficient as the material.

3.1.11 half-life, radioactive—for a single radioactive decay process, the time required for the activity to decrease by half.

3.1.12 saturation thickness—the minimum thickness of a material that produces a backscatter that is not changed when the thickness is increased. (See also Appendix X1.)

3.1.13 sealed source or isotope—a radioactive source sealed in a container or having a bonded cover, the container or cover being strong enough to prevent contact with and dispersion of the radioactive material under the conditions of use and wear for which it was designed.

3.1.14 source geometry—the spatial arrangement of the source, the aperture, and the detector with respect to each other.

4. Summary of Test Method

4.1 When beta particles impinge upon a material, a certain portion of them is backscattered. This backscatter is essentially a function of the atomic number of the material.

4.2 If the body has a surface coating and if the atomic numbers of the substrate and of the coating material are sufficiently different, the intensity of the backscatter will be between two limits: the backscatter intensity of the substrate and that of the coating. Thus, with proper instrumentation and if suitably displayed, the intensity of the backscatter can be used for the measurement of mass per unit area of the coating, which, if the density remains the same, is directly proportional to the thickness.

4.3 The curve expressing coating thickness (mass per unit area) versus beta backscatter intensity is continuous and can be subdivided into three distinct regions, as shown in Fig. 1. The normalized count rate, \( x_n \), is plotted on the X-axis, and the logarithm of the coating thickness, on the Y-axis. In the range \( 0 \leq x_n \leq 0.35 \), the relationship is essentially linear. In the range \( 0.35 \leq x_n \leq 0.85 \), the curve is nearly logarithmic; this means that, when drawn on semilogarithmic graph paper, as in Fig. 1, the curve approximates a straight line. In the range \( 0.85 \leq x_n \leq 1 \), the relationship is nearly hyperbolic.

4.4 Radiation other than the beta rays are emitted or backscattered by the coating or substrate, and may be included in the backscatter measurements. Whenever the term backscatter is used in this method, it is to be assumed that reference is made to the total radiation measured.

FIG. 1 Normalized Backscatter
5. Significance and Use

5.1 The thickness or mass per unit area of a coating is often critical to its performance.

5.2 For some coating-substrate combinations, the beta backscatter method is a reliable method for measuring the coating nondestructively.

5.3 The test method is suitable for thickness specification acceptance if the mass per unit area is specified. It is not suitable for specification acceptance if the coating thickness is specified and the density of the coating material can vary or is not known.

6. Instrumentation

6.1 In general, a beta backscatter instrument will comprise: (1) a radiation source (isotope) emitting primarily beta particles having energies appropriate to the coating thickness to be measured (see Appendix X2), (2) a probe or measuring system with a range of apertures that limit the beta particles to the area of the test specimen on which the coating thickness is to be measured, and containing a detector capable of counting the number of backscattered particles (for example, a Geiger-Müller counter (or tube)), and (3) a readout instrument where the intensity of the backscatter is displayed. The display, in the form of a meter reading or a digital readout can be: (a) proportional to the count, (b) the normalized count, or (c) the coating thickness expressed either in thickness or mass per unit area units.

7. Factors Affecting the Measuring Accuracy

7.1 Counting Statistics:

7.1.1 Radioactive disintegration takes place randomly. Thus, during a fixed time interval, the number of beta particles backscattered will not always be the same. This gives rise to statistical errors inherent to radiation counting. In consequence, an estimate of the counting rate based on a short counting interval (for example, 5 s) may be appreciably different from an estimate based on a longer counting interval, particularly if the counting rate is low. To reduce the statistical error to an acceptable level, it is necessary to use a counting interval long enough to accumulate a sufficient number of counts.

7.1.2 At large total counts, the standard deviation ($\sigma$) will closely approximate the square root of the total count, that is $\sigma = \sqrt{\bar{X}}$; in 95% of all cases, the true count will be within $X \pm 2\sigma$. To judge the significance of the precision, it is often helpful to express the standard deviation as a percentage of the count, that is, $100\sqrt{\bar{X}/X}$, or $100/\sqrt{X}$. Thus, a count of 100 000 will give a value ten times more precise than that obtained with a count of 1000. Whenever possible, a counting interval should be chosen that will provide a total count of at least 10 000, which corresponds to a statistical error of 1% for the count rate. It should be noted, however, that a 1% error in the count rate can correspond to a much larger percentage error in the thickness measurement, the relative error depending on the atomic number spread or ratio between coating and substrate materials.

7.1.3 Direct-reading instruments are also subject to these statistical random errors. However, if these instruments do not permit the display of the actual counting rate or the standard deviation, the only way to determine the measuring precision is to make a large number of measurements at the same coated location on the same coated specimen, and calculate the standard deviation by conventional means.

Note 1—The accuracy of a thickness measurement by beta backscatter is generally poorer than the precision described in 6.1, inasmuch as it also depends on other factors that are described below. Methods to determine the random errors of thickness measurements before an actual measurement are available from some manufacturers.

7.2 Coating and Substrate Materials—Because the backscatter intensity depends on the atomic numbers of the substrate and the coating, the repeatability of the measurement will depend to a large degree on the difference between these atomic numbers; thus, with the same measuring parameters, the greater this difference, the more precise the measurement will be. As a rule of thumb, for most applications, the difference in atomic numbers should be at least 5. For materials with atomic numbers below 20, the difference may be reduced to 25% of the higher atomic number; for materials with atomic numbers above 50, the difference should be at least 10% of the higher atomic number. Most plastics and related organic materials (for example, photoresists) may be assumed to have an equivalent atomic number close to 6. (Appendix X3 gives atomic numbers of commonly used coating and substrate materials.)

7.3 Aperture:

7.3.1 Despite the collimated nature of the sources used in commercial backscatter instruments, the backscatter recorded by the detector is, nearly always, the sum of the backscatter produced by the test specimen exposed through the aperture and that of the aperture plate(n). It is, therefore, desirable to use a material with a low atomic number for the construction of the plate and to select the largest aperture possible. Measuring errors will be increased if the edges of the aperture opening are worn or damaged, or if the test specimen does not properly contact these edges.

7.3.2 Because the measuring area on the test specimen has to be constant to prevent the introduction of another variable, namely the geometrical dimensions of the test specimen, it is essential that the aperture be smaller than the coated area of the surface on which the measurement is made.

7.4 Coating Thickness:

7.4.1 In the logarithmic range, the relative measuring error is nearly constant and has its smallest value.

7.4.2 In the linear range, the absolute measuring error, expressed in mass per unit area or thickness, is nearly constant, which means that as the coating thickness decreases, the relative measuring error increases. At or near $x_n = 0.35$, the relative errors of the linear and logarithmic ranges are about the same. Thus, the relative error at this point may, for most practical purposes, be used to calculate the absolute error over the linear range.

7.4.3 In the hyperbolic range, the measuring error is always large because a small variation in the intensity of the beta backscatter will produce a large variation in the measured coating thickness.

7.4.4 For instruments that indicate only backscatter count rate and not thickness directly, the count rate is normally converted to a thickness by means of an appropriate graph.
Such graphs are generally valid only within a specific range of coating thicknesses so that extrapolation of a linear range calibration curve (straight line on rectangular coordinates) into the logarithmic thickness range will result in measurement errors. Similarly, extrapolation of a logarithmic range calibration into the linear thickness range will also produce significant errors. Many instruments that indicate coating thickness directly are limited to the combined linear and logarithmic coating thickness ranges but will be in error if measurements are attempted in the hyperbolic thickness range. The instrument manufacturer’s instructions must be followed relative to the limiting coating thicknesses beyond which the particular instrument being used may give substantial errors.

7.5 Resolving Time of the Detector—Because of the dead time of Geiger-Müller tubes (see 3.1.8), the number of pulses displayed by the readout instrument is always less than the actual number of backscattered beta particles. Normally, this does not diminish the measuring accuracy significantly unless the count rate is so high as to saturate the detector.

7.6 Source Geometry—The greatest measurement precision is obtained with the source placed in a particular position with respect to the test specimen. This position depends on the collimation of the beam of beta particles from the source and the location, form, and size of the aperture. If possible, most of the beta particles emitted by the source should be backscattered from the test specimen, and not from the aperture plate(n). The instructions furnished by the manufacturer of the instrument for mounting the source shall be followed exactly.

7.7 Curvature—This test method is sensitive to the curvature of the test specimen. However, the normalized backscatter curve remains nearly the same if the surface of the test specimen does not protrude into the aperture of the platen by more than about 50 µm. By the use of specially selected aperture platens or masks where the isotope is premounted in a fixed, optimum position, it is possible to obtain nearly identical readings on both flat and curved specimens. This permits the use of flat calibration standards for the measurement of curved specimens. The relationship between maximum aperture size and specimen surface curvature is peculiar, in most cases, to the individual instrument design. These details are therefore best obtained from the manufacturer’s data.

7.8 Substrate Thickness:
7.8.1 Test Specimens with Single-Layer Coatings:
7.8.1.1 This test method is sensitive to the thickness of thin substrates, but for each isotope and material there is a critical thickness, called “saturation thickness,” beyond which the measurement will no longer be affected by an increase of the substrate thickness. This thickness depends on the energy of the isotope and on the density of material. If the saturation thickness is not supplied by the manufacturer, it should be determined experimentally.

7.8.1.2 If the substrate thickness is less than the saturation thickness, effective saturation thickness can sometimes be obtained by backing up the substrate with more of the same material, but only if the substrate is not coated on both sides. If the substrate is of constant thickness, the instrument may be calibrated for that thickness of substrate. However, if the substrate thickness is less than the saturation thickness and also varies in thickness, this method will not yield a single value for the coating thickness, but a range of values with an upper and lower limit.

7.8.2 Test Specimens with Multiple-Layer Coatings:
7.8.2.1 If the intermediate layer adjacent to the coating is thicker than the saturation thickness, this test method will not be affected by any variations in the substrate thickness as long as the instrument is calibrated with standards having the intermediate coating material as the basis material.

7.8.2.2 If the thickness of the intermediate layer is less than saturation thickness, but constant in thickness, the instrument may be calibrated for that particular combination of materials. However, if the thickness of this intermediate layer is less than saturation thickness and varies in thickness, this method will not yield a single value for the coating thickness, but a range of values with an upper and lower limit.

7.9 Surface Cleanliness—Foreign material, such as dirt, grease, and corrosion products, will produce erroneous readings. Natural oxide coatings, which form on some metal coatings, also tend to produce low readings, especially if the measurement requires the use of an isotope having an energy of less than 0.25 MeV.

7.10 Substrate Material—To obtain accurate thickness readings, it is necessary that the backscatter produced by the substrate materials of the test specimen and that of the calibration standard be the same. If they are different, other calibration standards will have to be used, or appropriate corrections made. Beta backscatter instruments are available that can automatically make these corrections.

7.11 Density of Coating Material—The beta backscatter method is basically a method of comparing the mass per unit area of the coating of the test specimen to that of the calibration standard. If the instrument readout is in units of mass per unit area, the linear thickness is obtained by dividing by the coating density:

\[ T = \frac{M \times 10}{D} \]

If the instrument readout is in linear units and if there is a difference between the coating densities of the calibration standards and of the test specimens, a density correction must be applied:

\[ T = \frac{T^* \times D^*}{D} \]

where:
\[ T = \text{linear thickness of coating of test specimen, } \mu\text{m}, \]
\[ T^* = \text{linear thickness readout of instrument, } \mu\text{m}, \]
\[ D = \text{density of coating of test specimen, } g/cm^3, \]
\[ D^* = \text{density of coating of calibration standard, } g/cm^3, \] and
\[ M = \text{mass per unit area of coating of test specimen, } mg/cm^2. \]

In addition to porosity, voids, and inclusions, codeposited materials can influence the density of the coating. For most metallic elements the effects are usually considered negligible for deposits obtained under normal conditions from properly maintained electroplating baths free of contamination. The only documented exception is gold, the density of which is dependent on the deposition process.
7.12 Composition of Coating—Because the composition of a coating affects the mass of coating per unit area, it will also affect the instrument response (amount of backscattered beta radiation). This effect may be negligible with alloying elements having densities close to each other, such as cobalt-nickel alloys. Very small quantities of alloying elements, such as those present in high gold alloy deposits, also have little effect.

7.13 Energy of Beta Particles:

7.13.1 Because the precision of the measurement is not constant over the entire range of measurement for a given isotope, but is the best in the logarithmic portion of the normalized beta backscatter curve (see Fig. 1), the isotope should, whenever possible, be selected in such a manner that the expected measurements fall into the range 0.35 ≤ \( x_n \) ≤ 0.85 of the normalized curve. See Appendix X2 for a list of isotopes used with beta backscatter gages.

7.13.2 In general, instructions for selecting the proper isotope are furnished by the manufacturer.

7.14 Measurement Time—Too short a measurement time will yield a poor measurement precision. The selection of the measurement time will, therefore, depend on the measurement precision that is required. Each time the measurement time is increased by a factor of \( n \), the counting measurement precision will improve by a factor of approximately \( 1/\sqrt{n} \).

7.15 Activity of Radioactive Source—The count rate is dependent on the activity of the source. An old source may have a low activity, requiring excessive time to make a good measurement (see 6.1). As a practical guide, the source should be replaced before its half-life has elapsed.

7.16 Coating-Substrate Combination—The measurement precision depends on the difference between the atomic numbers of the coating and substrate materials. The greater this difference, the better the precision (see also 7.2).

7.17 Surface Roughness—Measurement accuracy can be significantly influenced by the roughness of the coating surface, but the effect is minimized if the energy of the beta particles is high and the atomic number of the coating is low.

7.18 Detector—Errors can be introduced by erratic operation of the detector. If instability or drift is suspected, the user is advised to consult the manufacturer.

7.19 Wear of Calibration Standards:

7.19.1 Coating thickness standards used to calibrate beta backscatter instruments are subject to wear when used and thus to a decrease in thickness.

7.19.2 The thickness of a calibration standard should be checked from time to time by comparing it with another calibration standard or reference sample that has not been used since the last check.

8. Calibration of Instruments

8.1 Beta backscatter instruments shall be calibrated with standards before measurements are made and also each time the measuring conditions are changed. To obtain the best possible measurement precision, the largest possible aperture suitable for the area to be measured should be selected. Select the calibration measuring time, the number of calibration measurements to be made on each calibration standard, and the test piece measuring time in accordance with the manufacturer’s instructions to obtain the required measurement precision (see 8.8) with the measuring time, aperture, isotope, and number of readings to be used for measuring the test piece. For certain types of measurement application, this may require unusually long measuring times (greater than 80 s).

NOTE 2—A measurement is that value obtained under the same conditions of time, aperture, isotope, and number of readings as used to measure the test piece. It may be a single reading or an average of two or more readings.

Before use, the calibration shall be checked as described in 8.8. During use, the calibration shall have been checked within the preceding 4 h as recommended by the instrument manufacturer. Attention shall be given to the factors listed in Section 7 and the procedures in Section 10.

8.2 In addition to the zero point, the complete calibration curve can be defined either by two points of the logarithmic range, or by a single point, if the slope in the logarithmic range is known. In the first case, two calibration standards are required, in the second, only one.

8.3 The instrument shall be calibrated with standards having a uniform coating thickness. Whenever possible, these standards shall have an accuracy of ±5 % at a 95 % confidence interval, or better. The coating and substrate materials of the standard should have the same or equivalent atomic numbers as the substrate and coating materials of the test specimen. Standards corresponding to the bare substrate material and the coating material are also considered to be “calibration” standards. Sometimes it is also possible to use foils of the coating material for calibration. These are placed on, and in contact with, the substrate. It is necessary that the foil be clean, smooth, and uniform in thickness, and that the contact between foil and substrate be intimate.

8.4 Before an instrument is calibrated, the condition of the calibration standards shall have been checked. Scratched, worn, or pitted standards shall not be used to calibrate the instrument.

8.5 If coating materials have the same or equivalent atomic numbers, but different densities, the normalized backscatter curves will be essentially parallel in the logarithmic region. Under these circumstances, thickness measurements must be corrected for the difference in densities (see also 7.11). If “equivalent” calibration standards are used for the calibration of the instrument, their suitability shall be established prior to the measurements.

8.6 The substrate thickness for the test specimen and the calibration standards should be the same, unless the saturation thickness as defined in 7.8.1 is exceeded. If they are different, appropriate corrections have to be made (see 7.10).

8.7 The curvature of the calibration standard and of the test specimen shall be the same, except if it can be demonstrated that the readings from a flat or curved specimen are essentially the same. If this is not possible, the readings will have to be corrected.

8.8 Before use, the calibration should be checked as follows. Take 5 measurements on each calibration standard, removing and replacing the standard after each reading, under the same conditions of time, aperture, isotope, and number of readings as the conditions to be used for measuring the test piece. The average of each set of five measurements shall be
within 3 % of the stated thickness of the corresponding calibration standard. Failure to meet these requirements invalidates the calibration.

NOTE 3—Failure to meet these requirements may indicate faulty (worn) calibration standards, a worn aperture platen, improper standard positioning on the aperture platen, insufficient measuring time, or improper compensation for isotope decay.

9. Referee Test

9.1 If a referee test is required to resolve a disagreement, it shall be performed by using “suitable” Standard Reference Material (SRM) thickness standards from the National Institute of Standards and Technology (NIST), if such standards are available.

9.2 A “suitable” SRM standard is an SRM standard of the same substrate/coating combination for which the beta backscatter instrument was calibrated and the thickness of which is within the range of the calibration, preferably close to that of the test items being measured.

9.3 The SRM shall be measured five times, each measurement being made under the same conditions as used for the measurement of the test piece. If the average of the five measurements of the SRM differs from the certified mass per unit area or equivalent thickness by more than 10 %, the calibration is not valid.

NOTE 4—SRMs are issued by the NIST and include “coating thickness” SRMs for some coating systems. The stated mass per unit area of each “coating thickness” SRM is certified to be within 5 % of the true mass per unit area.

10. Procedure

10.1 Operate each instrument in accordance with the manufacturer’s instructions, paying attention to the factors listed in Section 6. Calibrate the instrument in accordance with Section 8.

10.2 Check the calibration of the instrument at the test site each time the instrument is put into service and at frequent intervals during use in accordance with 8.1.

10.3 Precautions—Observe the following precautions:

10.3.1 Substrate Thickness—The substrate thickness shall exceed the saturation thickness or the calibration shall be made with a substrate having the same thickness and properties as the test specimen (see 7.8).

10.3.2 Measuring Aperture—The size of the measuring aperture depends on the size and shape of the test specimen. Follow the manufacturer’s recommendations concerning the choice of a measuring aperture. The measuring aperture shall not be larger than the coated area available on the test specimen. The test specimen shall be seated firmly and securely against the measuring opening.

10.3.3 Curved Specimens—It shall be verified that the aperture used for the measurement is correct for the radius of curvature of the test specimen and, if the calibration has not been made with standards having the same curvature as the test specimen, that the calibration is applicable to the measurement.

10.3.4 Substrate Material—The backscatter produced by the substrate of the standard shall be the same as that produced by the test specimen. Verify this by actual tests. If the two differ, follow the manufacturer’s instructions regarding corrections or use new standards that agree with the test specimen (see 7.10).

10.3.5 Surface Cleanliness—Remove all foreign materials, such as dirt, grease, lacquer, oxides, and conversion coatings, from the surface before the measurement by cleaning without removing any coating material. Avoid measuring specimen areas having visible defects, such as flux and acid spots.

10.3.6 Measuring Time—Use a sufficient measuring time to obtain a repeatability of readings that will yield the desired precision.

11. Report

11.1 The report shall include the following information:

11.1.1 Type of instrument used,
11.1.2 Size of aperture,
11.1.3 Measurement time,
11.1.4 Description of test specimen and measurement area,
11.1.5 If applicable a statement that a correction for density was made,
11.1.6 Type of calibration standards and the measurement mode used,
11.1.7 Thickness of coating as determined from the measurements,
11.1.8 Statistical measurement parameters of the measurement series as required.
11.1.9 Identification of testing facility and operator,
11.1.10 Date of measurements.

12. Precision and Bias

12.1 The equipment, its calibration, and its operation shall be such that the coating thickness can be determined with an uncertainty of less than 10 % at 95 % confidence level.

12.2 Instruments suitable for compliance with section 12.1 are available commercially.

12.3 The measurement bias is the discrepancy remaining between the measured thickness and the true thickness if all random errors are eliminated. It is, therefore, no greater than, and attributable to (1), the calibration error of the instrument and (2) the quality of the calibration standard used to calibrate the instrument.

13. Keywords

13.1 aperture, beta backscatter, coating thickness, isotope
X1. SATURATION THICKNESS AS A FUNCTION OF DENSITY FOR VARIOUS ISOTOPES

![Graph showing saturation thickness as a function of density for various isotopes](image-url)
## X2. ISOTOPES USED WITH BETA BACKSCATTER GAGES

<table>
<thead>
<tr>
<th>Isotope or Source</th>
<th>Symbol</th>
<th>Emax, MeV</th>
<th>Approximate Half-Life, years</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon</td>
<td>C-14</td>
<td>0.16</td>
<td>5750.7</td>
</tr>
<tr>
<td>Promethium</td>
<td>Pm-147</td>
<td>0.22</td>
<td>2.8</td>
</tr>
<tr>
<td>Thallium</td>
<td>Tl-204</td>
<td>0.77</td>
<td>3</td>
</tr>
<tr>
<td>Lead-210 – bismuth-210</td>
<td>Pb-210 – Bi-210</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Radium D + E</td>
<td>Ra D + E</td>
<td>1.17</td>
<td>19.4</td>
</tr>
<tr>
<td>Strontium – yttrium</td>
<td>Sr-90 – Y-90</td>
<td>2.27</td>
<td>28</td>
</tr>
<tr>
<td>Ruthenium – rhodium</td>
<td>Ru-106 – Rh-106</td>
<td>3.54</td>
<td>1</td>
</tr>
</tbody>
</table>

## X3. ATOMIC NUMBERS OF SOME COMMONLY USED COATINGS AND SUBSTRATES

<table>
<thead>
<tr>
<th>Element</th>
<th>Atomic Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>13</td>
</tr>
<tr>
<td>Cadmium</td>
<td>48</td>
</tr>
<tr>
<td>Chromium</td>
<td>24</td>
</tr>
<tr>
<td>Cobalt</td>
<td>27</td>
</tr>
<tr>
<td>Copper</td>
<td>29</td>
</tr>
<tr>
<td>Gold</td>
<td>79</td>
</tr>
<tr>
<td>Iron</td>
<td>26</td>
</tr>
<tr>
<td>Lead</td>
<td>82</td>
</tr>
<tr>
<td>Magnesium</td>
<td>12</td>
</tr>
<tr>
<td>Nickel</td>
<td>28</td>
</tr>
<tr>
<td>Organic materials</td>
<td>~6</td>
</tr>
<tr>
<td>Platinum</td>
<td>78</td>
</tr>
<tr>
<td>Rhodium</td>
<td>45</td>
</tr>
<tr>
<td>Silver</td>
<td>47</td>
</tr>
<tr>
<td>Tin</td>
<td>50</td>
</tr>
<tr>
<td>Titanium</td>
<td>22</td>
</tr>
<tr>
<td>Zinc</td>
<td>30</td>
</tr>
</tbody>
</table>

## X4. REPRODUCIBILITY OF MEASUREMENTS

X4.1 The following table summarizes the results of a round robin participated in by 46 laboratories and conducted by ASTM Committee B08. Each laboratory measured two specimens of gold on nickel, 0.7 and 1.3 µm, using an 0.8-mm 

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aperture with the promethium isotope. Measurement time was
30 s and calibration measurements were 240 s each. Each
specimen was measured ten times after the instrument was
calibrated. The calibration and set of ten measurements were
repeated five times. Subsequent to the round robin, Section 8
on calibration was revised to incorporate tighter control of the
calibration procedure in order to reduce the variations between
laboratories.

Standard Deviations
(Square roots of components of variance for various sources of
variability)

<table>
<thead>
<tr>
<th>Source of Variability</th>
<th>Thickness</th>
<th>Repeatability of a Single Measurement</th>
<th>Between Calibrations</th>
<th>Between Laboratories</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness</td>
<td>0.7 µm</td>
<td>0.015 µm</td>
<td>0.014</td>
<td>0.047</td>
</tr>
<tr>
<td>1.3 µm</td>
<td></td>
<td>0.025 µm</td>
<td>0.027</td>
<td>0.054</td>
</tr>
</tbody>
</table>

These data indicate the overall performance of the laborato-
ries and not necessarily the adequacy of the test method even
though the laboratories were instructed to follow this test
method. Also similar measurements of other coating systems
are likely to yield different results.
Standard Test Method for Measurement of Coating Thickness by X-Ray Spectrometry

This standard is issued under the fixed designation B 568; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the use of X-ray spectrometry to determine thickness of metallic and some nonmetallic coatings.

1.2 The maximum measurable thickness for a given coating is that thickness beyond which the intensity of the characteristic secondary X radiation from the coating or the substrate is no longer sensitive to small changes in thickness.

1.3 This test method measures the mass of coating per unit area, which can also be expressed in units of linear thickness provided that the density of the coating is known.

1.4 Problems of personnel protection against radiation generated in an X-ray tube or emanating from a radioisotope source are not covered by this test method. For information on this important aspect, reference should be made to current documents of the National Committee on Radiation Protection and Measurement, Federal Register, Nuclear Regulatory Commission, National Institute of Standards and Technology (formerly the National Bureau of Standards), and to state and local codes if such exist.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

2.2 International Standard:
ISO 3497 Metallic Coatings—Measurement of Coating Thickness—X-ray Spectrometric Methods

3. Terminology

3.1 Definitions of technical terms used in this test method may be found in Terminology E 135.

4. Summary of Test Method

4.1 Excitation—The measurement of the thickness of coatings by X-ray spectrometric methods is based on the combined interaction of the coating and substrate with incident radiation of sufficient energy to cause the emission of secondary radiation characteristic of the elements composing the coating and substrate. The exciting radiation may be generated by an X-ray tube or by certain radioisotopes.

4.1.1 Excitation by an X-Ray Tube—Suitable exciting radiation will be produced by an X-ray tube if sufficient potential is applied to the tube. This is on the order of 35 to 50 kV for most thickness-measurement applications. The chief advantage of X-ray tube excitation is the high intensity provided.

4.1.2 Excitation by a Radioisotope—Of the many available radioisotopes, only a few emit gamma radiations in the energy range suitable for coating-thickness measurement. Ideally, the exciting radiation is slightly more energetic (shorter in wavelength) than the desired characteristic X rays. The advantages of radioisotope excitation include more compact instrumentation essentially monochromatic radiation, and very low background intensity. The major disadvantage of radioisotope excitation is the much lower intensities available as compared with X-ray tube sources. X-ray tubes typically have intensities that are several orders of magnitude greater than radioisotope sources. Due to the low intensity of radioisotopes, they are unsuitable for measurements on small areas (less than 0.3 mm in diameter). Other disadvantages include the limited number of suitable radioisotopes, their rather short useful lifetimes, and the personnel protection problems associated with high-intensity radioactive sources.

4.2 Dispersion—The secondary radiation resulting from the exposure of an electroplated surface to X radiation usually contains many components in addition to those characteristic of the coating metal(s) and the substrate. It is necessary, therefore, to have a means of separating the desired components so that their intensities can be measured. This can be done either by diffraction (wavelength dispersion) or by electronic discrimination (energy dispersion).

4.2.1 Wavelength Dispersion—By means of a single-crystal spectrogoniometer, wavelengths characteristic of either the coating or the substrate may be selected for measurement.

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2 Annual Book of ASTM Standards, Vol 03.05.
Published data in tabular form are available that relate spectrogoniometer settings to the characteristic emissions of elements for each of the commonly used analyzing crystals.

4.2.2 Energy Dispersion—X-ray quanta are usually specified in terms of their wavelengths, in angstroms (Å), or their equivalent energies in kiloelectron volts (keV). The relationship between these units is as follows:

\[(\text{keV})/(\text{Å}) = 12.396\]

where:

- keV = the quantum energy in thousands of electron volts
- Å = the equivalent wavelength in angstroms (10⁻¹⁰ m).

In a suitable detector (see 4.3.2), X rays of different energies will produce output pulses of different amplitudes. After suitable amplification, these pulses can be sorted on the basis of their amplitudes and stored in certain designated channels of a multichannel analyzer, each adjacent channel representing an increment of energy. Typically, a channel may represent a span of 20 eV for a lithium-drifted silicon detector or 150 to 200 eV for a proportional counter. From six to sixty adjacent channels can be used to store the pulses representing a selected characteristic emission of one element, the number of channels depending on the width of the emission peak (usually displayed on the face of a cathode ray tube). The adjacent channels used to store the pulses from the material under analysis are called the “region of interest” or ROI.

4.3 Detection:

4.3.1 Wavelength Dispersive Systems—The intensity of a wavelength is measured by means of an appropriate radiation detector in conjunction with electronic pulse-counting circuitry, that is, a scaler. With wavelength dispersive systems, the types of detectors commonly used as the gas-filled types and the scintillation detector coupled to a photomultiplier tube.

4.3.2 Energy-Dispersive Systems—For the highest energy resolution with energy-dispersive systems, a solid-state device such as the lithium-drifted silicon detector must be used. This type of detector is maintained at a very low temperature in a liquid-nitrogen cryostat (77K). Acceptable energy resolution for most thickness measurement requirements can be realized with proportional counters, and these detectors are being used on most of the commercially available thickness gages based on X-ray spectrometry. In setting up a procedure for coating-thickness measurement using an energy-dispersive system, consideration should be given to the fact that the detector “sees” and must process not only those pulses of interest but also those emanating from the substrate and from supporting and masking materials in the excitation enclosure. Therefore, consideration should be given to restricting the radiation to the area of interest by masking or collimation at the radiation source. Similarly, the detector may also be masked so that it will see only that area of the specimen on which the coating thickness is to be determined.

4.4 Basic Principle—A relationship exists between coating thickness and secondary radiation intensity up to the limiting thickness mentioned in 1.2. Both of the techniques described below are based on the use of primary standards of known coating thicknesses which serve to correlate quantitatively the radiation intensity and thickness.

4.5 Thickness Measurement by X-Ray Emission—In this technique, the spectrogoniometer is positioned to record the intensity of a prominent wavelength characteristic of the coating metal or, in the case of an energy-dispersive system, the multichannel analyzer is set to accept the range of energies comprising the desired characteristic emission. The intensity of the coating’s X-ray emission (coating ROI) will be at a minimum for a sample of the bare substrate where it will consist of that portion of the substrate fluorescence which may overlap the ROI of the coating and a contribution due to background radiation. This background radiation is due to the portion of the X-ray tube’s output which is the same energy as the coating’s X-ray emission. The sample will always scatter some of these X rays into the detector. If the characteristic emission energies of the coating and substrate are sufficiently different, the only contribution of the substrate will be due to background. For a thick sample of the solid coating metal or for an electroplated specimen having an “ininitely thick” coating, the intensity will have its maximum value for a given set of conditions. For a sample having a coating of less than “infinite” thickness, the intensity will have an intermediate value. The intensity of the emitted secondary X radiation depends, in general, upon the excitation energy, the atomic numbers of the coating and substrate, the area of the specimen exposed to the primary radiation, the power of the X-ray tube, and the thickness of the coating. If all of the other variables are fixed, the intensity of the characteristic secondary radiation is a function of the thickness or mass per unit area of the coating. The exact relationship between the measured intensity and the coating thickness must be established by the use of standards having the same coating and substrate compositions as the samples to be measured. The maximum thickness that can be measured by this method is somewhat less than what is, effectively, infinite thickness. This limiting thickness depends, in general, upon the energy of the characteristic X-ray and the density and absorption properties of the material under analysis. The typical relationship between a coating thickness and the intensity of a characteristic emission from the coating metal is illustrated by the curve in the Appendix, Fig. 1.

4.6 Thickness Measurements by X-Ray Absorption—In this technique the spectrometer, in the case of a wavelength-dispersive system, is set to record the intensity of a selected emission characteristic of the basis metal. In an energy-dispersive system, the multichannel analyzer is set to accumulate the pulses comprising the same energy peak. The intensity will be a maximum for a sample of the uncoated basis metal and will decrease with increasing coating thickness. This is because both the exciting and secondary characteristic radiations undergo attenuation in passing through the coating. Depending upon the atomic number of the coating, when the coating thickness is increased to a certain value, the characteristic radiation from the substrate will disappear, although a certain amount of scattered radiation will still be detected. The measurement of a coating thickness by X-ray absorption is not applicable if an intermediate coating is present because of the indeterminate absorption effect of intermediate layer. The typical relationship between coating thickness and the intensity
of a characteristic emission from the substrate is shown in the Appendix, Fig. 2.

4.7 Thickness and Composition Measurement by Simultaneous X-ray Emission and Absorption (Ratio Method)—It is possible to combine the X-ray absorption and emission techniques when coating thicknesses and alloy composition are determined from the ratio of the respective intensities of substrate and coating materials. Measurements by this ratio method are largely independent of the distance between test specimen and detector.

4.8 Multilayer Measurements—Many products have multilayer coatings in which it is possible to measure each of the coating layers by using the multiple-energy-region capability of the multichannel analyzer of an energy-dispersive system. The measuring methods permit the simultaneous measurement of coating systems with up to 3 layers. Or the simultaneous measurement of thickness and compositions of layers with up to 3 components. Such measurements require unique data processing for each multilayer combination to separate the various characteristic emissions involved, to account for the absorption by intermediate layers, and to allow for any secondary excitation which may occur between layers. Typical examples of such combinations are gold on nickel on copper and nickel on copper on steel.

4.9 Mathematical Deconvolution—When using a multichannel analyzer a mathematical deconvolution of the secondary radiation spectra can be used to extract the intensities of the characteristic radiation. This method can be used when the energies of the detected characteristic radiations do not differ sufficiently (for example, characteristic radiation from Au and Br). This method sometimes is described as 'numerical filtering' in order to distinguish from the technique of setting fixed region of interest (ROI) channel limits in the multichannel analyzer.

5. Significance and Use

5.1 This is a sensitive, noncontact, and nondestructive method for measuring the coating thickness (and in some cases, coating composition) of metallic and some nonmetallic coatings over a range of thicknesses from as little as 0.01 µm to as much as 75 µm depending on the coating and substrate materials. It can be used to measure coating and base combinations that are not readily measured by other techniques.

5.2 The coating thickness is an important factor in the performance of a coating in service.

6. Factors Affecting Accuracy

6.1 Counting Statistics—The production of X-ray quanta is random with respect to time. This means that during a fixed time interval, the number of quanta emitted will not always be the same. This gives rise to the statistical error which is inherent in all radiation measurements. In consequence, an estimate of the counting rate based on a short counting interval (for example, 1 or 2 s) may be appreciably different from an estimate based on a longer counting period, particularly if the counting rate is low. This error is independent of other sources of error such as those arising from mistakes on the part of the operator or from the use of inaccurate standards. To reduce the statistical error to an acceptable level, it is necessary to use a counting interval long enough to accumulate a sufficient number of counts. When an energy-dispersive system is being used it should be recognized that a significant portion of an intended counting period may be consumed as dead time, that is, time during which the count-rate capacity of the system is exceeded. It is possible to correct for dead-time losses. The manufacturer’s instructions for accomplishing this with his particular instrumentation should be followed.

6.1.1 The standard deviation, s, of this random error will closely approximate the square root of the total count; that is, 

\[ s = \sqrt{N} \]

The “true” count will lie within \( N \pm 2 \times 95\% \) of the time. To understand the significance of the precision, it is helpful to express the standard deviation as a percent of the count, \( 100\sqrt{N}/N = 100/\sqrt{N} \). Thus, 100 000 would give a standard deviation indicating 10 times the precision (one-tenth the standard deviation) obtained from 1000 counts. This is because \( (100/\sqrt{10000})/(100/\sqrt{100000}) = 10 \). This does not mean that the result would necessarily be ten times as accurate (see 7.2).

6.1.2 A counting interval should be chosen that will provide a net count of at least 10 000. This would correspond to a statistical error in the count rate of 1%. The corresponding standard deviation in the thickness measurement is a function of the slope of the calibration curve at the point of measurement. Most commercially available instruments display the standard deviation directly in units of thickness.

6.2 Coating Thickness—The precision of the measurement will be affected by the thickness range being measured. In the curve shown in the Appendix, Fig. 1, the precision will be best in the portion of the curve from approximately 0.25 to 7.5 µm. The precision rapidly becomes poorer in the portion of the curve above approximately 10 µm. The situation is similar for the absorption curve shown in the Appendix, Fig. 2. At coating thicknesses greater than approximately 10 µm, the intensity changes very little with the coating thickness and, therefore, the precision in that region is poor. These limiting thicknesses are, in general, different for each coating material.

6.3 Size of Measuring Area—To obtain satisfactory counting statistics (see 6.1) in a reasonably short counting period, the exposed area of the significant surface should be as large as practicably consistent with the size and shape of the specimen. Caution must be exercised, however, to see that the use of a large sample area in conjunction with high power input to the X-ray tube does not result in a signal so large as to exceed the count-rate capacity of the detection system.

6.4 Coating Composition—Thickness determinations by X-ray methods can be affected by the presence of foreign materials such as inclusions, codeposited material, and alloying metals as well as by voids and porosity. The sources of error will be eliminated by the use of calibration standards electroplated in the same type of solution under the same conditions as those used in the production of the coatings to be measured. If pores or voids are present, X-ray methods will give an indication of coating mass per unit area but not of thickness.

6.5 Density—If the density of the coating materials differs from that of the calibration standards, there will be a corresponding error in the thickness measurement. Commercially available X-ray fluorescence instruments allow the use of a
density correction procedure to compensate for small differences between the density of the coating material to be measured and the density of the calibration standards coating material. This procedure is commonly used for the measurement of hard gold coatings having a density of 17.5 g/cm³ with calibration standards having a soft (pure) gold coating, which has a density of 19.3 g/cm³. Variations in density can result either from variations in composition or from variations in plating conditions (see 6.4).

6.6 Substrate Composition—The effect of differences in substrate composition will be relatively minor on thickness measurements made by the X-ray emission method if an intensity ratio is used and if the X rays emitted by the substrate do not excite or overlap the radiation being measured. However, when thickness measurements are made by the X-ray absorption method the substrate composition of the test specimens must be the same as that of the standards.

6.7 Substrate Thickness—The effect of a thin substrate will be slight on thickness measurements by X-ray emission provided that an intensity ratio is used and if the X rays emitted by the substrate are not energetic enough to excite the radiation being measured. Care must be taken that the coating and substrate are thick enough to prevent the primary X-ray beam from reaching and fluorescing the material on which the sample is supported. However, when thickness is to be determined by the X-ray absorption technique, the thickness of the substrate must exceed a certain minimum or critical thickness. It must be established experimentally that the minimum thickness requirements have been met for a particular substrate-source combination, although it is sometimes possible to back up the test specimen substrates with a sufficient thickness of materials of the same composition. The X-ray absorption method cannot be used when one or more intermediate coating layers are present.

6.8 Surface Cleanliness—Foreign materials such as dirt, grease, or corrosion products will lead to inaccurate thickness determinations. Protective coatings such as lacquer or chromate conversion coatings over the coating to be measured will also affect the results.

6.9 Specimen Curvature—Thickness measurements should be made on flat surfaces if practical. In those cases where the measurement of thickness on curved surfaces cannot be avoided, a collimator should be used on the excitation beam, reducing the measurement area to a size that will minimize the effects of curvature. Spatial relationships between the curved surface, the excitation beam, and the detector are particularly important, and variations in these relationships can introduce errors in measurement. Calibration standards having the same radius of curvature as that of the test specimens can also be used to eliminate curvature effects.

6.10 Excitation Energy—The intensity of the characteristic secondary radiation from either the coating or the substrate is strongly affected by any variation in the excitation energy, that is, by changes in potential applied to the X-ray tube or changes in the tube current, or both. In general, the radiation intensity varies directly with the current and the square of the potential. Therefore, in any method based on a simple relationship between intensity and thickness, the final adjustment of excitation energy must be made with reference to the observed intensity from a standard sample used to construct the working curve. However, if the method is based on intensity ratios rather than absolute intensities, minor variations in excitation energy are compensated for.

6.11 Detector—Errors can be introduced by erratic operation of the detector system which includes the associated scaling circuitry as well as the detector tube itself. If instability is suspected, a series of twenty or more count measurements should be made on the same specimen without moving the specimen and the standard deviation of the series calculated. Most modern industrial X-ray instrumentation will perform this calculation automatically. The value should not be significantly greater than the square root of one measurement, \(\sqrt{N}\). Some forms of instability become evident if the same specimen is measured periodically.

6.11.1 All radiation-detection/pulse-processing systems have limitations with respect to reliable count-rate capability. Operation of the gas-filled and scintillation types above their count-rate capabilities will result in loss of counts and erroneously low readings. Operation of an energy-dispersive system at high-input pulse rates will require an excessively long time to obtain a statistically valid total, even with “dead-time” compensation (see 6.1).

6.12 Any extrapolation beyond the thickness range covered by the calibration standards excluding infinite thickness can result in serious measurement errors; therefore, it is necessary to take additional steps for measurements outside this range.

6.12.1 When making measurements in the range between the highest thickness standard and the saturation (or infinite thickness) standard, especially in the so-called hyperbolic range, one must always use additional thickness standards with values slightly above and below the presumed thickness of the test specimen. Instrument measurement precision will rapidly decrease with increasing thickness in the hyperbolic range. For this reason, significantly longer measurements times are usually required for measurement applications using the hyperbolic range.

6.12.2 The use of additional standards between the substrate standard and the lowest thickness standard will also improve the accuracy of the measurement in the lower range, which is also called the linear range.

6.13 Filter to Absorb Secondary Radiation—When measuring coating/substrate combinations having similar characteristic emission energies it is often helpful to use an absorber or filter made from an appropriate material to absorb the characteristic X-ray emission of the substrate or coating material to improve measurement accuracy and precision. In most commercially available XRF systems this absorber is a thin metal foil which is manually or automatically placed between the detector and the test specimen.

7. Calibration

7.1 General—In taking instrument readings for the purpose of establishing an instrument calibration, exactly the same instrumental conditions, including collimator size, voltage, and tube current, shall be used as those which will be used on test specimens.
7.2 Standards—Prolonged counting periods will not compensate for inaccurate standards. Standards representing various thickness ranges of a number of coatings on different substrates are generally available from thickness gage manufacturers. Those that are certified for thickness (as opposed to mass per unit area) are suitable provided they are used for coatings of the same density and composition. Calibration standards for gold coatings, certified as to mass of gold per unit area, with an accuracy of ±5 %, are available.3 If standards representing a particular type of coating and substrate are not available, their preparation may be undertaken only if thoroughly competent personnel in the fields of electroplating and analytical chemistry are available.

7.2.1 Calibration standards must be used in such a manner as to minimize wear and abrasion of the plated surface. If the standards are visibly scratched or abraded they should be replaced. It is recommended that two sets of standards be maintained, that is, a set of primary standards and a set of working standards. These should be used only to calibrate and periodically check the condition of the working standards. At the first signs of wear or discrepancy, the working standards should be replaced.

7.3 The instrument shall be calibrated with thickness standards having the same coating and substrate materials as those being measured. Exceptions are allowed if the intensity of the characteristic coating material emission is not influenced by the characteristic emission of the substrate material. An example of this situation is the measurement of silver on copper. The instrument calibration may be made with standards of silver on nickel. The intensity of the characteristic silver emission is not influenced by the characteristic emission of nickel or copper.

7.4 The coating of the calibration standards must have the same X-ray emission (or absorption) properties as the coating being measured. If the coating of the standards is electrodeposited from the same bath and under the same conditions as the coating to be measured, the X-ray properties may be assumed to be the same. If the coating on the standard is gold, but not electroplated under conditions known to be the same as the coating being measured, the X-ray properties may be assumed to be the same for mass per unit area measurements. Under such circumstances, thickness measurements must be corrected for density differences, unless density differences can be shown to be insignificant.

7.5 If the thickness is to be determined by the X-ray absorption technique, the substrate of the thickness standards shall have the same X-ray emission properties as that of the test specimen. This shall be verified by comparing the intensities of the selected characteristic radiations of both uncoated substrate materials.

7.6 In the X-ray absorption technique, the substrate thickness of the test specimen and the calibration standards shall be the same unless the critical thickness, as defined in 6.7, is exceeded.

7.7 If the curvature of the coating to be measured is such as to preclude calibration on a flat surface, the curvature of the standard and that of the test specimen shall be the same.

8. Standard Less Techniques by Fundamental Parameter Computer Simulation

8.1 If the software of the XRF instrument is capable to model the true physical properties of the coating and basis material characteristic X-ray emission (by fundamental parameter based computer simulation) then a measurement of coating thickness and coating composition is obtained which is not derived from an instrument calibration with standards. This standard less measurement shall be corrected by means of calibration standards. The standards correction procedure (calibration) performs the same way as the procedure used for establishing empirical instrument calibrations alone.

8.2 In cases when the coating(s) to be measured and the available calibration standard do not meet the conditions of 7.3, then the computer simulation based on the fundamental parameter technique will cover such situations, if the following conditions are fulfilled:

1) The composition of the coating(s) of standard and part to be measured does not differ considerably;

2) If the characteristic radiation of substrate components influences the radiation intensities which are used for calculating the thickness and composition of the coating, the composition of the substrate of standard and specimen shall not differ considerably.

9. Referee Test

9.1 If a referee test is required in order to resolve a disagreement, it shall be performed by using suitable Standard Reference Material (SRM)4 thickness standards from the National Institute of Standards and Technology (NIST), if such standards are available. A suitable SRM standard is an SRM standard of the same substrate/coating combination for which the XRF gage was calibrated, the thickness of which is within the range of the calibration, preferably close to that of the test specimens being measured. The SRM shall be measured 5 times, each measurement being made under the same conditions as used for the measurement of the test piece. If the average of the 5 measurements of the SRM differs from the certified mass per unit area of equivalent thickness by more than 10 %, the calibration is not valid.

Note 1—SRMs are issued by NIST and include coating thickness SRMs for some coating systems. The stated mass per unit area of each coating thickness SRM is certified to be within 5 % of the true mass per unit area.

10. Procedure

10.1 Operate each instrument in accordance with the manufacturer’s instructions, paying attention to the factors listed in Section 6. Calibrate it in accordance with Section 7.

10.2 Calibration Checks—The instrument calibration shall be checked periodically or before a test series, by remeasuring one of the calibration standards or a reference specimen with known mass per area or thickness. If there is a change of the measured thickness that is large enough to preclude meeting the requirements of section 11, recalibrate the instrument.

3 Available from the National Institute of Standards and Technology, Office of Standard Reference Materials, Gaithersburg, MD 20899.

4 SRMs may be purchased from the Office of Standard Reference Materials, National Institute of Standards and Technology, Gaithersburg, MD 20899.
10.3 Observe the following precautions:

10.3.1 Substrate Thickness—If the X-ray absorption technique is used, verify that the substrate thickness of the test specimens exceeds the critical thickness. If not, make sure that the calibration has been made with a substrate having the same thickness and emission properties as the test specimens.

10.3.2 Measurement Area—The size of the measurement area will depend on the size of the collimator used to restrict and control the size of the excitation beam. In no case shall the measurement area be larger than the coated area available on the test specimen. Suitable means must be provided to perfectly align the test specimen relative to the excitation beam.

10.3.3 Surface Cleanliness—Remove all foreign materials, such as dirt, grease, lacquer, oxides, and conversion coatings from the surface before the measurement by cleaning without removing any of the coating material. Avoid specimen areas having visible defects such as flux, acid spots, and dross in making measurements.

10.3.4 Measuring Time—Use a sufficient measuring time to obtain a repeatability that will yield the desired accuracy (see 6.1).

10.4 Computation of Results—Convert the intensity readings to thickness units. The conversion is made automatically by most commercial coating thickness instruments.

11. Precision and Bias

11.1 The equipment, its calibration, and its operation shall be such that the coating thickness can be determined with an uncertainty of less than 10% at 95% confidence level.

11.2 Although an uncertainty of less than 10% may be achieved consistently for a great number of applications, the uncertainty may be greater when the coating thickness is less than 1 µm.

11.3 Instruments suitable for compliance with section 11.1 are available commercially. For many coating systems the instruments are capable of making measurements with an uncertainty of less than 5% at 95% confidence level.

11.4 The measurement bias is the discrepancy remaining between the measured thickness and the true thickness if all random errors are eliminated. It is, therefore, no greater than, and attributable to (1) the calibration error of the instrument and (2) the quality of the calibration standard used to calibrate the instrument.

12. Report

12.1 The report shall include the following information:

12.1.1 Type of instrument used,

12.1.2 Size of collimator aperture,

12.1.3 Measurement time,

12.1.4 Description of test specimen and measurement area,

12.1.5 If applicable a statement that a correction for density was made,

12.1.6 Type of calibration standards and the measurement mode used,

12.1.7 Thickness of coating as determined from the measurements,

12.1.8 Statistical measurement parameters of the measurement series as required,

12.1.9 Identification of testing facility and operator, and

12.1.10 Date of measurements.

13. Keywords

13.1 absorption; collimator; emission; filter; ratio; x-ray

APPENDIX

(Nonmandatory Information)

X. CALIBRATION CURVES

X1.1 Since commercially available X-ray spectrometry thickness gages give a direct display in thickness units, it is not necessary for the user of such equipment to generate or to use calibration curves. However, the general shapes of calibration curves in the emission mode and in the absorption mode of operation are given here as general information.

X1.1.1 Emission—A typical calibration curve for determining a nickel coating thickness by X-ray emission is shown in Fig. X1.1. The intensities are background-corrected, that is, the intensity for Ni Kα is obtained from a sample of the unplated brass substrate and subsequently subtracted for each of the intensity readings obtained from electroplated samples.

X1.1.2 Absorption—A representative calibration curve for determining a nickel coating thickness by X-ray absorption is shown in Fig. X1.2. The intensities are background-corrected as they were in the emission technique. The emission now being measured comes from the substrate.
FIG. X1.1 Calibration Curve for the Determination of a Nickel Thickness by X-Ray Emission

Note—Intensity is that of the Ni Kα line after a subtraction of background intensity.

FIG. X1.2 Calibration Curve for the Determination of a Nickel Thickness by X-Ray Absorption

Note—Intensity is that of the Cu Kα line from the brass substrate after subtraction of background intensity.
Standard Practice for Qualitative Adhesion Testing of Metallic Coatings

This standard is issued under the fixed designation B 571; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This practice describes simple, qualitative tests for evaluating the adhesion of metallic coatings on various substrates.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Significance and Use

2.1 These tests are useful for production control and for acceptance testing of products.

2.2 Interpreting the results of qualitative methods for determining the adhesion of metallic coatings is often a controversial subject. If more than one test is used, failure to pass any one test is considered unsatisfactory. In many instances, the end use of the coated article or its method of fabrication will suggest the technique that best represents functional requirements. For example, an article that is to be subsequently formed would suggest a draw or a bend test; an article that is to be soldered or otherwise exposed to heat would suggest a heat-quench test. If a part requires baking or heat treating after plating, adhesion tests should be carried out after such post-treatment as well.

2.3 Several of the tests are limited to specific types of coatings, thickness ranges, ductilities, or compositions of the substrate. These limitations are noted generally in the test descriptions and are summarized in Table 1 for certain metallic coatings.

2.4 “Perfect” adhesion exists if the bonding between the coating and the substrate is greater than the cohesive strength of either. Such adhesion is usually obtained if good electroplating practices are followed.

2.5 For many purposes, the adhesion test has the objective of detecting any adhesion less than “perfect.” For such a test, one uses any means available to attempt to separate the coating from the substrate. This may be prying, hammering, bending, beating, heating, sawing, grinding, pulling, scribing, chiseling, or a combination of such treatments. If the coating peels, flakes, or lifts from the substrate, the adhesion is less than perfect.

2.6 If evaluation of adhesion is required, it may be desirable to use one or more of the following tests. These tests have varying degrees of severity; and one might serve to distinguish between satisfactory and unsatisfactory adhesion in a specific application. The choice for each situation must be determined.

2.7 When this guideline is used for acceptance inspection, the method or methods to be used must be specified. Because the results of tests in cases of marginal adhesion are subject to interpretation, agreement shall be reached on what is acceptable.

2.8 If the size and shape of the item to be tested precludes use of the designated test, equivalent test panels may be appropriate. If permitted, test panels shall be of the same material and have the same surface finish as the item to be tested and shall be processed through the same preplating, electroplating, and postplating cycle with the parts they represent.

3. Bend Tests

3.1 Bend the part with the coated surface away over a mandrel until its two legs are parallel. The mandrel diameter should be four times the thickness of the sample. Examine the deformed area visually under low magnification, for example, 4×, for peeling or flaking of the coating from the substrate, which is evidence of poor adhesion. If the coating fractures or blisters, a sharp blade may be used to attempt to lift off the coating. With hard or brittle coatings, cracking usually occurs in the bend area. Such cracks may or may not propagate into the substrate. In either case, cracks are not indicative of poor adhesion unless the coating can be peeled back with a sharp instrument.

3.2 Bend the part repeatedly, back and forth, through an angle of 180° until failure of the basis metal occurs. Examine the region at low magnification, for example, 10×, for separation or peeling of the coating. Prying with a sharp blade will indicate unsatisfactory adhesion by lift off of the coating.
4. Burnishing Test

4.1 Rub a coated area of about 5 cm with a smooth-ended tool for approximately 15 s. A suitable tool is a steel rod 6 mm in diameter with a smooth hemispherical end. The pressure shall be sufficient to burnish the coating at each stroke but not so great as to dig into it. Blisters, lifting, or peeling should not develop. Generally, thick deposits cannot be evaluated satisfactorily.

5. Chisel-Knife Test

5.1 Use a sharp cold chisel to penetrate the coating on the article being evaluated. Alternatively the chisel may be placed in back of an overhang area of the coating or at a coating-substrate interface exposed by sectioning the article with a saw. A knife may be substituted for the chisel with or without hammering or light tapping. If it is possible to remove the deposit, the adhesion is not satisfactory. Soft or thin coatings cannot be evaluated for adhesion by this method.

6. Draw Test

6.1 Form a suitable sample about 60 mm in diameter into a flanged cap approximately 38 mm in diameter, to a depth up to 18 mm, through the use of a set of adjustable dies in an ordinary punch press. Penetration of the male die may be continued until the cap fractures. The adhesion of the coating may be observed directly or evaluated further by techniques described in Section 5 for detachment from the substrate. If there is peeling or flaking of the coating or if it can be detached, the adhesion is not satisfactory.

6.2 Results from this technique must be interpreted cautiously, because the ductilities of both the coating and substrate are involved.

7. File Test

7.1 Saw off a piece of the coated specimen and inspect it for detachment at the deposit/substrate interface. Apply coarse mill file across the sawed edge from the substrate toward the coating so as to raise it, using an approach angle of approximately 45° to the coating surface. Lifting or peeling is evidence of unsatisfactory adhesion.

7.2 This technique is not suitable for thin or soft coatings.

8. Grind-Saw Test

8.1 Hold the coated article against a rough emery wheel so that the wheel cuts from substrate toward the deposit in a jerky or bumpy fashion. A hack saw may be substituted for the wheel, making sure to saw in the direction that tends to separate the coating from the substrate. Lifting or peeling is evidence of unsatisfactory adhesion.

8.2 This technique is especially effective on hard or brittle coatings but is not suitable for thin or soft coatings.

9. Heat-Quench Test

9.1 Heat the coated article in an oven for a sufficient time for it to reach the temperature shown in Table 2. Maintain the...
temperature of the oven within 10°C of the nominal. Coatings and substrates that are sensitive to oxidation should be heated in an inert or reducing atmosphere or a suitable liquid. Then quench the part in water or other suitable liquid at room temperature.

9.2 Flaking or peeling of the deposit is evidence of unsatisfactory adhesion. Blisters may erupt during the heat and quench test when plating solution is entrapped in substrate surface pits or pores which are bridged by the deposit. If the deposited coating cannot be peeled or lifted from the substrate in an area adjacent to the blister(s), the appearance of blisters should not be interpreted as evidence of inferior adhesion.

9.3 Diffusion and subsequent alloying of metals may improve the bond strength of electrodeposits. In some cases, a brittle layer may be created by the materials involved causing peeling as a result of fracture rather than poor adhesion. This would not give a correct indication of the as-plated bond strength.

9.4 This test is nondestructive if the procedure does not create unwanted effects on parts.

10. Impact Test

10.1 Use a hammer or impact device coupled with a suitable backing block to support the article to be tested to deform the sample. Reproducible results are more easily obtained by the use of a suitably modified impact tester where the force is reproducible and the impact head contour is in the form of a 5-mm diameter ball, shock loaded by a falling weight or swinging pendulum weight. The severity of the test may be altered by changing the load and diameter of the ball. Exfoliation or blisters in and around indentations are evidence of inadequate adhesion.

10.2 This test is sometimes difficult to interpret. Soft and ductile coatings are generally not suited for evaluation.

11. Peel Test

11.1 Bond a strip of steel or brass about 1.5 mm thick and 20 mm wide by solder or suitable adhesive to a properly flat area of the coated surface of the article. Adhesive-backed tape may be considered as a possible alternative. Heat curing of the adhesive may be used, keeping in mind considerations noted in 9.3. The angle of pull shall be 90° to the surface. For reproducible results, the rate of pull, the thickness and width of the strip, and deposit thickness must be standardized. Failure in the coating/substrate interface is evidence of inadequate adhesion.

11.2 The tensile and shear strengths of adhesives and solders limit the range of adhesion strengths that can be evaluated. A quantitative analysis of the factors involved has been published.3

12. Push Test

12.1 Drill a blind hole 0.75 cm in diameter from the underside until the point of the drill tip comes within approximately 1.5 mm of the deposit/substrate interface on the opposite side. Supporting the material on a ring about 2.5 cm in diameter, apply steady pressure over the blind hole using a hardened steel punch 0.6 cm in diameter until a button sample is pushed out.3 Exfoliation or peeling of the coating in the button or crater areas is evidence of inadequate adhesion.

12.2 Soft, very ductile, and thin deposits are generally not suited for this technique.

13. Scribe-Grid Test

13.1 Scribe three or more parallel lines or a rectangular grid pattern on the article using a hardened steel tool ground to a sharp (30°) point with a distance between the scribed lines of approximately ten times the nominal coating thickness, with a minimum distance of 0.4 mm. In scribing the lines, use sufficient pressure to cut through the coating to the substrate in a single stroke. If any portion of coating between the lines breaks away from the substrate, the adhesion is inadequate.

13.2 When apparently satisfactory adhesion is indicated, apply a pressure-sensitive tape, having an adhesion bond strength of at least 45 g/mm, using firm finger pressure onto a clean grid area. Make sure that any loose coating particles from scribing do not remain. Shortly thereafter, remove the tape by seizing a free end and pulling it off rapidly (not jerked) back upon itself as close to an angle of 180° as possible. The adhesion is not adequate if the tape has deposit adhering to it that comes from the area between the scribed lines. Deposit continuous to the scribed lines is not considered.

13.2.1 The tape supplier4 shall specify that their tape has a sufficient adhesion bond strength. The tape shall be wide enough to cover three or more parallel lines of the scribed area.

13.3 Generally, thick deposits are not suitable for evaluation unless a chisel or other sharp instrument is used to pry the exposed coating/substrate interface, in which case this technique becomes a variant of Section 5.

14. Test-Coating Systems

14.1 Recommended adhesion tests for a variety of coating systems are given in Table 1.

15. Precision and Bias

15.1 No statement is made about the precision and bias of these tests because of their qualitative nature.

16. Keywords

16.1 adhesion; metallic coatings

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4 Permacel 99, a product of and manufactured by Permacel, New Brunswick, NJ 08903, is reported to be a suitable product for this purpose.
1. Scope

1.1 This test method covers the determination of the hardness of metallic coatings upon various substrates. The measurements are made with the Knoop indenter under a test load of 0.245 N (25 gf) or 0.981 N (100 gf).

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   - E 384 Test Method for Microhardness of Materials

3. Special Requirements

3.1 In addition to the requirements listed herein, the test method shall be performed in accordance with Test Method E 384.

3.1.1 The measurement shall be made on the cross section with the long diagonal of the indentation parallel to the substrate using a Knoop indenter.

**Note 1—**When a nondestructive test is necessary, the measurement may be made normal to the surface provided that the whole of the indentation is clearly delineated in the field of the microscope at the magnification specified in Test Method E 384, and the coating thickness is at least 0.7 that of the long diagonal. The outlines of the indentation must be sharp and undistorted by any mechanical effects resulting from the thinness of the coating such as “butterfly” fractures, etc. Values obtained from measurements made normal to the electroplated surface may not be equivalent to those obtained on microsections and are not, therefore, reportable according to Section 6.

3.1.2 The thickness of the coating shall be at least 38 µm for soft coatings such as gold, copper, and silver, and 25 µm for hard coatings such as nickel, cobalt, iron, and hard gold. This is approximately equivalent to 0.6 times the length of the long Knoop diagonal.

3.1.3 Make the indentation near the center of the cross section of the coating.

3.2 No single test load is applicable to all coatings. Use a load of 0.245 N for soft coatings (from 50 to 300 HK) and a load of 0.981 N for hard coatings (greater than 300 HK).

3.2.1 A load of less than 0.245 N is not recommended. However, when the length of the diagonal is great enough at a load of 0.245 N, which may occur for very soft coatings such as tin and indium, a lighter load or a lower magnification shall be specified provided the requirements of 3.3 are met. When testing extremely hard coatings, a load greater than 0.981 N may be used provided this is specified in the purchase order. The Knoop hardness number decreases appreciably with loads from 0.245 N to 0.981 N especially for the higher hardness numbers.

3.3 Measure the length of the long diagonal to within 0.25 µm or 0.4 %, whichever is larger, at the magnification specified in Test Method E 384.

3.4 When readings are taken in a direction parallel to the substrate, space them from each other by at least the length of the long diagonal. When two readings are taken in the direction of the short axis, separate them by at least the length of the diagonal perpendicular to the substrate (short diagonal). In such cases, the distance from the substrate and from the outer surface of the coating to an edge of the indentation shall be at least the width of the short diagonal.

3.5 If the electroplated coating is cracked, or if cracks or “butterfly” markings (Test Method E 384, 7.2) appear when the indentation load is applied or removed, the hardness measurement is invalid.

4. Preparation of Test Specimen

4.1 Overplate the specimen to a thickness of at least 12 µm with a metal of a contrasting color whose hardness approximates that of the coating.

4.2 Cut out a section of the specimen approximately perpendicular to the electroplated coating.

4.3 Mount and polish the section as described in Test Method E 384.

**Note 2—**Procedures 4.1 and 4.2 are interchangeable at the discretion of the operator. A light etch following polishing may remove significant work hardening.

4.4 When the electrodeposited is a metal which may alloy with the basis metal (for example, indium over copper or copper alloys), or if the deposit has a low transformation temperature (such as nickel-phosphorus alloys), a low-temperature mounting procedure shall be used.
5. Procedure

5.1 Insert the mounted specimen into the testing machine so that the test surface is parallel to the long axis of the indenter, and measure the hardness in accordance with the manufacturer’s instructions and in accordance with Test Method E 384, Section A.

6. Report

6.1 Test results shall be based on at least five measurements, and the report shall include the following statistics:

6.1.1 The range, that is, the difference, in hardness units, between the highest and lowest values.

6.1.2 The average (arithmetic mean):

\[ \bar{x} = \frac{\sum x}{n} \]  

(1)

6.1.3 The estimated standard deviation:

\[ s = \sqrt{\frac{\sum (x - \bar{x})^2}{n(n-1)}} \]  

or

\[ s = \sqrt{n\frac{\sum x^2 - (\sum x)^2}{n(n-1)}} \]  

(2)

6.1.4 The coefficient of variation:

\[ v = \frac{s}{\bar{x}} \]  

(3)

where, in 6.1.2-6.1.4:

- \( s \) = the standard deviation,
- \( x \) = an individual value,
- \( \bar{x} \) = arithmetic mean of the set of individual values,
- \( n \) = the number of measurements,
- \( \sum x^2 \) = the sum of the squares of all of the individual values,
- \( (\sum x)^2 \) = the square of the total of the individual values, and
- \( v \) = the coefficient of variation.

6.2 Report the hardness as a Knoop hardness number (HK), and indicate the load by subscript notation expressed as the force, in newtons, divided by 0.00981 (a number equal to that which is obtained when the test force is expressed in grams-force). For example: 410 HK\(_{100}\), 30 HK\(_{500}\), 200 HK\(_{25}\).

7. Precision and Bias

7.1 Precision—An interlaboratory comparison program is now in progress which when completed will be the basis of a statement on precision.

7.2 Bias—There is no basis for defining the bias of this test method.

7.3 Reference hardness specimens of electroformed nickel along with certified hardness readings as found by the National Institute of Standards and Technology (NIST) are currently available.\(^3\)

8. Test Coupons

8.1 If specifically permitted by the governing specification or the party requesting the test, test coupons may be used when the production parts have insufficient electroplating thickness for measurement. The values obtained may not reflect the “true” hardness of the thin coatings on production parts. However, the values may be useful when they correlate with other coating properties such as wear resistance. The test may serve as a useful tool for electroplating bath control, particularly in the case of coatings such as gold, for which the hardness will be sensitive to the composition of the bath and other electroplating variables. The electroplating conditions for test coupons such as current density, temperature, agitation, and solution composition, shall be kept as close as possible to those employed on production parts in the plating process under test.

\(^3\) Available from the Office of Reference Materials, NIST, Gaithersburg, MD 20899.
Standard Specification for Electrodeposited Coatings of Tin-Lead Alloy (Solder Plate)\(^1\)

This standard is issued under the fixed designation B 579; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (\(\epsilon\)) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This specification covers the requirements for electrodeposited tin-lead coatings on fabricated articles of iron, steel, copper, and copper alloys, to protect them against corrosion (Note 1), to improve and preserve solderability over long periods of storage, and to improve anti-galling characteristics.

Note 1—Some corrosion of tin-lead coatings may be expected in outdoor exposure. In normal indoor exposure, tin-lead is protective on iron, copper, and copper alloys. Corrosion may be expected at discontinuities (pits or pores) in the coating. Porosity decreases as the thickness is increased. A primary use of the tin-lead coating (solder) is with the printed circuit industry as a solderable coating and as an etch mask material.

1.2 This specification applies to electrodeposited coatings containing a minimum of 50 % and a maximum of 70 % tin. The specification applies to mat, bright, and flow-brightened tin-lead coatings.

Note 2—Tin-lead plating baths are composed of tin and lead fluoborates and of addition agents to promote stability. The final appearance may be influenced by the addition of proprietary brighteners. Without brighteners, the coatings are mat; with brighteners, they are semibright or bright. Flow-brightened coatings are obtained by heating mat coatings to above the melting point of tin-lead for a few seconds and then quenching; palm oil, hydrogenated oils, or fats are used as a heat-transfer medium at a temperature of 260 ± 10°C (500 ± 20°F), but other methods of heating are also in use. The maximum thickness for flow-brightening is about 7.5 \(\mu\)m (0.3 mil); thicker coatings tend to reflow unevenly. The shape of the part is also a factor; flat surfaces tend to reflow more unevenly than wires or rounded shapes (Note 3).

Note 3—Volatile impurities in tin-lead coatings will cause bubbling and foaming during flow-brightening resulting in voids and roughness. The impurities can arise from plating solution addition agents and from improper rinsing and processing.

1.3 This specification does not apply to sheet, strip, or wire in the unfabricated form or to threaded articles having basic major diameters up to and including 19 mm (0.75 in.).

2. Referenced Documents

2.1 ASTM Standards: \(^2\)

B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Guide for Preparation of High-Carbon Steel for Electroplating
B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings
B 322 Practice for Cleaning Metals Prior to Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
B 504 Test Method for Measurement of Thickness of Metal Coatings by the Coulometric Method
B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
E 105 Practice for Probability Sampling of Materials
E 122 Practice for Calculating Sample Size to Estimate, With a Specified Tolerable Error, the Average for Characteristic of a Lot or Process

2.2 Other Standards:

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes\(^3\)
MIL-STD-414 Sampling Procedures and Tables for Inspection by Variables for Percent Defective\(^3\)

3. Classification and Service Condition

3.1 Orders for articles to be plated in accordance with this specification shall specify, in addition to the ASTM designation...
number and year of issue, the classification notation indicating the basis metal and thickness of tin-lead coating required, or the service condition number indicating the severity of service required for the coating. In addition, when specifying a tin-lead coating composition, the first number shall refer to the tin content in percent.

3.1.1 Classification Notation:

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Classification</th>
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<tbody>
<tr>
<td>Fe/</td>
<td>Iron or steel basis metals</td>
</tr>
<tr>
<td>Cu/</td>
<td>Copper or copper alloy basis metals</td>
</tr>
<tr>
<td>/Sn-Pb</td>
<td>Tin-lead coating and its composition number, when required; for example, Sn60-Pb60</td>
</tr>
<tr>
<td>Number</td>
<td>Minimum coating thickness in micrometres (5 to 50)</td>
</tr>
<tr>
<td>Suffix</td>
<td></td>
</tr>
<tr>
<td>f</td>
<td>flow-brightened</td>
</tr>
<tr>
<td>b</td>
<td>bright</td>
</tr>
<tr>
<td>m</td>
<td>mat</td>
</tr>
</tbody>
</table>

An example of complete classification notation is as follows: Cu/Sn-Pb60-Pb40/5F

3.1.2 Service Condition Number:

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<th>Service Condition</th>
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</tr>
<tr>
<td>3</td>
<td>severe exposure</td>
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<tr>
<td>2</td>
<td>moderate exposure</td>
</tr>
<tr>
<td>1</td>
<td>mild exposure</td>
</tr>
</tbody>
</table>

Note 4—See Appendix X1 for additional description of exposure conditions and examples of typical end uses. The coating thicknesses given for each service condition are guidelines and are not intended to be absolute values.

4. Significant Surfaces

4.1 Significant surfaces are defined as those surfaces normally visible (directly or by reflection) that are essential to the appearance or serviceability of the article when assembled in normal position; or those surfaces that can be the source of corrosion products that will deface visible surfaces on the assembled article. When necessary, the significant surfaces shall be indicated on the drawing of the part, or by the provision of suitably marked samples.

Note 5—When significant surfaces include areas on which the specified thickness of deposit cannot readily be controlled, such as threads, holes, deep recesses, bases of angles, and similar areas, the purchaser and the manufacturer should recognize the necessity for either thicker deposits on the more accessible surfaces or for special racking. Special racks may involve the use of conforming, auxiliary bipolar electrodes, or nonconducting shields.

5. Materials and Manufacture

5.1 Defects in the surface of the basis metal, such as scratches, porosity, nonconducting inclusions, roll and die marks, cold shuts, and cracks, may adversely affect the appearance and the performance of coatings applied thereto despite the observance of the best plating practices. Accordingly, the plater’s responsibility for defects in the coating resulting from such conditions shall be waived, except when he is also in the position of prime contractor supplying plated parts.

Note 6—In order to minimize problems of this sort, the specifications covering the basis material or the item to be plated should contain appropriate limitations on such basis metal conditions.

5.2 When required the basis metal shall be subjected to such polishing or buffing operations as are necessary to yield deposits with the desired final appearance (Section 6).

5.3 Proper preparatory procedures and thorough cleaning of the basis metal surface are essential in order to assure satisfactory adhesion and corrosion performance of the coating. Accordingly, it is suggested that the following Practices for the preparation of various basis metals for electroplating be followed when appropriate: B 183, B 281, and B 322 and Guide B 242.

5.4 When necessary, preliminary samples showing the finish shall be supplied to and approved by the purchaser. Where rack marks are inevitable, their location shall be the subject of agreement between supplier and purchaser.

6. Physical Composition

6.1 Composition—The tin-lead coating composition shall be as follows (Note 7):

Note 7—Only the tin content need be determined. Lead is usually determined by difference. A sample of the deposit can be obtained by plating on a stainless steel panel from which the coating can be peeled or by employing any recognized stripping method. The alloy composition of the deposit can be determined by methods such as gravimetric or volumetric analysis, density measurements, atomic absorption spectrophotometry, X-ray fluorescence, and beta backscatter.

In addition, the alloy composition produced by a plating solution may be obtained by comparing the weight of a tin-lead coating deposited by a given number of ampere-hours to the weight of lead coating produced in a lead fluoride coulometer in series with the plating bath.

6.2 Appearance—The tin-lead coating shall be smooth, fine grained, continuous, adherent, and shall be free of visible blisters, pits, nodules, indications of burning, excessive buildup, staining, and other defects. Flow-brightened coatings shall not have dewetted areas or beads, and shall be free of the oil used in the fusion process.

6.3 Thickness—The thickness of the coating on significant surfaces shall conform to the requirements in Table 1 and Table

### TABLE 1 Tin-Lead Alloy Coatings on Steel

<table>
<thead>
<tr>
<th>Service Condition</th>
<th>Classification Number</th>
<th>Minimum Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>μm   mil</td>
</tr>
<tr>
<td>SC4(^a)</td>
<td>Fe/SnPb 30</td>
<td>30   1.2</td>
</tr>
<tr>
<td>SC3(^a)</td>
<td>Fe/SnPb 20</td>
<td>20   0.8</td>
</tr>
<tr>
<td>SC2</td>
<td>Fe/SnPb 10</td>
<td>10   0.4</td>
</tr>
<tr>
<td>SC1</td>
<td>Fe/SnPb 5</td>
<td>5    0.2</td>
</tr>
<tr>
<td>SC1</td>
<td>Fe/SnPb 5(^b)</td>
<td>5    0.2</td>
</tr>
</tbody>
</table>

\(^a\) An undercoat of 2.5 µm (0.1 mil) copper is recommended for SC3 and SC4.  
\(^b\) f = flow brightened or m = mat or b = bright
2. 6.3.1 Thickness Measurements—Tin-lead alloy thickness measurements shall be made on those areas of the significant surfaces where the coating would be expected to be thinnest. The method of determining the thickness shall be agreed upon by the manufacturer and purchaser. Several methods are available depending upon the thickness of coating, the shape of the article, and the basis metal. They include beta backscatter, coulometric, magnetic, microscopical, and X-ray fluorescence test methods. The methods are outlined in 9.1.

Note 8—Thicknesses determined by beta backscatter, coulometry, and X-ray fluorescence are a function of the composition as well as the thickness of the coating.

6.4 Adhesion—The adhesion of the coating shall be adequate to pass the tests described in 9.2.

6.5 Solderability:

6.5.1 When specified by the purchaser, the coating shall be tested by one of the methods described in 9.2. The results shall be evaluated in accordance with each procedure described in that section.

6.5.2 When specified by the purchaser, the coating on copper and copper alloys shall, before solderability testing, be subjected to the preliminary artificial aging treatment described in 9.3.6 to determine if they may be expected to retain their solderability during periods of storage.

Note 9—See Appendix X2 for design considerations that have an effect on the selection of thickness of the coating and, ultimately, on the solderability of the electrodeposit.

7. Hydrogen Embrittlement

7.1 High-tensile strength steels, and severely cold-worked steels, are susceptible to embrittlement by hydrogen in both cleaning and plating operations. The embrittling hydrogen shall be removed by heat treatment. Procedures for baking to minimize embrittlement before and after plating are covered in Sections 2 and 7 of Guide B 242.

8. Sampling

8.1 Test methods are time consuming and often destructive; therefore 100 % inspection is usually impractical. The purchaser should select a suitable sampling plan for the acceptance testing of lots of coated items. In order that the manufacturer (plater) may know the quality standard he is expected to meet, the plan selected should be made part of the purchase contract.

8.2 General information on sampling procedures is given in Recommended Practices E 105 and E 122. Standard sampling plans are suggested in Military Standards MIL-STD-105 and MIL-STD-414.

9. Test Methods

9.1 Thickness:

9.1.1 To meet the thickness specifications of the coatings, the plater is advised to:

9.1.1.1 Maintain regular control of all solutions,

9.1.1.2 Inspect the equipment at regular intervals, and

9.1.1.3 Check thickness at periodic intervals.

9.1.2 The following ASTM methods are acceptable for measuring local thickness of the coating: B 487, B 499, B 504, B 567, and B 568.

9.2 Adhesion:

9.2.1 Burnishing Test—Rub an area of not more than 630 mm² (1 in.²) of the coated surface, selected at the discretion of the inspector, rapidly and firmly for 15 s with a smooth metal implement. A suitable burnishing implement is a copper or steel disk used edgewise and broadside. Maintain a pressure sufficient to burnish the coating at every stroke, but not so great as to cut it. Poor adhesion will be shown by the appearance of a loose blister which grows as burnishing is continued. If the quality of the coating is poor also, the blister may crack and the coating peel away from the basis metal.

9.2.2 Quenching Test—Heat the coating article in an oven for a sufficient time to reach 150 ± 10°C (300 ± 20°F) and quench in room-temperature water. The adhesion is inadequate if the coating blisters, cracks, or peels.

9.2.3 Reflow Test—Parts may be evaluated by immersion in a bath of palm oil at a temperature of 205 – 260°C (400 – 500°F) until the deposit melts. A bright coating completely covering the significant surfaces indicates adequate adhesion.

9.2.4 Bend Test—Bend a sample, with the coated surface away, over a mandrel until its two legs are parallel. The mandrel shall have a diameter equal to the thickness of the sample. Examination at 4× magnification should show no evidence of peeling or cracking.

9.3 Solderability:

9.3.1 General:

9.3.1.1 Methods for testing the solderability of tin-lead coated articles are based on the measurement of the extent of wetting by molten solder or the determination of the minimum time required to produce full or perfect wetting by solder.

9.3.1.2 The extent of wetting can be observed by manual or automatic immersion in molten solder under controlled conditions.

9.3.1.3 Determine the minimum wetting time by carrying a specimen in a fixture through a standing wave of solder at a controlled speed and by measuring the shortest time of immersion that will give complete wetting.

9.3.2 Dip Tests (Non-Automated):  

9.3.2.1 Sample—For small articles of suitable shape and size take the whole article for testing. For larger articles, cut a portion of suitable size for testing. A recommended panel size

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Table 2: Tin-Lead Alloy Coatings on Copper, Copper Alloys, and Nonmetals

<table>
<thead>
<tr>
<th>Service Condition</th>
<th>Classification Number</th>
<th>Minimum Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>µm</td>
</tr>
<tr>
<td>SC4</td>
<td>Cu/SnPb 30</td>
<td>30</td>
</tr>
<tr>
<td>SC3</td>
<td>Cu/SnPb 15</td>
<td>15</td>
</tr>
<tr>
<td>SC2</td>
<td>Cu/SnPb 8</td>
<td>8</td>
</tr>
<tr>
<td>SC1</td>
<td>Cu/SnPb 5</td>
<td>5</td>
</tr>
<tr>
<td>SC1</td>
<td>Cu/SnPb SnPb 5C</td>
<td>5</td>
</tr>
</tbody>
</table>

A If the basis metal is a brass containing more than 15 % zinc, the tin-lead coating shall be preceded by an undercoat of at least 2.5 µm (0.1 mil) of copper and nickel to prevent the diffusion of zinc into the tin-lead. The same undercoating shall also be applied when the basis metal is beryllium copper to assure adhesion of tin-lead coating.

B Nonmetals shall be suitably sensitized and metalized prior to tin-lead coating.

C 1 = flow-brightened or

m = mat or

b = bright
required to maintain maximum protection.

gases, a deposit of 30 to 125 µm (1.2 to 5.0 mil) may be subjected to abrasion or is exposed to corrosive liquids or

cerosed film free of pores. If the coating is complete coating of tin-lead free of pores. If the coating is considered in this category is the use of the tin-lead as an etch resist in the production of printed-circuit boards.

Another application considered in this category is the use of the tin-lead as an etch resist in the production of printed-circuit boards.

Progressively increase contact times with the solder using separate specimens, and determine the least time required for complete wetting and the onset of dewetting by visual examination of the series of specimens. The best conditions of solderability would have the shortest wetting time, and would show no signs of dewetting within the longest contact time required. A minimum wetting time under 2 s is evidence of good solderability. An auxiliary attachment is available for the determination of spread values. The specimen is lowered onto the surface of the solder and a delay timer built into the equipment holds the test piece in contact with the solder for any preselected time up to 10 s. Determine spread values as in 9.3.4.

9.3.4 Spread Test:

9.3.4.1 This method involves placement of a fixed volume of solder on the surface of a specimen with a few drops of rosin flux (Type W flux, MIL-F-14256) and heating the specimen for a fixed period of time at a controlled temperature.

9.3.4.2 The area of spread can be measured with a planimeter.

9.3.4.3 The height of the solder blob can be measured with a stage micrometer which can be set to subtract the thickness of the basis metal and the “spread factor” calculated. A hot plate held at 250 ± 5°C (480 ± 9°F) may be substituted for the oven, used in the Pessel method, as a source of heat.4

9.3.5 Globule Test:

9.3.5.1 This test method was devised for assessing the solderability of wires, component leads, etc.

9.3.5.2 This method consists of lowering the specimen of wire (or component lead) previously fluxed, horizontally onto a molten globule of solder, which is thereby cut in two. The time in seconds for the solder to flow around the wire and unite above it is a measure of the solderability. Use a fresh pellet of solder for each test, the size of the pellet being determined by the diameter of the specimen wire. Commercial test machines are available.

9.3.6 Artificial Aging (When Specified By the Purchaser):

9.3.6.1 Place the sample for test in a suitable vessel above boiling water and leave it there, with the water boiling continuously, for 24 h. Keep the vessel covered and ensure that the sample does not come into contact with the wall of the vessel and that its lower edge is not less than 50 mm (2 in.) or more than 100 mm (4 in.) above the surface of the boiling water. Arrange the cover on the vessel and the steam condenser, if used, so that they do not discharge condensed water over the sample. Disregard any discoloration of the sample occurring during this aging treatment. After the 24-h treatment, remove the sample from the steam and allow it to dry in the air.

9.3.6.2 Test methods outlined in 9.3.2, 9.3.3, 9.3.4, or 9.3.5 are used to assess the solderability of the aged specimens.

10. Keywords

10.1 electrodeposited coatings, tin-lead alloy (solder plate); solder, tin-lead alloy; tin-lead

APPENDIXES

X1. EXAMPLES OF APPROPRIATE SERVICE CONDITIONS

X1.1 SC4—Very severe service conditions require a complete coating of tin-lead free of pores. If the coating is subjected to abrasion or is exposed to corrosive liquids or gases, a deposit of 30 to 125 µm (1.2 to 5.0 mil) may be required to maintain maximum protection.

X1.2 SC3—Severe service conditions include exposure to dampness and to industrial atmospheres. Coatings of 12 to 30 µm (0.5 to 1.0 mil) have been reported to be satisfactory, particularly for preserving a solderable coating for a shorter storage period than that given in SC3. Also, as in SC3, another application considered in this category is the use of the tin-lead as an etch resist in the production of printed-circuit boards.

X1.3 SC2—Moderate service conditions include dry or interior atmosphere. Coatings of 8 to 12 µm (0.3 to 0.5 mil), including flow-brightening, have been reported to be satisfactory, particularly for preserving a solderable coating for a longer storage period (for example, 9 months). Another application considered in this category is the use of the tin-lead as an etch resist in the production of printed-circuit boards.

X1.4 SC1—Mild service conditions with less severe requirements than SC2. Deposits of 5 µm (0.2 mil) and less have been reported satisfactory for providing and preserving a solderable coating for short periods of storage (for example, 3 months).
X2. DESIGN CONSIDERATIONS

X2.1 General:

X2.1.1 The properties of electrodeposited tin-lead coatings satisfy the requirements of solderability, corrosion resistance, etc., outlined in the scope of this specification and their use can be recommended for most applications. Attention is drawn to the effects of temperature and to long-term storage of tin-lead plated articles which may be factors in designing for special applications.

X2.1.2 Temperature Effects:

X2.1.2.1 Tin-lead coatings are soft and will withstand considerable flexing and twisting of the basis metal without serious damage. At room temperature, mat tin-lead coatings will oxidize slowly but flow-brightened and bright tin-lead coatings oxidize less readily.

X2.1.2.2 Interdiffusion between tin-lead coatings and copper or copper alloys does take place. The diffusion is slow at room temperature and rapid at elevated temperatures. Evidence of diffusion is the formation of a layer of copper-tin compound at the interface and, if the substrate is brass, diffusion of zinc to the surface. Diffusion may lead to darkening of a thin coating and impairment of its solderability, particularly after long storage. With such thin coatings, a diffusion barrier of nickel may be advantageous, but users should consider the use of a thicker coating when solderability has to be maintained over a period of years. An undercoat of nickel or copper must be used as a diffusion barrier on brass.
1. Scope

1.1 This specification covers requirements for electrolytically formed porous oxide coatings on aluminum and aluminum alloy parts in which appearance, abrasion resistance, electrical properties, and protection against corrosion are important. Nonporous, barrier layer anodic coatings used for electrical capacitors are not covered. Seven types of coatings as shown in Table 1 are provided. Definitions and typical examples of service conditions are provided in Appendix X1.

NOTE 1—It is recognized that uses exist in which modifications of the coatings covered by this specification may be required. In such cases the particular properties desired by the purchaser should be the subject of agreement between the purchaser and the manufacturer.

2. Referenced Documents

2.1 ASTM Standards:
- B 110 Test Method for Dielectric Strength of Anodically Coated Aluminum
- B 117 Practice for Operating Salt Spray (Fog) Testing Apparatus
- B 136 Test Method for Measurement of Stain Resistance of Anodic Coatings on Aluminum
- B 137 Test Method for Measurement of Coating Mass per Unit Area on Anodically Coated Aluminum
- B 244 Test Method for Measurement of Thickness of Anodic Coatings on Aluminum and of Other Nonconductive Coatings on Nonmagnetic Basis Metals with Eddy-Current Instruments
- B 368 Test Method for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test)
- B 457 Test Method for Measurement of Impedance of Anodic Coatings on Aluminum
- B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section

B 538 Method of FACT (Ford Anodized Aluminum Corrosion Test) Testing
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
D 658 Test Method for Abrasion Resistance of Organic Coatings by Air Blast Abrasion
E 429 Test Method for Measurement and Calculation of Reflecting Characteristics of Metallic Surfaces Using Integrating Sphere Instruments
E 430 Test Methods for Measurement of Gloss of High-Gloss Surfaces by Goniophotometry

2.2 Other Standards:
- MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes
- MIL-STD-414 Sampling Procedures and Tables for Inspection by Variables for Percent Defective

3. Manufacture

3.1 Defects in the surface of the basis metal, such as scratches, porosity, inclusions, roll and die marks, cold shuts, and cracks, will adversely affect the appearance and performance of applied coatings despite the observance of best anodizing practices. Accordingly, defects in the coating that result from such conditions shall not be cause for rejection.

NOTE 2—To minimize problems of this sort, the specifications covering the basis material or the item to be anodized should contain appropriate limitations on such basis metal conditions.

<table>
<thead>
<tr>
<th>Type</th>
<th>Coating (Industry) Description</th>
<th>Minimum Film Thickness (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Engineering Hard Coat</td>
<td>50</td>
</tr>
<tr>
<td>B</td>
<td>Architectural Class I</td>
<td>18</td>
</tr>
<tr>
<td>C</td>
<td>Architectural Class II</td>
<td>10</td>
</tr>
<tr>
<td>D</td>
<td>Automotive—Exterior</td>
<td>8</td>
</tr>
<tr>
<td>E</td>
<td>Interior—Moderate Abrasion</td>
<td>5.0</td>
</tr>
<tr>
<td>F</td>
<td>Interior—Limited Abrasion</td>
<td>3</td>
</tr>
<tr>
<td>G</td>
<td>Chromic Acid</td>
<td>1</td>
</tr>
</tbody>
</table>

TABLE 1 Anodic Coatings Descriptions

NOTE 1—Hard coatings may vary in thickness from 12 µm to more than 100 µm. If the thickness of Type A is not specified, it shall be 50 µm min. Type A coatings will not be sealed unless so specified.

1 This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.07 on Chemical Conversion Coatings.


3 Annual Book of ASTM Standards, Vol 03.02.

4 Annual Book of ASTM Standards, Vol 02.05.

5 Discontinued; see 1986 Annual Book of ASTM Standards, Vol 02.05.

6 Annual Book of ASTM Standards, Vol 06.01.

7 Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.
3.2 The basis metal shall be subjected to such mechanical finishing operations, cleaning, and chemical or electrolytic pretreatments as are necessary to yield anodic coatings with the final quality and appearance specified by the purchaser.

3.3 Except where specifically excluded, anodized parts shall be sealed in water or aqueous chemical solutions of such purity, composition, pH, and temperature, as to impart the properties specified herein.

4. Significant Surfaces

4.1 Significant surfaces are defined as those normally visible (directly or by reflection) which are essential to the appearance of serviceability of the article when assembled in normal position; or those surfaces which can be the source of corrosion products that will deface visible surfaces and interfere with functional surfaces on the assembled article. When necessary, the significant surfaces shall be the subject of agreement between purchaser and manufacturer and shall be indicated on the drawings of the parts, or by the provision of suitably marked samples.

Note 3—When significant surfaces are involved on which the specified thickness or density of the coating cannot readily be controlled, such as threads, holes, deep recesses, and similar areas, the purchaser and the manufacturer should recognize the necessity for either thicker films on the more accessible surfaces or for special racking.

5. Manner of Specifying Requirements

5.1 Coating Description—When ordering articles to be finished in accordance with this specification, the purchaser shall state:

5.1.1 The ASTM designation number,
5.1.2 The coating type and description (see Table 1),
5.1.3 The minimum anodic film thickness,
5.1.4 Special post anodic treatments,
5.1.5 Applicable quality assurance requirements (see Section 6),
5.1.6 Significant surface appearance requirements such as color, texture, or reflectivity, and
5.1.7 The alloy to which the coating is to be applied.

5.2 Supplementary Coating—Any supplementary coating that is required in addition to normal or special sealing must either be described in detail along with its requirements or the appropriate specification(s) must be referenced.

6. Quality Assurance

6.1 Anodic oxide coatings can be produced to have many different characteristics. No single coating can be expected to have all of these characteristics. Therefore, the quality assurance requirements for a given coating should be selected to control those properties necessary to the expected end use for the product.

6.2 Anodic coatings supplied under this specification shall meet the minimum requirements for film thickness as stated in Table 1.

6.3 The following ASTM methods are applicable to anodic coatings within the scope of this specification: B 110, B 117, B 136, B 137, B 244, B 368, B 457, B 487, B 538, D 658, E 429, and E 430. The selection of tests to be required and the level of performance against each test, with the exception of minimum film thickness, shall be subject to agreement between purchaser and manufacturer. The Dye Stain Test, as described in Method B 136, shall not be required for Type G coatings or for Types B through F coatings sealed only in dichromate solutions, or for unsealed Type A coatings.

7. Workmanship and Appearance

7.1 Workmanship—The anodic coatings shall be continuous, smooth, adherent, uniform in appearance, and shall be free of powdery areas (burns), loose films, stains, discolorations, and discontinuities such as pits, breaks and scratches, or other damage. The size and number of contact marks shall be the minimum consistent with good practice. The location of contact marks shall be in areas of minimum exposure to service environmental conditions when important to the function of the part.

7.2 Appearance—If applicable, the color and finish appearance (bright, dull, or satin) shall be a reasonably close approximation to that of a sample consisting of treated pieces agreed upon as the standard range by the manufacturer and the purchaser.

Note 4—This range, representing the limits that the manufacturer will supply and that the purchaser will accept, should be established before any work is performed to meet this specification.

8. Sampling

8.1 Test methods are time consuming and often destructive; therefore 100 % inspection is usually impractical. The purchaser should select a suitable sampling plan for the acceptance testing of lots of coated items. In order that the manufacturer may know the quality standard he is expected to meet, the plan selected should be made a part of the purchase contract.

8.2 Information on sampling procedures is given in Method B 602. Standard sampling plans are suggested in Military Standards MIL-STD-105 and MIL-STD-414.
## X1. DEFINITIONS AND TYPICAL EXAMPLES OF SERVICE CONDITIONS

<table>
<thead>
<tr>
<th>SC No.</th>
<th>Definition</th>
<th>Typical Applications</th>
<th>Applicable Coating Types</th>
</tr>
</thead>
<tbody>
<tr>
<td>Very severe (5)</td>
<td>Exposure to atmospheric weathering that can be expected to extend for many years or to prolonged high bearing load wear conditions.</td>
<td>Unmaintained exterior architectural facades, machinery parts, marine</td>
<td>A and B</td>
</tr>
<tr>
<td>Severe (4)</td>
<td>Exposure that includes likely damage from denting, scratching, and abrasive wear coupled with corrosive environments.</td>
<td>1-Automotive—exterior, 2-maintained architectural exterior facades, windows</td>
<td>C and D</td>
</tr>
<tr>
<td>Moderate (3)</td>
<td>Exposure that is likely to include occasional wetting with coating subject to moderate wear or abrasion.</td>
<td>Lighting reflectors—exterior, athletic equipment, appliances, nameplates, lawn furniture</td>
<td>E</td>
</tr>
<tr>
<td>Mild (2)</td>
<td>Exposure indoors in normally dry atmospheres with coating subject to minimum wear or abrasion.</td>
<td>Automotive—interior, houseware items, lighting reflectors—enclosed</td>
<td>F</td>
</tr>
<tr>
<td>Crevise condition (1)</td>
<td>Exposure to humid atmospheres with little or no abrasive condition. Particularly for lap joints.</td>
<td>Spot-welded or riveted assemblies such as aircraft and electronic components.</td>
<td>G</td>
</tr>
</tbody>
</table>

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Standard Test Method for Measurement of Thickness of Transparent or Opaque Coatings by Double-Beam Interference Microscope Technique

1. Scope

1.1 This test method covers the measurement of the thickness of transparent metal oxide and metallic coatings by utilizing a double-beam interference microscope.\(^2\)

1.2 The test method requires that the specimen surface or surfaces be sufficiently mirrorlike to form recognizable fringes.

1.3 This test method can be used nondestructively to measure 1 to 10\(\mu\)m thick transparent coatings, such as anodic coatings on aluminum. The test method is used destructively for 0.1 to 10 \(\mu\)m thick opaque coatings by stripping a portion of the coating and measuring the step height between the coating and the exposed substrate. The stripping method can also be used to measure 0.2 to 10 \(\mu\)m thick anodic coatings on aluminum.

1.4 The test method is usable as a reference method for the measurement of the thickness of the anodic film on aluminum or of metallic coatings when the technique includes complete stripping of a portion of the coating without attack of the substrate. For anodic films on aluminum, the thickness must be greater than 0.4 \(\mu\)m; the uncertainty can be as great as 0.2 \(\mu\)m. For metallic coatings, the thickness must be greater than 0.25 \(\mu\)m; the uncertainty can be as great as 0.1 \(\mu\)m.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method\(^3\)

3. Summary of Test Method

3.1 While observing the specimen surface through the interference microscope, the top surface of the coating and the substrate surface are located with white light interference fringe group(s). Then the elevation difference between the two surfaces is ascertained by counting the number of monochromatic fringes by which the white light fringes are displaced. The number of fringes, multiplied by one half of the light wavelength, is the film thickness.

3.2 When light is reflected, it undergoes a phase shift, the magnitude of which depends on the material and on its structure. The uncertainty of the thickness measurement due to this phenomenon is, theoretically, less than \(\frac{1}{8}\) the wavelength of the light for metals and \(\frac{1}{4}\) wavelength for nonmetallic coatings on metal. Those uncertainties are included in those given in 1.4. They can be eliminated for measurements made in accordance with 1.3 and 7.1.2 by coating the specimen after the stripping operation with a thin but uniform reflective layer of a metal by evaporation. The two reflecting surfaces will then be of the same material and the phase shifts will be the same.

3.3 The aperture of the microscope objective contributes to the fringe displacement by an amount determined by the aperture size. Therefore, a correction\(^4\) is added equal to \(\frac{a^2}{4}\) where \(a\), expressed in radians, is the arc sine of the numerical aperture of the microscope objective.

NOTE 1—When the angle is given in radians and is less than 0.6, the angle is approximately equal to its sine.

3.4 With a reticle such as shown in the figures, the fringe count is likely to have an uncertainty of \(\frac{1}{10}\) wavelength (\(\frac{1}{5}\)fringe interval). More precise measurements can be made with the aid of a filar micrometer eyepiece.

4. Significance and Use

4.1 The thickness of a coating is often critical to its performance.

4.2 For some coating-substrate combinations, the interference microscope method is a reliable method for measuring coating thickness.

4.3 This test method is suitable for specification acceptance.

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\(^1\) This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on Test Methods.


\(^3\) Annual Book of ASTM Standards, Vol 02.05.

5. Apparatus

5.1 Interference Microscope equipped with a reticle or filar micrometer eyepiece for linear measurements.

5.2 Incandescent and Monochromatic Light Sources.

6. Sample Preparation for Destructive Technique

6.1 Anodic Coating on Aluminum—After masking (Note 2), the coating is stripped by immersion in a solution containing 33 g/L chromic acid (CrO₃) and 0.5 cm³/L phosphoric acid (H₃PO₄) (85%). Operating temperature is 85 to 95°C.

NOTE 2—Masking for both transparent and opaque coatings can be accomplished by applying an adhesive tape such as 3M #470 or equivalent with its edge at a location where the thickness measurement is desired. The tape must be sufficiently adherent and impermeable to protect the coating beneath from subsequent stripping action.

NOTE 3—In certain cases, this method causes attack of the basis metal. The attack is usually accompanied by pitting, which is easily observable in the interference microscope by comparing the general contour exhibited by the fringes on the unstripped portion with the general contour on the stripped portion. If such attack occurs, the method is not valid.

6.2 Metallic Coatings on Metallic Substrates—After masking (Note 2), the coating is stripped without attack of the substrate (see Appendix X1).

7. Thickness Measurement

NOTE 4—Many surfaces have microscopical ridges or valleys produced by a previous operation (such as rolling or polishing). Measurements of film thickness are made best with the fringes oriented in a direction perpendicular to the directional surface roughness.

7.1 Transparent Coatings:

7.1.1 Nondestructive Technique:

7.1.1.1 As the surface of a specimen is viewed through the interference microscope using the incandescent illuminator (white light), adjust the microscope fine-focus knob and the reference mirror controls so that a group of strong fringes (arising from the coating-substrate interface) and a group of weak fringes (arising from the coating-air interface) are both in view as illustrated in Fig. 1A.

7.1.1.2 Determine the number of monochromatic fringes between the centers of the white light fringe groups. Appendix X2 indicates alternative ways of performing this procedure.

7.1.2 Destructive Technique:

7.1.2.1 Position the boundary between the stripped and unstripped portion of the specimen in the field of view of the microscope.

7.1.2.2 As the surface of the specimen is viewed through the interference microscope using the white light, adjust the microscope fine-focus knob and the reference mirror controls so that the group of fringes arising from the bare substrate and the weak fringes arising from the coating-air interface are both in view; as illustrated in Fig. 2A.

7.1.2.3 Determine the number of monochromatic fringes between the centers of the white light fringe groups. Appendix X2 indicates alternative ways of performing this procedure.

7.1.2.4 Calculate thickness \( T \) as follows:

\[
T = \frac{n \lambda}{2 \mu} \left[1 + \left(\frac{\alpha}{4}\right)^2\right]
\]

where:

- \( n \) = number of fringes,
- \( \lambda \) = wavelength of monochromatic light, \( \mu \) m,
- \( \mu \) = refractive index of coating for light of wave length, \( \lambda \), and
- \( \alpha \) = arc sine (numerical aperture of objective) in radians.

Thus for the thickness of the anodic coating on aluminum represented in Fig. 1,

\[
T = \frac{[(24 \times 0.546) / (2 \times 1.62)] [1 + (0.78^2/4)]}{2} = 4.66 \mu m
\]

where the monochromatic source is a mercury green light with a wavelength of 0.546 \( \mu m \), where the refractive index of the anodic coating is 1.62, and where alpha is equal to 0.78.

7.1.2.5 Destructive Technique:

7.1.2.5.1 Position the boundary between the stripped and unstripped portion of the specimen in the field of view of the microscope.

7.1.2.5.2 As the surface of the specimen is viewed through the interference microscope using the incandescent illuminator, adjust the microscope fine-focus knob and the tilt of the reference mirror so that the fringe group on both sides of the boundary is in the field of view, as illustrated in Fig. 3A.

7.2 Opaque Coatings—Destructive Technique:

7.2.1 Position the boundary between the stripped and unstripped portions of the specimen in the field of view of the microscope.

7.2.2 As the surface of the specimen is viewed through the interference microscope using the incandescent illuminator, adjust the microscope fine-focus knob and the tilt of the reference mirror so that the fringe group on both sides of the boundary is in the field of view, as illustrated in Fig. 3A.

\[ T = \frac{n \lambda}{2 \mu} \left[1 + \left(\frac{\alpha}{4}\right)^2\right] \]
7.2.3 Determine the number of monochromatic fringes between the centers of the white light fringe groups. Appendix X2 indicates alternative ways of performing this procedure.

7.2.4 Calculate thickness $T$ as follows:

$$T = \frac{n\lambda}{2} [1 + (\alpha^2/4)]$$

where:
- $n$ = number of fringes,
- $\lambda$ = wavelength of monochromatic light, $\mu$m, and
- $\alpha$ = arc sine (numerical aperture of objective) in radians.

8. Accuracy Requirement

8.1 Transparent Coating on Metal Substrate—The entire procedure shall be such that the coating thickness can be determined either within $\pm 0.2 \mu$m or within 5% of the coating thickness, whichever is greater.

8.2 Metal Coating on Metal Substrate—The entire procedure shall be such that the coating thickness can be determined either within $\pm 0.1 \mu$m or within 5% of the coating thickness, whichever is greater.

9. Precision and Bias

9.1 A satisfactory interlaboratory comparison of this test method has not yet been conducted.
X1. STRIPPING OF METALLIC COATINGS

X1.1 The cell and electronic equipment used for the coulometric method of measuring coating thickness, Method B 504, provides a convenient way of masking and stripping a small area of coating. Chromium coatings may be stripped from nickel or steel by anodic dissolution at 5 to 10V in a 5 g/L sodium carbonate ($\text{Na}_2\text{CO}_3$) solution using at least a full-wave rectifier filtered with 10 000 µF capacitance.

X2. COUNTING MONOCHROMATIC FRINGES

X2.1 White Light Fringes—Chromatic aberrations impose a limit to the way a microscope can be used, and the extent of these aberrations should be determined. With white light and with a specimen and the microscope adjusted so that the central fringe of the color fringes crosses the center of the field, the central fringe usually has a different color near the edge of the field. For example, the central fringe may be black at the center off the field and composed of contiguous red, black, and green layers near the edge. If the fringe pattern is moved perpendicularly across the field, the black central line may become colored and an adjacent line becomes black so that the original central line loses its identity.

X2.1.1 The change is associated with chromatic aberrations that give rise to measurement errors. The operator is advised to scan the field with the central fringe and to note the extent of aberrations. The observations described in the following paragraphs should be confined to those parts of the field within which the central fringe of each fringe group does not change color.

X2.2 For alternative means of measuring the fringe displacement see X2.3, X2.4, X2.5, and X2.6. The methods of X2.4 and X2.5 can be used if chromatic aberrations interfere with the method of X2.3. The method of X2.6 completely avoids any chromatic aberrations, but is difficult to use if the fringe displacement is much more than about five fringes and cannot be used for the nondestructive technique (7.1.1).

X2.3 Monochromatic Fringes with Stationery White Light Fringes—The microscope is adjusted as described in 7.1.1.1, 7.1.2.2, or 7.2.2 so that the two groups of color fringes are in the field. The positions of the central fringes on the reticle and the reticle interval between them are noted (Fig. 1(A), Fig. 2(A), and Fig. 3(A)). Monochromatic light is then substituted for the white light without disturbing the specimen or microscope settings, and the monochromatic fringes within the same reticle interval are counted (Fig. 1(B), Fig. 2(B), and Fig. 3(B)).

X2.4 Monochromatic Fringes with Resetting of White Light Fringes:

X2.4.1 The microscope is adjusted using white light so that the eyepiece hairline is over the central fringe that locates the coating (or substrate surface), Fig. 4(A). Using the fine focus control on the “compensator” control (the compensator adjusts the relative path lengths of the two interferometer beams), the amount of adjustment required to bring the central fringe locating the substrate (or coating) surface to the original position of the first central fringe Fig. 4(B), is noted and is estimated (from previous experience) in terms of number of monochromatic fringes. The first position, Fig. 4(A), is reinstated, the monochromatic light is substituted for the white light. The monochromatic fringes are shifted with respect to the hairline by the previously estimated number of fringes. Then with white light, it is noted what additional adjustment is required to bring the second central fringe into position, and an

---

improved estimate is made of the total adjustment required in terms of the number of monochromatic fringes. Beginning with reinstatement of the first position, the process is repeated several times until the estimated number of fringes proves to be the adjustment needed to bring the second central fringe into position, Fig. 4(B).

X2.4.2 For accurate measurements, the procedure described in X2.4.1 is used to determine the whole number of fringe spacings. Additional displacement of less than one fringe spacing is estimated directly from the monochromatic fringes (Fig. 5).

X2.5 Stage Elevation—The microscope is equipped with a means of precisely moving the stage relative to the microscope objective with the movement being calibrated using monochromatic fringes. The movement can be controlled with a cantilever system and micrometer, with a piezoelectric device, or with the fine focus control. One measures the movement required to shift the position of one central fringe to that of the other, Fig. 4.

X2.6 Beveled Boundary—If a boundary is formed by stripping part of the coating and if the boundary is beveled so that each monochromatic fringe can be followed across the boundary, white light need not be used. The eyepiece hairline is superimposed over one of the monochromatic fringes and one counts the number of fringes it traverses (Fig. 5); that is, the number of fringes by which the fringe pattern is displaced. The method is difficult to use if the displacement is much more than about five fringes.
Standard Specification for Sintered Aluminum Structural Parts

This standard is issued under the fixed designation B 595; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers sintered aluminum structural parts made primarily from aluminum powders to which controlled amounts of master alloys or elemental copper, magnesium, and silicon have been added by blending.

1.2 This specification covers the following variables:

1.2.1 Composition—Depending upon levels of copper, magnesium, and silicon content, two grades, and

1.2.2 Density—Type.

1.3 Parts ordered to this specification will be in one of the following conditions:

1.3.1 As-sintered,

1.3.2 As-repressed for additional density, or

1.3.3 All other conditions plus heat treated.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

2. Referenced Documents

2.1 ASTM Standards:

B 243 Terminology of Powder Metallurgy

B 328 Test Method for Density, Oil Content, and Interconnected Porosity of Sintered Metal Structural Parts and Oil-Impregnated Bearings

E 8 Test Methods for Tension Testing of Metallic Materials

3. Terminology

3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243. Additional descriptive information is available in the Related Material Section of Volume 02.05 of the Annual Book of ASTM Standards.

4. Ordering Information

4.1 Orders for material to this specification shall include the following information:

4.1.1 Dimensions (see 9.1),

4.1.2 Chemical composition (see 6.1),

4.1.3 Density (see 7.1),

4.1.4 State of heat treatment,

4.1.5 Mechanical property requirements (see 8.1), and

4.1.6 Certification (see 14.1).

5. Materials and Manufacture

5.1 Structural parts shall be made by molding and sintering metal powders to produce finished parts conforming to the requirements of this specification.

6. Chemical Composition

6.1 The material shall conform to the requirements of Table 1.

6.2 The chemical analysis shall be made in accordance with the methods prescribed in the latest edition of the Annual Book of ASTM Standards, Vol 03.05, or any other approved method agreed upon between the manufacturer and the purchaser.

7. Density

7.1 The parts shall conform to the density range prescribed in Table 2.

7.2 The density shall be measured in accordance with Test Method B 328.

7.3 If the density does not vary more than 0.1 g/cm³ from one section of the structural part to any other section, the overall density shall fall within the limits prescribed in Table 2.

7.4 If the density varies more than 0.1 g/cm³ from one section of the structural part to any other section, the manufacturer and the purchaser shall agree upon a critical section of the part where the stresses are the highest. The density of this critical section rather than the average density shall fall within the limits prescribed in Table 2.

8. Mechanical Properties

8.1 The manufacturer and the purchaser shall agree on qualification tests for the determination of mechanical properties.

8.2 These tests shall be performed on production parts.

8.3 These tests shall be determined after consideration of the function of the part.

8.4 The limits and sampling plan shall be agreed upon between the manufacturer and purchaser.

8.5 All shipments of parts subsequent to the establishment
of testing conditions shall conform to the limits agreed upon.

NOTE 1—The mechanical properties in tension and compression that may be expected from standard specimens molded to size are given in Appendix X1 of this specification.

9. Dimensions and Tolerances
9.1 Permissible variations in dimensions shall be within the limits specified on the drawings describing the structural parts accompanying the order or shall be within the limits specified in the order.

10. Workmanship, Finish, and Appearance
10.1 Structural parts shall be uniform in composition.
10.2 When parts are cut or fractured, the exposed surface shall be of uniform appearance.

11. Sampling
11.1 Lot—Unless otherwise specified, a lot shall consist of parts of the same form and dimensions made from powders of the same composition molded and sintered under the same conditions and submitted for inspection at one time.
11.2 Chemical Analysis—If required by purchase agreement, at least one sample for chemical analysis shall be taken from each lot. A representative sample of chips may be obtained by dry milling, drilling or crushing at least two pieces with clean dry tools without lubrication. In order to obtain oil-free chips, the parts selected for test shall have the oil extracted in accordance with Test Method B 328, if necessary.

11.3 Mechanical Tests—The manufacturer and purchaser shall agree on a representative number of specimens for mechanical tests.

12. Inspection
12.1 Unless otherwise specified, inspection of parts supplied on contract shall be made by the purchaser.

13. Rejection and Rehearing
13.1 Parts that fail to conform to the requirements of this specification may be rejected. Rejection should be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with test results, the producer or supplier may make claim for rehearing.

14. Certification
14.1 When specified in the purchase order or contract, a producer’s certification shall be furnished to the purchaser that the parts were manufactured, sampled, tested, and inspected in accordance with this specification and have been found to meet the requirements. When specified in the purchase order or contract, a report of the test results shall be furnished.
14.2 The purchase order shall specify whether or not the certification includes chemistry.
14.3 Upon request of the purchaser in the contract or order, the certification of an independent third party indicating conformance to the requirements of this specification may be considered.

15. Keywords
15.1 as-repressed; as-sintered; density; interconnected porosity; oil-impregnated; powder metallurgy; sintered aluminum; structural parts; thermal condition

APPENDIX

(Nonmandatory Information)

X1. MECHANICAL PROPERTIES AND HARDNESS DATA

X1.1 Data for the mechanical properties of sintered aluminum specimens are given in Table X1.1. The data do not constitute a part of this specification. They merely indicate to the purchaser the mechanical properties that may be expected from special tension specimens conforming to the density and chemical requirements specified. It should be understood that the values represent specimens molded to size and not specimens cut from commercial parts. See Fig. 20 of Test Methods E 8.
TABLE X1.1 Typical Properties

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Type</th>
<th>Ultimate Tensile Strength</th>
<th>Tensile Yield Strength (0.2% offset)</th>
<th>Elongation in 25 mm or 1 in.,%</th>
<th>Apparent Rockwell Hardness</th>
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<td>15 000</td>
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<tr>
<td>AT6-6061 II</td>
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<td>30 000</td>
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<td>ACT4-2014 I</td>
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<td>279</td>
<td>40 000</td>
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</table>

Note: Typical sintering atmosphere for the above grades may be nitrogen, dissociated ammonia, or vacuum.

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Standard Test Method for Attribute Sampling of Metallic and Inorganic Coatings

This standard is issued under the fixed designation B 602; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method gives sampling plans that are intended for use in the inspection of metallic and inorganic coatings for conformance to ASTM standard specifications.

1.2 The plans in this test method, except as noted, have been selected from some of the single sampling plans of MIL-STD-105D. The specific plans selected are identified in Tables 1-3 of this test method. The plan of Table 4, which is used for destructive testing, is not from the Military Standard. This standard does not contain the Military Standard’s requirement for tightened inspection when the quality history of a supplier is unsatisfactory.

1.3 The plans are based on inspection by attributes, that is, an article of product is inspected and is classified as either conforming to a requirement placed on it, or as nonconforming. Sampling plans based on inspection by variables are given in Method B 762. Variables plans are applicable when a test yields a numerical value for a characteristic, when the specification imposes a numerical limit on the characteristic, and when certain statistical criteria are met. These are explained in Method B 762.

1.4 The plans in this test method are intended to be generally suitable. There may be instances in which tighter or looser plans or onesthat are more discriminating are desired. Additional plans that may serve these needs are given in Guide B 697. Also, Guide B 697 describes the nature of attribute sampling plans and the several factors that must be considered in the selection of a sampling plan. More information and an even greater selection of plans are given in MIL-STD-105D, MIL-STD-414, ANSI/ASQC Z1.9, Refs (1-7), and in Guide B 697.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
   B 762 Method of Variables Sampling of Metallic and Inorganic Coatings

2.2 Military Standards:
   MIL–STD–105D Sampling Procedures and Tables for Inspection by Attributes
   MIL-STD-414 Sampling Procedures and Tables for Inspection by Variables for Percent Defective
   ANSI Standard:
   ANSI/ASQC Z1.9-1979 Sampling Procedures and Tables for Inspection by Variables for Percent Non-Conformance

3. Terminology

3.1 Definitions:
   destructive test—a test that destroys the tested article or makes it nonconforming to a requirement.
   inspection lot—a collection of articles of the same kind that is submitted to inspection for acceptance or rejection as a group.
   nondestructive test—a test that neither destroys the tested article nor makes it nonconforming to a requirement.
   sample—articles randomly selected from an inspection lot whose quality is used to decide whether or not the inspection lot is of acceptable quality.

4. Significance and Use

4.1 Sampling inspection permits the estimation of the overall quality of a group of product articles through the inspection of a relatively small number of product items drawn from the group.

4.2 The selection of a sampling plan provides purchasers and sellers a means of identifying the minimum quality levels that are considered to be satisfactory.

4.3 Because sampling plans will only yield estimates of the quality of a product, the results of the inspection are subject to error. Through the use of sampling plans, the risk of error is
5. General

5.1 In sampling inspection, a relatively small number of articles (the sample) is selected randomly from a larger number of articles (the inspection lot) and is inspected for conformance to the requirements placed on the articles. Based on the results, a decision is made either to accept or reject the inspection lot. Sampling is used, rather than inspection of every article in a lot, to reduce cost. Also, some test methods are destructive, in which cases sampling inspection must be used to avoid destroying the lot.

5.2 There is always a risk that a sample will not be representative of the lot from which it is drawn. The larger the sample, the smaller this risk, but the larger the cost of inspection. So the selection of a sampling plan involves the balancing of the costs of inspection against the consequences of accepting an undesirable number of nonconforming articles.

If every article in an inspection lot conforms to its requirements, every article in the sample will conform also. Such lots will always be accepted. If only a few articles in an inspection lot are nonconforming, the chances are that the sample will indicate that the lot is acceptable; but there is a small chance that the sample will indicate that the lot is unacceptable. The larger the proportion of nonconforming articles in an inspection lot, the more likely it will be that the sample will indicate that the lot is unacceptable. In the extreme case of every article in an inspection lot being nonconforming, a sample will always indicate that the lot is unacceptable.

5.3 For a given sampling plan, the chance of accepting an inspection lot that contains nonconforming items is often described in terms of the Acceptance Quality Level (AQL) and the Limiting Quality Level (LQL). The AQL is the quality level known and controlled.

### TABLE 1 Level I—Sampling Plan for Nondestructive Tests

<table>
<thead>
<tr>
<th>Inspection Lot Size</th>
<th>Sample Size</th>
<th>Acceptance Number</th>
<th>AQL, %</th>
<th>50/50 Point, %</th>
<th>LQL, %</th>
<th>AOQL, %</th>
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<tbody>
<tr>
<td>1 to 20</td>
<td>all</td>
<td>0</td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>21 to 280</td>
<td>20</td>
<td>0</td>
<td>0.26</td>
<td>3.4</td>
<td>11.0</td>
<td>1.8</td>
</tr>
<tr>
<td>281 to 1,200</td>
<td>80</td>
<td>1</td>
<td>0.44</td>
<td>2.1</td>
<td>4.8</td>
<td>1.1</td>
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<tr>
<td>1,201 to 2,200</td>
<td>125</td>
<td>2</td>
<td>0.55</td>
<td>2.1</td>
<td>4.3</td>
<td>1.1</td>
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<td>2,201 to 10,000</td>
<td>200</td>
<td>3</td>
<td>0.68</td>
<td>1.8</td>
<td>3.3</td>
<td>0.97</td>
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<tr>
<td>10,001 to 35,000</td>
<td>315</td>
<td>5</td>
<td>0.83</td>
<td>1.8</td>
<td>2.9</td>
<td>1.0</td>
</tr>
<tr>
<td>Over 35,000</td>
<td>500</td>
<td>7</td>
<td>0.80</td>
<td>1.5</td>
<td>2.4</td>
<td>0.90</td>
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</table>

A Taken from MIL-STD-105D, Single Sampling Plan, Level II, AQL = 0.65, Normal Inspection.
B The smallest lots are 100 % inspected, and so there is no sampling risk. For this reason, there are no AQL, etc.

### TABLE 2 Level II—Sampling Plan for Nondestructive Tests

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<th>Inspection Lot Size</th>
<th>Sample Size</th>
<th>Acceptance Number</th>
<th>AQL, %</th>
<th>50/50 Point, %</th>
<th>LQL, %</th>
<th>AOQL, %</th>
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B The smallest lots are 100 % inspected, and so there is no sampling risk. For this reason, there are no AQL, etc.

### TABLE 3 Level III—Sampling Plan for Nondestructive Tests

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<th>Acceptance Number</th>
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<th>50/50 Point, %</th>
<th>LQL, %</th>
<th>AOQL, %</th>
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<td>3.0</td>
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<td>2.9</td>
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</table>

B The smallest lots are 100 % inspected, and so there is no sampling risk. For this reason, there are no AQL, etc.

### TABLE 4 Sampling Plan for Destructive Test

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<th>Inspection Lot Size</th>
<th>Sample Size</th>
<th>Acceptance Number</th>
<th>AQL, %</th>
<th>50/50 Point, %</th>
<th>LQL, %</th>
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<tr>
<td>Over 35,000</td>
<td>55</td>
<td>3</td>
<td>2.5</td>
<td>6.6</td>
<td>12</td>
</tr>
</tbody>
</table>

A AOQLs are not given because destructive tests cannot be used to screen rejected lots. This plan is not found in MIL-STD-105D.
that is considered to be acceptable. The LQL is the quality level that is considered to be barely tolerable. A sampling plan is selected that will accept most submitted inspection lots of AQL quality and reject most lots of LQL quality. In this test method the AQL given for a sampling plan is the quality level of lots (expressed as the percentage of nonconforming articles) that have a 95% chance of being accepted. The LQL is the quality level of lots that have a 10% chance of being accepted or, in other words, a 90% chance of being rejected. Also given with each sampling plan in this test method, is the quality level of an inspection lot that has a 50% chance of being accepted. This is called the 50/50 point.

5.4 If all of the articles in a rejected inspection lot are inspected, and if nonconforming articles are removed and replaced with conforming articles, and then if the now 100% conforming lot is resubmitted, the average quality level for a series of lots taken as a whole will be better because of the addition of the 100% conforming lot. When the incoming lots are of a good quality level, the average quality level of a series of lots will be even better when the rejected lots are screened and resubmitted. When incoming lots are of a poor quality level, the average quality of a series of lots will again be good because many of the incoming lots will be rejected and upgraded. At intermediate quality levels of incoming lots, the average quality level of a series of lots will not be as good as in either of the above cases. The poorest average quality level that can result from the use of a given sampling plan when screening of rejected lots is done is called the Average Outgoing Quality Limit (AOQL). If corrective action is taken by the supplier so that there is a low rejection rate of initially supplied lots, the average quality level will be better than the AOQL (Note 1). This 100% inspection of rejected lots cannot, of course, be used if the inspection test method is destructive. Screening of rejected lots will substantially increase the cost of inspection if the incoming lots are much worse than AQL quality. Screening is to be used only when required by the purchaser.

**Note 1**—The AOQLs given for Tables 1-3 are strictly correct only when the sample is small with respect to the lot. If the sample is consistently a significant part of the lot, the correct AOQL will be smaller than the tabulated value. The correct values are obtained by multiplying the tabulated values by:

$$1 - \frac{\text{sample size}}{\text{lot size}}$$

5.5 This test method contains four sampling plans. Three are intended to be used when the inspection methods are nondestructive. One of these (Table 2) is considered to be standard and is the one that is followed unless the user of this method specifies either a higher quality level (Table 1) or a lower one (Table 3). The fourth plan is intended to be used when the inspection methods are destructive. This last plan utilizes smaller samples and so reduces the cost of inspection but with a sacrifice in the ability to distinguish between acceptable and unacceptable lots.

6. Ordering Information

6.1 Unless otherwise specified by the purchaser, the sampling plan given in Table 2 will be used for nondestructive testing, and the plan given in Table 4 for destructive testing.

6.2 When either a nondestructive or a destructive test can be used to inspect an article for conformance to a particular requirement, the purchaser should specify which test is to be used. When a test is neither clearly nondestructive nor destructive (see Note 2, 8.3), the purchaser should specify which it is considered to be.

7. Formation of Inspection Lot

7.1 An inspection lot shall be formed from articles that are of the same kind, that have been produced to the same specification, and that have been coated by a single supplier at one time or at approximately the same time under essentially identical conditions.

8. Sampling

8.1 General—A sample shall be selected from the inspection lot. If the test method to be used is nondestructive, the sample size shall be that directed in 8.2. If the test method is destructive, the sample size shall be that directed in 8.3.

8.2 Nondestructive Tests—For nondestructive testing, the size of the sample shall be that specified for the sampling plan level that is required by the purchaser. The sampling plans are given for Level I in Table 1, for Level II in Table 2, and for Level III in Table 3. If the purchaser does not specify the level, Level II shall be used.

8.3 Destructive Tests—For destructive testing, the size of the sample shall be that specified in Table 4.

**Note 2**—The nature of a destructive test can be such that the tested article can be reclaimed, for example by stripping and reapplying the coating. Other tests can destroy the coating in nonessential locations, in which case the item can still be functional. In these instances the purchaser needs to decide and state whether the tests are to be considered destructive or nondestructive.

**Note 3**—The plan given for destructive tests uses smaller samples than the plans given for nondestructive tests. There may be cases in which destruction of even these smaller quantities is undesirable. For example, the articles may be expensive or the inspection lot may be small. Often in such cases test specimens are coated along with the articles and are used to represent them in the destructive tests. The permission to use test specimens and the requirements covering them and their use should be set forth in the applicable coating specification, purchase order, or other governing document.

8.4 The sample shall be drawn from the inspection lot randomly, that is, in a manner that assures each article an equal chance of being selected regardless of other considerations such as its location in the inspection lot, its appearance, its quality, its location on a fixture during coating, and its chronological relationship to the other articles. Random sampling procedures are given in the Annex of this test method.

9. Inspection and Lot Disposition

9.1 Each article in the sample shall be inspected as directed in the applicable coating standard. If the number of articles that do not conform to a particular requirement is equal to or less than the acceptance number of the sampling plan, the inspection lot is acceptable with respect to that requirement, otherwise the inspection lot is not acceptable (Note 4). Inspection lots that are unacceptable with respect to one or more requirements shall be rejected.

**Note 4**—The acceptability of an inspection lot is determined with respect to each requirement independently from all other requirements.
The acceptance number applies to each requirement in turn; it is not added. For example, if a sample of 50 articles drawn in accordance with Table 2 is found to contain two defectives with respect to thickness and a third one with respect to appearance, the inspection lot is acceptable because although three articles were defective no more than two, the acceptance number, were defective with respect to a single requirement.

10. Resampling

10.1 When required by the purchaser, inspection lots that are rejected for nonconformance to a requirement where conformance can be determined by a nondestructive test may be 100 % inspected by the seller and resubmitted for acceptance after the seller has removed all nonconforming articles and replaced them with conforming articles. The same sampling plan that was used when the lot was first inspected shall be used for the reinspection of the screened lot. The resubmitted lot shall be inspected only for the characteristics for which it was rejected.

ANNEX

(Mandatory Information)

A1. DRAWING OF SAMPLES

A1.1 Random Sampling

A1.1.1 If the articles in a lot are thoroughly mixed, sorted, or arranged without bias as to quality (for example, barrel electroplated articles), a sample drawn anywhere from the lot will meet the requirement of randomness. If the articles are not so mixed, and if it is thought to be impractical to mix them, bias will result if the entire sample is drawn from a single or a few layers. Other bias in sampling, such as taking articles from the same place on a plating rack, taking articles from the output of one electroplating bath and not others, and taking articles that appear to be conforming or to be nonconforming, must be avoided. Bias can be avoided by numbering the articles, randomly selecting a group of numbers equal to the sample size, and inspecting the articles with the selected numbers. A method for doing this is described in the following.

A1.1.2 When random numbers are used to select a sample, each article in the lot is identified by a different number. This may be done by placing the units in racks or trays where the positions in the racks are numbered. If the units have serial numbers, the serial numbers can be used. Random numbers may be obtained from books pertaining to statistics. A table of random numbers (Table A1.1) has been included in this Annex. Some pocket calculators are designed to generate random numbers.

A1.1.3 As an example assume that a sample of 13 articles is to be selected from an inspection lot of 80 articles. The articles are numbered 1 through 80. A pencil is allowed to fall blindly at some number in Table A1.1. Starting at this point, a coin is tossed to decide whether to go up or down the column; heads, up; tails, down. If the pencil falls on column 10, line 11, and the coin is tails, the decision is to read down the column until 13 numbers are chosen. Take the first two digits in each group of five digits. The selection of random numbers is made as follows: the 85s are rejected because they are over 80, and the second 06 is rejected because it has already appeared. The sample then consists of articles numbered 31, 20, 8, 26, 53, 65, 64, 46, 22, 6, 41, 67, and 14.

A1.2 Constant-Interval Sampling

A1.2.1 When product items are arranged in an order without regard to quality, such as articles in a tray, a sample can be drawn by using the constant-interval procedure. Here, a constant interval is maintained between the items drawn for the sample. For example, every 9th, 19th, or 24th unit is selected. The first item drawn from the lot can be determined from the table of random numbers. All other items are then drawn at a constant interval following the first item. The constant interval is determined by dividing the lot size by the sample size and by rounding the quotient down to the nearest whole number.

A1.2.2 As an example assume that a lot of 3000 items is to be visually examined for freedom from such defects as blisters, pits, nodules, porosity, and staining. In accordance with Table 3, a sample of 135 items is to be drawn. The constant interval is 24 (3000 divided by 125). A random number from 1 to 24 is selected either from a table (see A1.1.2) or by another appropriate method. After the first item is taken, the remaining items in the required sample are drawn by selecting every 24th item from the lot until 125 are selected.
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### REFERENCES

4. ibid, Vol 5, No. 3, March 1977, pp. 8–12

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Standard Specification for 
Decorative Electroplated Coatings of Copper Plus Nickel 
Plus Chromium on Plastics

This standard is issued under the fixed designation B 604; the number immediately following the designation indicates the year of 
original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A 
superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope 
1.1 This specification covers the requirements for several 
grades and types of electrodeposited copper plus nickel plus 
chromium coatings on plateable plastic substrates where ap-
pearance, durability and resistance to thermal cycling are 
important to service performance. Five grades of coatings are 
provided to correlate with the service conditions under which 
each is expected to provide satisfactory performance. 

1.2 This specification covers the requirements for coatings 
applied subsequent to the application of metal film by auto-
catalytic deposition or subsequent to the application of any 
strike coatings after autocatalytic deposition. 

1.3 The following caveat pertains only to the test method 
portions of Section 6, Annex A1, and Appendix X2, Appendix 
X3, and Appendix X4 of this specification. This standard does 
not purport to address all of the safety concerns, if any, 
associated with its use. It is the responsibility of the user of this 
standard to establish appropriate safety and health practices 
and determine the applicability of regulatory limitations prior 
to use.

2. Referenced Documents 
2.1 ASTM Standards:
B 368 Method for Copper-Accelerated Acetic Acid-Salt 
Spray (Fog) Testing (CASS Test)2 
B 487 Test Method for Measurement of Metal and Oxide 
Coating Thicknesses by Microscopical Examination of a 
Cross Section2 
B 489 Practice for Bend Test for Ductility of Electrodepos-
ited and Autocatalytically Deposited Metal Coatings on 
Metals2 
B 504 Test Method for Measurement of Thicknesses of Met-
tallic Coatings by the Coulometric Method2 
B 530 Test Method for Measurement of Coating Thic-
nesses by the Magnetic Method: Electrodeposited Nickel 
Coatings on Magnetic and Nonmagnetic Substrates2 
B 532 Specification for Appearance of Electroplated Plastic 
Surfaces2 
B 533 Test Method for Peel Strength of Metal Electroplated 
Plastics2 
B 556 Guide for Measurement of Thin Chromium Coatings 
by the Spot Test2 
B 567 Test Method for Measurement of Coating Thickness 
by the Beta Backscatter Method2 
B 568 Test Method for Measurement of Coating Thickness 
by X-Ray Spectrometry2 
B 602 Test Method for Attribute Sampling of Metallic and 
Inorganic Coatings2 
B 659 Guide for Measuring Thickness of Metallic and 
Inorganic Coatings2 
B 727 Practice for Preparation of Plastics Materials for 
Electroplating2 
B 764 Test Method for Simultaneous Thickness and Elec-
trochemical Potential Determination of Individual Layers 
in Multilayer Nickel Deposit (STEP Test)2 
D 1193 Specification for Reagent Water3 
E 50 Practices for Apparatus, Reagents, and Safety Precau-
tions for Chemical Analysis of Metals, Ores, and Related 
Materials4

3. Terminology 
3.1 Definitions: 
3.1.1 significant surfaces—those surfaces normally visible 
(directly or by reflection) that are essential to the appearance or 
serviceability of the article when assembled in normal position 
or that can be the source of corrosion products that deface 
visible surfaces on the assembled article.

4. Classification 
4.1 Five grades of coatings designated by service condition 
numbers and several types of coatings defined by classification 
numbers are covered by this specification.
4.2 Service Condition Number:

4.2.1 The service condition number indicates the severity of exposure for which the grade of coating is intended, in accordance with the following scale:

- SC5—extended very severe
- SC4—very severe
- SC3—severe
- SC2—moderate
- SC1—mild

4.2.2 Service condition numbers are further defined in Appendix X1 where they are related to the severity of exposure encountered by electroplated articles.

4.3 Coating Classification Number—The coating classification number is a means of specifying the types and thicknesses of coatings appropriate for each grade and is comprised of the following:

4.3.1 The symbol for the substrate (PL) indicating it is plateable plastic, followed by a slash mark,

4.3.2 The chemical symbol for copper (Cu),

4.3.3 A number giving the minimum thickness of the copper coating in micrometres,

4.3.4 A lower-case letter designating the type of copper electrodeposit (see 4.4 and 6.3.1),

4.3.5 The chemical symbol for nickel (Ni),

4.3.6 A number giving the minimum thickness of the nickel in micrometres,

4.3.7 A lower-case letter designating the type of nickel electrodeposit (see 4.4 and 6.3.2),

4.3.8 The chemical symbol for chromium (Cr), and

4.3.9 A lower-case letter or letters designating the type of chromium (see 4.4 and 6.3.3).

4.4 Symbols for Expressing Classification—The following lower-case letters shall be used in coating classification numbers to describe the types of coatings:

- a — ductile copper deposited from acid-type baths
- b — single-layer nickel deposited in the fully-bright condition
- d — double- or triple-layer nickel coatings
- r — regular (that is, conventional) chromium
- mc — microcracked chromium
- mp — microporous chromium

4.5 Example of Complete Classification Number—A coating on plastic comprising 15 µm minimum ductile acid copper plus 15 µm minimum double-layer nickel plus 0.25 µm minimum microporous chromium has the classification number, as given in Table 1. On request, the manufacturer shall inform the purchaser of the classification number of the coating applied.

5. Ordering Information

5.1 When ordering articles to be electroplated in accordance with this standard, the purchaser shall state the following:

5.1.1 ASTM designation number.

5.1.2 Either the classification number of the specific coating required (see 4.3) or the substrate material and the service condition number denoting the severity of the conditions it is required to withstand (see 4.2). If the service condition number is quoted and not the classification number, the manufacturer is free to supply any of the types of coatings designated by the classification number corresponding to the service condition number, as given in Table 1. On request, the manufacturer shall inform the purchaser of the classification number of the coating applied.

5.1.3 The appearance required, for example, bright, dull, or satin. Alternatively, samples showing the required finish or range of finish shall be supplied or approved by the purchaser.

5.1.4 The significant surfaces, to be indicated on drawings of the parts, or by the provision of suitably marked specimens (see 3.1).

5.1.5 The positions on significant surfaces for rack or contact marks, where such marks are unavoidable (see 6.1.1).

5.1.6 The extent to which defects shall be tolerated on nonsignificant surfaces.

5.1.7 The ductility if other than the standard value (see 6.4).

5.1.8 The extent of tolerable surface deterioration after corrosion testing (see 6.6.3).

5.1.9 Sampling methods and acceptance levels (See Section 7).

5.1.10 Whether thermal cycle and corrosion testing shall be conducted individually on separate specimens as described in 6.6 and 6.7, or sequentially using the same specimens as described in 6.8, and whether the specimens shall be unmounted or mounted in a manner simulating assembly when these tests are conducted.

5.2 The minimum values of the electrochemical potential differences between individual nickel layers as measured in accordance with Test Method B 764 within the limits given in 6.10.

6. Product Requirements

6.1 Visual Defects:

6.1.1 The significant surfaces of the electroplated articles shall be free of visible defects, such as blisters, pits, roughness, cracks, and uncoated areas, and shall not be stained or discolored. On articles where a visible contact mark is unavoidable, its position shall be specified by the purchaser. The electroplated article shall be free of damage and clean.

6.10 Differences between individual nickel layers as measured in these tests are conducted.
6.1.2 Defects in the surface of the molded plastic, such as cold shots, ejection marks, flash, gate marks, parting lines, splay and others, may adversely affect the appearance and performance of coatings applied thereto despite the observance of the best electroplating practice. Accordingly, the electroplater’s responsibility for defects in the coating resulting from the plastic-molding operation shall be waived (Note 1).

**Note 1**—To minimize problems of this type, the specifications covering the items to be electroplated should contain appropriate limitations on the extent of surface defects. Practice B 532 distinguishes between defects that arise primarily in molding and those that arise in electroplating operations.

6.2 **Pretreatments**—Proper preparatory procedures are essential for satisfactory performance of electrodeposited coatings on plastics. Procedures described in Practice B 727 may be followed. In the case of patented processes, the instructions provided by the suppliers of those processes shall be followed.

6.3 **Process and Coating Requirements**—Following preparatory operations, plastic articles are placed in electroplating solutions as required to produce the composite coating described by the specific coating classification number or by coating one of the specified classification numbers listed in Table 1 appropriate for the specified service condition number.

6.3.1 **Type of Copper**—Ductile copper shall be deposited from acid-type baths containing organic additives that promote leveling by the copper deposit.

6.3.2 **Type of Nickel**—For double- or triple-layer nickel coatings, the bottom layer shall contain less than 0.005 mass % sulfur (Note 2). The top layer shall contain greater than 0.04 mass % sulfur (Note 3), and its thickness shall not be less than 10 % of the total nickel thickness. In double-layer coatings, the thickness of the bottom layer shall be not less than 60 % of the total nickel thickness. In triple-layer coatings, the bottom layer shall be not less than 50 % nor more than 70 %. If there are three layers, the intermediate layer shall contain not less than 0.15 mass % sulfur and shall not exceed 10 % of the total nickel thickness. These requirements for multilayer nickel coatings are summarized in Table 2.

6.3.3 **Thickness of Chromium Deposit**—The minimum permissible thickness of the chromium deposit shall be 0.25 µm on significant surfaces. The thickness of chromium is designated by the same symbol as the type instead of by numerals as in the case of copper and nickel (see 4.4).

### TABLE 2 Summary of the Requirements for Double- and Triple-Layer Nickel Coatings

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<td>Bottom(s)</td>
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<td>equal to or greater than 50 %</td>
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<td>10 % max</td>
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<tr>
<td>Top (b)</td>
<td></td>
<td>greater than 0.04 %</td>
<td>equal to or greater than 40 %</td>
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Test Method: Appendix X3

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**Note 1**—The sulfur content is specified in order to indicate which type of nickel electroplating solution must be used. Although no simple method is yet available for determining the sulfur content of a nickel deposit on a coated article, chemical determinations are possible using specially prepared test specimens. See Appendix X2 for the determination of sulfur in electrodeposited nickel.

**Note 2**—It will usually be possible to identify the type of nickel by microscopical examination of the polished and etched section of an article prepared in accordance with Test Method B 487. The thickness of the individual nickel layers in double-layer and triple-layer coatings, as well as the electrochemical relationships between the individual layers can be measured by the STEP test in accordance with Test Method B 764.

6.4 **Ductility**—The minimum value of the ductility shall be 8 % for copper and for nickel when tested by the method given in Appendix X3. Greater ductility may be requested but shall be subject to agreement between the purchaser and the manufacturer.

6.5 **Coating Thickness**:

6.5.1 The minimum coating thickness shall be as designated by the coating classification number.

6.5.2 It is recognized that requirements may exist for thicker coatings than are covered by this specification.

6.5.3 The thickness of a coating and its various layers shall be measured at points on the significant surfaces (see 4.2 and Note 4.)

**Note 4**—When significant surfaces are involved on which the specified thickness of deposit cannot readily be controlled, such as threads, holes, deep recesses, bases of angles, and similar areas, the purchaser and the manufacturer should recognize the necessity for either thicker deposits on the more accessible surfaces or for special racking. Special racks may involve the use of conforming, auxiliary, or bipolar electrodes, or nonconducting shields.

6.5.3.1 The coulometric method described in Test Method B 504 may be used to measure thickness of the chromium, the total thickness of the nickel, and the thickness of the copper. The STEP test, Test Method B 764, which is similar to the coulometric method, may be used to determine the thicknesses of individual layers of nickel in a multilayer coating.

6.5.3.2 The microscopical method described in Test Method B 487 may be used to measure the thickness of each nickel layer and of the copper layer.

6.5.3.3 The beta backscatter method described in Test Method B 567 may be used when the total thickness of a copper/nickel/chromium composite coating is to be measured, without any indication of the thickness of each individual layer.

6.5.3.4 Other methods may be used if it can be demonstrated that the uncertainty of the measurement is less than 10 %, or less than that of any applicable method mentioned in 6.4.3. Other methods are outlined in Test Methods B 530 and B 568 and Guides B 556 and B 659.

6.6 **Corrosion Testing**:

6.6.1 Coated articles shall be subjected to the corrosion test for a period of time that is appropriate for the particular service condition number (or for the service condition number corresponding to a specified classification number) as shown in Table 3. The test is described in detail in the referenced ASTM standard.

**Note 5**—There is no direct relation between the results of an accelerated corrosion test and the resistance to corrosion in other media because several factors, such as the formation of protective films, influence the
progress of corrosion and vary greatly with the conditions encountered. The results obtained in the test should, therefore, not be regarded as a direct guide to the corrosion resistance of the tested materials in all environments where these materials may be used. Also, performance of different materials in the test cannot always be taken as a direct guide to the relative corrosion resistance of these materials in service.

6.6.2 After subjecting the article to the treatment described in the relevant test method, it shall be examined for evidence of corrosion penetration to the substrate or the copper layer, and for blistering of the coating. Any evidence of copper corrosion, blistering of the coating, or substrate exposure shall be cause for rejection. It is to be understood that occasional widely scattered corrosion defects may be observed after the testing period. In general, “acceptable resistance” shall mean that such defects are not, when viewed critically, significantly defacing or otherwise deleterious to the function of the electroplated part.

6.6.3 Surface deterioration of the coating itself is expected to occur during the testing of some types of coatings. The extent to which such surface deterioration will be tolerated shall be specified by the purchaser.

6.7 Thermal Cycle Testing:

6.7.1 Coated articles shall be subjected to three cycles of the thermal cycle test as outlined in Annex A1. The specified service condition number of the coating (or the service condition number corresponding to the specified classification number) shall correspond to the service condition number in Annex A1 for determining the temperature extremes as outlined therein.

6.7.2 After having been subjected to three cycles of the appropriate thermal cycle test, the coated article shall show no visible defects, such as cracking, blistering, peeling, sink marks, and distortions.

Note 6—There is no direct relation between the results of thermal cycle testing and performance in service, because it is not always possible to predict and control the thermal exposure of the coated article in service or during storage. Therefore, the results of thermal cycling should be used to control the quality of electroplated plastic articles and not as direct guide to performance in service.

6.8 Combined Thermal Cycle and Corrosion Testing:

6.8.1 Corrosion testing may be combined with thermal cycle testing for articles electroplated according to the requirements of SC5, SC4, and SC3 by using the same coated articles in each test in sequence as described in this section. The use of combined thermal cycle and corrosion testing obviates the need to conduct the individual tests described in 6.6 and 6.7.

6.8.2 Expose the coated articles to one 16-h cycle according to the procedures outlined in Method B 368 (CASS test).

6.8.3 Parts shall be rinsed with demineralized water only after each CASS test cycle.

6.8.4 Subject the electroplated articles to the thermal cycle test procedure given in Annex A1.

6.8.5 Steps 6.8.2 through 6.8.4 represent one cycle of combined thermal cycle and corrosion testing. For articles electroplated to SC5 or SC4, repeat for two additional times. For articles electroplated to SC3, repeat one additional time.

6.8.6 Coated articles shall be examined for defects after each cycle of combined thermal cycle-corrosion testing as indicated in 6.6.2 and 6.7.2.

6.9 Adhesion—Test Method B 533 provides a procedure for measurement of the peel strength (adhesion) of metal-electroplated plastics using standard specimens. Since there is no direct correlation between results obtained on standard specimens and actual molded parts, the method is useful to determine that processing solutions are capable of giving acceptable results. The thermal cycle test described in 6.7 and the subsequent examination of the electroplated articles described in 6.7.2, or alternatively, the combined thermal cycle test described in 6.7.2, or alternatively, the combined thermal cycle and corrosion tests described in 6.8, are recommended instead of other tests.

6.10 STEP Test Requirement:

6.10.1 The electrochemical potential differences between individual nickel layers shall be measured for multilayer coatings corresponding to SC5, SC4, and SC3 in accordance with Test Method B 764 (STEP test). See Note 7.

Note 7—Universally accepted STEP values have not been established but some agreement exists for the required ranges. The STEP values depend on which two nickel layers are being measured: (a) the STEP potential difference between the semi-bright nickel layer and the bright nickel layer is within the range of 100 to 200 mV. For all combinations of nickel layers, the semi-bright nickel layer is more noble (cathodic) than the bright nickel; (b) the STEP potential difference between the high-activity nickel layer and the bright nickel layer in triple-layer nickel coatings is within the range of 15 to 35 mV. The high-activity layer is more active (anodic) than the bright nickel layer; and (c) the STEP potential difference between the bright nickel layer and a nickel layer between the bright nickel layer and the chromium layer is within 0 to 30 mV. The bright nickel layer is more active (anodic) than the nickel layer applied prior to the chromium.

6.11 Sulfur Content:

6.11.1 The sulfur content of the nickel deposit shall meet the maximum or minimum values as stated in 6.3.2 and Table 2.

6.11.2 Methods for sulfur determinations are given in Appendix X2.

6.12 Density and Measurement of Discontinuities in Chromium:

6.12.1 The density of cracks or pores in microcracked or microporous chromium deposits shall meet minimum values. Microcracked chromium shall have more than 30 cracks/mm in any direction over the whole of the significant surface. Microporous chromium shall contain a minimum of 100 pores/mm² in any direction over the whole of the significant surface. The cracks and pores shall be invisible to the unaided eye.

6.12.2 Methods for measuring the discontinuities are given in Appendix X4. See X4.4 for a means of determining corrosion sites by corrosion testing.
7. Sampling Requirement

7.1 A random sample of the size required by Test Method B 602 shall be selected from the inspection lot (see 7.2). The articles in the lot shall be inspected for conformance to the requirements of this specification and the lot shall be classified as conforming or not conforming to each requirement according to the criteria of the sampling plans in Test Method B 602.

Note 8—Test Method B 602 contains three sampling plans for the original inspection of coated articles. Two are to be used where the test methods are nondestructive, that is, the test method does not make the article nonconforming. The third plan is used where the test method is destructive. If it is not clear if the test is destructive or not, the purchaser should identify which test methods are destructive, and which are nondestructive. In some instances, both nondestructive and destructive test methods may exist for the testing of the conformance of a coating to a particular requirement. The purchaser should state which is to be used.

7.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specifications, that have been coated by a single supplier at one time, or at approximately the same time, under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

ANNEX
(Mandatory Information)

A1. Thermal Cycling of Electroplated Plastics

Note A1.1—This test method is used to ensure compliance of electroplated plastics with the thermal cycle requirements given in 6.7 and 6.8.

A1.1 Apparatus—The apparatus shall consist of a circulating air heating chamber and cooling chamber sufficiently powered, insulated, and controlled to closely maintain the preset temperature. The two chambers may be separate, or may be built so as to constitute a single chamber. The controller and recorder used for chamber control, calibration, and records shall be accurate to ±1°C. All points within the working area of the test chamber shall remain within ±3°C of the set temperature. The air circulation shall be controlled to permit a consistent rate of heating or cooling of the parts during the test.

A1.2 Elapsed Time After Electroplating—The elapsed time between completion of the electroplating operation and thermal cycle testing may influence the results. The elapsed time shall be 24 ± 2 h.

A1.3 Procedure:
A1.3.1 Parts may be introduced into the chamber unmounted, or mounted in a manner simulating assembly as specified by the purchaser.
A1.3.2 Load the chamber with the desired quantity of parts to be tested.
A1.3.3 Record the location of parts within the chamber, the loading and the size of the parts being tested.
A1.3.4 The thermal cycle temperature limits corresponding to the specified service condition number shall be chosen from Table A1.1.
A1.3.5 Each thermal cycle shall consist of either placing the samples in a room-temperature chamber and heating the chamber to the high limit, or placing the samples directly into a chamber at the high limit, and performing the following:
A1.3.5.1 Expose the parts for one hour at the high limit.
A1.3.5.2 Allow the parts to return to 20 ± 3°C and maintain at this temperature for 1 h. This is frequently accomplished by removing the parts from the chamber.
A1.3.5.3 Expose the parts for one hour at the low limit.
A1.3.5.4 Allow the parts to return to 20 ± 3°C and maintain at this temperature for 1 h. Steps A1.3.5.1 through A1.3.5.4 constitute one full thermal cycle.
A1.3.6 When the number of cycles specified in 6.7 and 6.8 has been completed, inspect the parts for coating defects produced by thermal cycling. See Specification B 532, Table 1, for the limits established for visual defects.
A1.4 Recording of Test Results—The recording of the test results shall include the following:

| TABLE A1.1 Recommended Thermal Cycling Temperature Limits |
|-------------------------------|------------------|
| Service Condition Number      | Temperature Limits, °C |
| SC5 — Extended very severe    | 85 -40            |
| SC4 — Very severe             | 80 -40            |
| SC3 — Severe                  | 80 -30            |
| SC2 — Moderate                | 75 -30            |
| SC1 — Mild                    | 60 -30            |
A1.4.1 A statement that the test was performed according to Specification B 604, Annex A1.
A1.4.2 The service condition number for which the part was tested.
A1.4.3 The tray construction (if a tray is used) and chamber loading.
A1.4.4 The last calibration date of the controller and records.
A1.4.5 The extent, nature, and location of the defects.
A1.5 Precision and Bias—The precision and bias of this test method have not been established.

APPENDIXES

(Nonmandatory Information)

X1. DEFINITIONS AND EXAMPLES OF SERVICE CONDITIONS FOR WHICH THE VARIOUS SERVICE CONDITION NUMBERS ARE APPROPRIATE

X1.1 Service Condition No. SC 5 (Extended Very Severe)—Service conditions that include likely damage from denting, scratching, and abrasive wear in addition to exposure to corrosive environments where long-term protection of the substrate is required; for example, conditions encountered by some exterior components of automobiles.

X1.2 Service Condition No. SC 4 (Very Severe)—Service conditions that include likely damage from denting, scratching, and abrasive wear in addition to exposure to corrosive environments; for example, conditions encountered by exterior components of automobiles and by boat fittings in salt water service.

X1.3 Service Condition No. SC 3 (Severe)—Exposure that is likely to include occasional or frequent wetting by rain or dew or possibly strong cleaners and saline solutions; for example, conditions encountered by porch and lawn furniture, bicycle and perambulator parts, hospital furniture and fixtures.

X1.4 Service Condition No. SC 2 (Moderate)—Indoor exposure in places where condensation of moisture may occur; for example, in kitchens and bathrooms.

X1.5 Service Condition No. SC 1 (Mild)—Indoor exposure in normally warm, dry atmospheres with coating subject to minimum wear or abrasion.

X2. DETERMINATION OF SULFUR IN ELECTRODEPOSITED NICKEL

The following two methods for the determination of sulfur in electroplated nickel are given as guidelines for use to test compliance of the type of nickel deposit with the appropriate definition given in 6.3.2. They represent methods that have been used with success commercially; they are not ASTM standards, nor is it the intent in publishing these methods to preclude the use of other methods or variations in these methods.

X2.1 Total Sulfur in Electroplated Nickel by Combustion-Iodate Titration

X2.1.1 Scope—This method covers the determination of sulfur in concentrations from 0.005 to 0.5 mass %.

X2.1.2 Summary of Method—A major part of the sulfur in the sample is converted to sulfur dioxide (SO₂) by combustion in a stream of oxygen using an induction furnace. During the combustion, the SO₂ is absorbed in an acidified starch-iodide solution and titrated with potassium iodate solution. The latter is standardized against steels of known sulfur content to compensate for characteristics of a given apparatus and for day-to-day variation in the percentage of sulfur recovered as SO₂. Compensation is made for the blank because of accelerators and crucibles.

Note X2.1—Instruments are available for measuring the sulfur dioxide from combustion by infrared detection methods and using built-in computers to integrate and display the sulfur content as a percentage.

X2.1.3 Interferences—The elements ordinarily present in electroplated nickel do not interfere.

X2.1.4 Apparatus—Induction heating apparatus for determination of sulfur by direct combustion as described in Practices E 50 (Apparatus No. 13).

X2.1.5 Reagents:

X2.1.5.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first determined that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

X2.1.5.2 Purity of Water—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D 1193.

X2.1.5.3 Hydrochloric Acid (3 + 97)—Mix 3 volumes of concentrated hydrochloric acid (HCl) (sp gr 1.19) with 97 volumes of water.

X2.1.5.4 Iron (Low-Sulfur) Accelerator—Chips.

X2.1.5.5 Iron (Low-Sulfur) Accelerator—Powder.  
X2.1.5.6 Potassium Iodate, Standard Solution A (1 mL = 0.1 mg S)—Dissolve 0.2225 g of potassium iodate (KIO₃) in 900 mL of water and dilute to 1 L.  
X2.1.5.7 Potassium Iodate, Standard Solution B (1 mL = 0.02 mg S)—Transfer 200 mL of potassium iodate Solution A (1 mL = 0.1 mg S) to a 1-L volumetric flask, dilute to volume, and mix.

Note X2.2—The sulfur equivalent is based on the complete conversion of sulfur to sulfur dioxide. The recovery of sulfur as the dioxide may be less than 100%, but it is consistent when the temperature and the rate of oxygen flow are maintained constant. An empirical factor must be determined by an analysis of a standard sample.

X2.1.5.8 Starch-Iodide Solution—Transfer 1 g of soluble or arrowroot starch to a small beaker, add 2 mL of water, and stir until a smooth paste is obtained. Pour the mixture into 50 mL of boiling water. Cool, add 1.5 g of potassium iodide (KI), stir until dissolved, and dilute to 100 mL.

X2.1.5.9 Tin (Low-Sulfur) Accelerator—Granular.

X2.1.6 Standards—Standards for calibration are National Institute of Standards and Technology (formerly National Bureau of Standards) steels of the proper sulfur content.

X2.1.7 Sample Preparation:

X2.1.7.1 Prepare a test panel of cold-rolled steel 150 mm long by 100 mm wide by 1 mm thick or any other convenient size. Clean, acid dip, and electroplate with approximately 7.5 µm of an adherent nickel deposit and thoroughly rinse. Buffed nickel or buffed stainless steel may also be used as alternatives to steel electroplated with nickel.

X2.1.7.2 Passivate the test panel anodically at 3 V for 5 to 10 s in a hot alkaline cleaner (temperature 70 to 80°C) containing 30 g/L of sodium hydroxide (NaOH) and 30 g/L of trisodium phosphate (Na₃PO₄) or 60 g/L of any other suitable anodic alkaline cleaner.

X2.1.7.3 Coat the passivated test panel with 25 to 37 µm of nickel deposited from the same solution using the same parameters as for the coated articles represented by the test specimen.

X2.1.7.4 Remove the edges of the electroplated panel with a hand or power shear or any other convenient method that permits ready separation of the test foil.

X2.1.7.5 Separate from the panel, wash the nickel foil electroplate with water to remove salts, and blot dry. Cut into pieces 2 to 3 mm per side with a scissors. Transfer to a 100-mL beaker, cover with water, and heat to boiling. Pour off the water pieces 2 to 3 mm per side with a scissors. Transfer to a 100-mL beaker, cover with water, and heat to boiling. Pour off the water

X2.1.7.6 Transfer 200 mL of potassium iodate Solution A (1 mL = 0.1 mg S) to a 1-L volumetric flask, dilute to volume, and mix.

X2.1.7.7 Dissolve 0.2225 g of potassium iodate as follows:

\[ \text{Sample weight, g} = \frac{A \times B}{(C - D) \times 100} \]  

where:

\( A \) = standard sample used, g
\( B \) = sulfur in the standard sample, %
\( C \) = KIO₃ solution required for titration of the standard sample (Note X2.5), mL, and
\( D \) = KIO₃ solution required for titration of the blank, mL (Note X2.5).

Note X2.3—Always fill the titration vessel to the same point.
Note X2.4—The oxygen flow rate may be adjusted to meet the requirements of individual operators or equipment; however, the flow rate must be the same for the test samples and the standard samples.

X2.1.7.8 After the unit has been at operating temperature for at least 45 s, place the covered crucible containing the sample and accelerators on the pedestal. With the oxygen flow adjusted, raise the crucible, close the furnace, and turn on the power. Burn the sample for 8 to 10 min. Titrate continuously with the KIO₃ solution at such a rate as to maintain as nearly as possible the original intensity of the blue color. The end point is reached when the original blue color is stable for 1 min. Record the final buret reading and drain the titration vessel through the exhaust stopcock.

X2.1.7.9 Blank—Determine the blank by placing the same amount of accelerators used in the test sample in a preignited crucible. Cover and proceed as in X2.1.10.3.

X2.1.11 Calculation—Calculate the sulfur factor of the potassium iodate as follows:

\[ \text{Sulfur factor, g/unit volume} = \frac{A \times B}{(C - D) \times 100} \]  

X2.1.11.1 Calculate the percentage of sulfur in the test sample as follows:

\[ \text{Sulfur, mass %} = \frac{(E - D)F}{G} \times 100 \]  

where:

\( E \) = weight of sample, g
\( F \) = weight of sample, g
\( G \) = weight of sample, g

Note X2.5—Use apparent percentage of sulfur for “direct-reading” burets.

X2.1.11.2 Calculate the percentage of sulfur in the test sample as follows:
where:
\[ E = \text{KIO}_3 \text{ solution required for titration of the test sample, mL (Note X2.5),} \]
\[ D = \text{KIO}_3 \text{ solution required for titration of the blank, mL,} \]
\[ F = \text{average sulfur factor of the KIO}_3, \text{ g/unit volume, for the standards used (see X2.1.11), and} \]
\[ G = \text{sample used, g.} \]

**X2.2 Determination of Sulfur in Electroplated Nickel by the Evolution Method**

**X2.2.1 Scope**—This method covers the determination of sulfide sulfur in electroplated nickel in the range from 0.005 to 0.2 mass %.

**X2.2.2 Summary of Method**—Sulfide sulfur is evolved as hydrogen sulfide (H\(_2\)S) on dissolving the sample of hydrochloric acid (HCl) containing a small amount of platinum as an accelerator for dissolution. The sulfur is precipitated as zinc sulfide (ZnS) in the receiving vessel and then titrated with standard potassium iodate solution. Values are based on potassium iodide (KIO\(_3\)) as the primary standard.

**X2.2.3 Apparatus:**

X2.2.3.1 The apparatus is shown in Fig. X2.1. It may be assembled using a 50-mL Erlenmeyer flask with a No. 19/38 outer joint. A wash bottle fitted with a No. 19/38 inner joint can be cut to fit the 50-mL flask. The exit tube can be bent and connected to the 6-mm gas tube with tubing.

X2.2.3.2 A nitrogen cylinder with valves and pressure regulator.

X2.2.3.3 Buret, 10-mL.

X2.2.4 Reagents:

X2.2.4.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

X2.2.4.2 Purity of Water—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D 1193.

X2.2.4.3 Ammoniacal Zinc Sulfate Solution—Dissolve 50 g of zinc sulfate (ZnSO\(_4\)·7H\(_2\)O) in 250 mL of water, add 250 mL of ammonium hydroxide (NH\(_4\)OH sp gr 0.90) and mix. Transfer to a flask and allow to stand about 24 h and filter into a polyethylene bottle.

X2.2.4.4 Hexachloroplatinic Acid Solution (10 g/L)—Dissolve 0.5 g of hexachloroplatinic acid (H\(_2\)PtCl\(_6\)·6H\(_2\)O) in about 40 mL of water, add 5 mL of hydrochloric acid (HCl sp gr 1.19), and dilute to 50 mL.

X2.2.4.5 Hydrochloric Acid-Platinum Chloride Solution—Prepare 500 mL of diluted hydrochloric acid (HCl sp gr 1.19, 1 part acid in 1 part water). Add 2.5 mL of the hexachloroplatinic acid solution and mix.

X2.2.4.6 Potassium Iodate, Standard Solution (0.1 N)—Dry the crystals of potassium iodate (KIO\(_3\)) at 180°C for 1 h. Dissolve 3.570 g of the KIO\(_3\) in about 200 mL of water, transfer to a 1-L volumetric flask, dilute to volume, and mix.

X2.2.4.7 Potassium Iodate, Standard Solution (0.005 N)—Transfer 25 mL of 0.1 N KIO\(_3\) solution to a 500-mL volumetric flask with a pipet, dilute to volume, and mix.

X2.2.4.8 Starch Solution (10 g/L)-Potassium Iodide (50 g/L) Solution—Add about 5 mL of water to 1 g of soluble starch with stirring until a paste is formed and add to 100 mL of boiling water. Cool, add 5 g of potassium iodide (KI), and stir until the KI is dissolved.

X2.2.5 Sample Preparation—Prepare sample as outlined in X2.1.7.

X2.2.6 Weight of Sample—Select and weigh to the nearest 0.1 mg an amount of sample as follows:

<table>
<thead>
<tr>
<th>Expected Sulfur Content, mass %</th>
<th>Weight of Sample, g ± 0.02</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.005 to 0.07</td>
<td>1.0</td>
</tr>
<tr>
<td>0.05 to 2</td>
<td>0.4</td>
</tr>
</tbody>
</table>

X2.2.7 Procedure:

X2.2.7.1 Weigh the specified amount of sample to the nearest 0.1 mg and transfer to the 50-mL evolution flask.

X2.2.7.2 Add 20 mL of water and 3 mL of ammoniacal zinc sulfate solution to the receiving flask.

X2.2.7.3 Adjust the hot plate to maintain the temperature of 25 mL of water in a 50-mL Erlenmeyer flask at 80°C.

X2.2.7.4 Add 15 mL of the hydrochloric acid-hexachloroplatinic acid solution to the sample. Assemble the apparatus as shown in Fig. X2.1 and start a very gentle stream of nitrogen through the system.

**NOTE X2.6—**A flow of about 30 cm\(^3\)/min is satisfactory. If the sample dissolves rapidly, the flow should be decreased during the time hydrogen is freely liberated.

X2.2.7.5 Continue the heating and flow of nitrogen until the sample is completely dissolved, then continue for 5 min (Note X2.6). Separate the gas delivery tube from the evolution head and remove the receiving flask with the delivery tube.

---

NOTE X2.7—The solution in the receiving flask will remain alkaline throughout the dissolution period if the hot plate temperature and the nitrogen flow are properly adjusted. Additional ammoniacal zinc sulfate solution may be added, if necessary, but the sample should be discarded if the receiving solution becomes acidic (less than pH 7 by test paper).

X2.2.7.6 Add 1 mL of the starch-iodide solution and 5 mL of diluted HCl (1 + 1) and mix. Titrate immediately with standard potassium iodate from a 10-mL buret to the first blue color. Draw some of the solution into the delivery tube with a rubber bulb and release along the neck of the flask to wash down any adhering zinc sulfide. Swirl the solution to wash the outside of the tube. Continue the titration to a permanent blue color.

X2.2.7.7 Run a blank titration to the same starch-iodide color on a mixture of 20 mL of water, 3 mL of ammoniacal zinc sulfide, 1 mL of starch-iodide solution, and 5 mL of diluted hydrochloric acid (1 part HCl sp gr 1.19 and 1 part water) in a 50-mL Erlenmeyer flask.

X2.2.8 Calculation—Calculate the mass percent of sulfide sulfur as follows:

\[
\text{Sulfide sulfur, mass \%} = \frac{(A - B) \times 0.005 \times 0.016 \times 100}{W}
\]

where:

\[
A = 0.005 \text{ N KIO}_3 \text{ solution used for the sample titration, mL,}
B = 0.005 \text{ N KIO}_3 \text{ solution used in the blank, mL, and}
W = \text{sample used, g.}
\]

X3. DUCTILITY TEST

NOTE X3.1—This test is used to test ensure compliance of the type of copper and nickel deposit with the appropriate definition given in 6.4. Refer to Practice B 489 for details on calculation of percent ductility.

X3.1 Preparation of Test Piece:

X3.1.1 Prepare a plated test strip 150 mm long, 10 mm wide, and 1 mm thick by the following method:

X3.1.1.1 Polish a sheet of the appropriate basis metal, similar to that of the articles being electroplated, except that if the basis metal is zinc alloy the sheet may be of soft brass. (Use a sheet sufficiently large to allow the test strip to be cut from its center after trimming off a border 25 mm wide all around.) Electroplate the polished side of the sheet with copper or nickel to a thickness of 25 µm under the same conditions and in the same bath as the corresponding articles.

X3.1.1.2 Cut the test strip from the electroplated sheet with a flat shear. Round or chamfer the longer edges of the test strip, at least on the electroplated side, by careful filing or grinding.

X3.2 Procedure—Bend the test strip with the electroplated side in tension (on the outside), by steadily applying pressure, through 180° over a mandrel of 11.5-mm diameter until the two ends of the test strip are parallel. Ensure that contact between the test strip and the mandrel is maintained during bending.

X3.3 Assessment—The electroplating is deemed to comply with the minimum requirement of an elongation of 8 % if after testing there are no cracks passing completely across the convex surface. Small cracks at the edges do not signify failure.

X4. DETERMINING THE NUMBER OF DISCONTINUITIES IN CHROMIUM ELECTROPLATING (DUBPERNELL TEST)

X4.1 Principle of the Method—Copper will be deposited on nickel exposed through discontinuities in chromium but not on the chromium, provided that potential is properly controlled (kept low enough to avoid activation of passive chromium).

X4.2 Preparation of Test Piece:

X4.2.1 Mask all edges not covered by the chromium with a nonconductive paint or pressure sensitive tape, including the wire used to make contact to the cathode bar. After masking, clean the specimen by soaking in a hot alkaline cleaner until the surface is free of water breaks. A mild scrubbing with a soft brush is helpful. Follow the cleaning by a thorough rinse in cold deionized water, then a dip in a 5 % by mass solution of H₂SO₄.

X4.2.2 Make freshly cleaned sample anodic at 0.8 V for 30 s in the copper plating bath, then switch to cathodic (see Fig. X4.1) at approximately 0.2 to 0.4 V, for 2 min (see Note X4.1 and Note X4.2). (Warning—Do not go beyond the specified anodic voltage or time because nickel will slowly dissolve or become passivated.)

X4.2.3 Following copper electroplating, carefully remove the specimen, rinse in cold then hot deionized water, and air dry. The specimen should not be wiped where pores or cracks are to be counted, nor should the part be force air dried. Drying can be accelerated by following the last water rinse by a rinse with alcohol (ethanol) or other volatile water miscible solvent.

X4.2.4 The copper deposits only on the underlying nickel that is exposed through discontinuities (pores and cracks) in the chromium.

X4.3 Assessment:

X4.3.1 The number of discontinuities in the chromium can be estimated by counting the copper nodules deposited within a known area of the specimen or the number of cracks in a known length. These determinations are facilitated with a metallurgical microscope fitted with a calibrated reticle in the
eyepiece, or from the photomicrographs taken of a representative field of the specimen. (See X4.4 for a guide to the determination of active corrosion sites in the chromium layer.)

X4.3.2 Current measured or recorded during the cathodic cycle, or both, serves as a reliable indicator of porosity. If current remains low (<1 mA/cm²) during the cathodic cycle, porosity is low. Rapidly rising current (ΔI/Δt ≈ 1 to 2 mA/min and high (2 to 4 mA/cm²) final current is indicative of high porosity. Use of a strip chart recorder provides a permanent record of the test current. With experience, direct counts of nodules of Cu deposited can be reduced to periodic verifications as the $I·t$ signature. A qualitative visual check (microscopically) will then suffice for regular routine use.

**NOTE X4.1**—Exact potential used is dependent on anode-cathode spacing. At a distance of 8 to 10 cm, 0.2 V usually produces the desired deposit. As spacing increases, the potential can be increased to 0.4 V.

**NOTE X4.2**—After cleaning, anodic treatment to repassivate chromium is essential. Plating time can be varied from 1 to 5 min. Two minutes has been found to be near optimum. With highly porous chromium, longer times incur risk of merging the deposit nodules, giving rise to ambiguities in counting pores (nodules).

**X4.3.3** **Warning**—Do not exceed 0.6 V cathodic. High cathodic potentials can activate chromium locally, giving rise to spurious high nodule counts. If this condition is suspected, it can be tested by gently wiping the copper off with a tissue. If copper adheres to specimen, it is probable the cathodic potential was too high, thus depositing copper on the chromium instead of just in the pores.

**X4.4** **Determination of Active Corrosion Sites By Corrosion Testing**:

**X4.4.1** Before testing, the part should be cleaned to eliminate water breaks. Magnesium oxide, warm water and soap, or solvents, or a combination thereof, might be necessary for thorough cleaning. After cleaning, examine the part under magnification to determine pore count and size. A magnification between 100 and 200 X is convenient for the size of pores typically found in microdiscontinuous chromium layers. If possible, photograph the part under magnification as a reference. Different photographs should be taken under magnification of all the significant surfaces and current densities to record the difference in pore count and size after corrosion.

**X4.4.2** To develop the active corrosion sites, subject the part to between 16 and 24 h of CASS testing. For convenience, subject the part to one cycle of CASS as determined by the corrosion specification for the part. After CASS testing, rinse the part in warm water to remove the salt layer. If the part contains corrosive product staining, it can be washed with a very mild sponge in warm water but the part should not be subjected to any abrasive cleaning. After drying either by hot air or solvent such as methanol, the part should be viewed again under the same magnification as previously used and in the same areas in which the pictures were taken. By comparing the pictures of these areas before and after corrosion, it is typically easy to distinguish between the pore sites that have started to corrode and those that have not. The corroding pore sites are typically distinctly larger than the uncorroded sites and have a darker and rougher texture. By means of the photograph at a known magnification, the active pore sites can be counted and the active sites per area can be calculated.

**X4.4.3** Even though the necessary work has not been conducted to establish a correlation between active corrosion sites and starting sites as measured by Dubpernell, it appears that for a given current density and deposit system there is a correlation between the number of pores before corrosion and the number of active corrosion sites after one cycle of CASS testing. Once this correlation has been established at a given installation, a simple 100 to 200 X viewing of a part might nondestructively indicate the service performance of the part after corrosion. The desired number of active corrosion sites has not been definitely established. This number depends
somewhat on the size of the pores before and after corrosion, the thickness and potentials of the deposit system, and the desired trade-off between basis metal corrosion and staining in CASS.

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Standard Specification for Electrodeposited Coatings of Tin-Nickel Alloy

This standard is issued under the fixed designation B 605; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers the requirements for electrodeposited tin-nickel alloy coatings from aqueous solutions intended for the corrosion protection of fabricated articles of iron, steel, zinc-base alloys, copper, and copper alloys. The composition of the alloy remains constant at 65/35 tin-nickel in spite of wide fluctuations in both composition and operating conditions. The composition corresponds quite closely to an equiatomic ratio, and the process favors the co-deposition of tin and nickel atoms at identical rates.

1.2 This specification does not apply to sheet, strip, or wire in the fabricated form. It also may not be applicable to threaded articles having basic major diameters up to and including 19 mm because of the nonuniformity of thickness that can be expected on fine threads. However, a decision to use the coating on such components may be made by the purchaser.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards: B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 246 Specification for Tinned Hard-Drawn and Medium-Hard-Drawn Copper Wire for Electrical Purposes
B 252 Guide for Preparation of Zinc Alloy Die Castings for Electroplating and Conversion Coatings
B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings
B 322 Practice for Cleaning Metals Prior to Electroplating
B 374 Terminology Relating to Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
B 507 Practice for Design of Articles to Be Electroplated on Racks
B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 634 Specification for Electrodeposited Coatings of Rhodium for Engineering Use
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 762 Test Method of Variables Sampling of Metallic and Inorganic Coatings
B 765 Guide for Selection of Porosity and Gross Defect Tests for Electrodeposits and Related Metallic Coatings
B 809 Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor (Flowers-of-Sulfur)
B 849 Specification for Pre-Treatments of Iron or Steel for Reducing Risk of Hydrogen Embrittlement
B 850 Guide for Post-Coating Treatments of Steel for Reducing the Risk of Hydrogen Embrittlement
D 3951 Practice for Commercial Packaging

3. Terminology

3.1 Definitions:

3.1.1 Many terms used in this standard are defined in Terminology B 374.

3.1.2 significant surface—that portion of a coated article’s surface where the coating is required to meet all the requirements of the coating specification for that article. Significant surfaces are those that are essential to the serviceability or
function of the article, or which can be a source of corrosion products or tarnish films that interfere with the function or desirable appearance of the article. Significant surfaces are those surfaces that are identified by the purchaser by, for example, indicating them on an engineering drawing of the product or marking a sample item of the product.

3.1.3 undercoating—a metallic coating layer between the basis metal or substrate and the topmost metallic coating. The thickness of an undercoating is usually greater than 0.8 µm. This is in contrast to strikes or flashes, whose thicknesses are generally lower.

4. Classifications

4.1 Coating Grades—Six grades of coatings, designated by service condition numbers, are covered by this specification. For each coating grade a coating thickness grade is specified (see Tables 1-3).

4.2 Service Condition Number—The service condition number indicates the severity of exposure for which the grade of coating is intended.

   SC0—mild service (copper and copper alloys only)
   SC1—mild service
   SC2—moderate service
   SC3—severe service
   SC4—very severe service
   SC5—extended severe service

   

4.3 Coating Thickness Notation—The coating thickness is specified for each service condition in the following manner: Basis metal/Undercoating (thickness)/Sn-Ni (thickness). For example, Fe/Cu4/Sn-Ni25 would indicate a 25 µm tin-nickel coating over an iron or steel article with a 4-µm thick copper undercoating. All thickness notations are minimum thicknesses.

5. Ordering Information

5.1 To make the application of this standard complete, the purchaser needs to supply the following information to the seller in the purchase order or other government documents.

5.1.1 The name, designation, and date of issue of this standard.
5.1.2 Location of significant surface(s) (see section 3.1.2),
5.1.3 The service number or coating thickness notation (see 4.2 and 4.3),
5.1.4 Undercoating, if required (see 6.2 and Tables 1-3),
5.1.5 Any requirement for submission of sample coated articles (see 7.2.1),
5.1.6 Whether or not location of rack marks is to be defined (see 7.2.1),
5.1.7 Any requirement for porosity testing and the criteria (see 7.5.2),
5.1.8 Heat treatment for stress relief, whether it has been performed by the purchaser, or is required (see 7.6),
5.1.9 Heat treatment after electroplating, if required (see 7.7),
5.1.10 Any packaging requirement (see section 7.8),
5.1.11 Inspection procedure to be used (see Section 9),
5.1.12 Any requirement for certification (see Section 11), and
5.1.13 Any requirement for test specimens (see 8.1.1).

6. Material and Process

6.1 Composition of Coating—Electrolytes that have been investigated for producing Sn-Ni alloy deposits include cyanide, fluoroborate, pyrophosphate, and acetate, but the only one in general commercial use is the fluoride-chloride formulation. The deposit contains 35 ± 5 % nickel with the remainder tin (see Note 2).

Note 2—The electrodeposited tin-nickel coating is a single-phase, metastable compound, corresponding approximately to the formula SnNi. It is stable at ordinary temperatures but starts to recrystallize at elevated temperatures. The safe working temperature of the coating is 300°C, although actual melting does not commence below 800°C. The coating is hard (700HV100). Like many such compounds, it is inherently somewhat brittle, but if it is free of internal stresses, the brittleness is not sufficient to impair its serviceability or to cause the coating to flake under impact. Because of the brittleness of the tin-nickel, however, it is not possible to fabricate parts by bending coated sheet material, because the compressive resistance of the article is intended.

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5.1.6 Whether or not location of rack marks is to be defined (see 7.2.1),
5.1.7 Any requirement for porosity testing and the criteria (see 7.5.2),
5.1.8 Heat treatment for stress relief, whether it has been performed by the purchaser, or is required (see 7.6),
5.1.9 Heat treatment after electroplating, if required (see 7.7),
5.1.10 Any packaging requirement (see section 7.8),
5.1.11 Inspection procedure to be used (see Section 9),
5.1.12 Any requirement for certification (see Section 11),
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## Table 1: Tin-Nickel Coatings on Steel

<table>
<thead>
<tr>
<th>Service Condition Number</th>
<th>Thickness Notation</th>
<th>Minimum Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 Fe/Cu4/Sn-Ni</td>
<td>as specified</td>
<td></td>
</tr>
<tr>
<td>4 Fe/Cu4/Sn-Ni</td>
<td>45</td>
<td>45</td>
</tr>
<tr>
<td>3 Fe/Cu4/Sn-Ni</td>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td>2 Fe/Cu4/Sn-Ni</td>
<td>15</td>
<td>15</td>
</tr>
<tr>
<td>1 Fe/Cu4/Sn-Ni</td>
<td>8</td>
<td>8</td>
</tr>
</tbody>
</table>

A Copper undercoat shall be at least 4.0 µm.

B Thickness of Sn-Ni shall be stated in a Thickness Notation. A statement of Service Condition 5 is not sufficient.

## Table 2: Tin-Nickel Coatings on Copper or Copper Alloys

<table>
<thead>
<tr>
<th>Service Condition Number</th>
<th>Thickness Notation</th>
<th>Minimum Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 Cu/Sn-Ni</td>
<td>as specified</td>
<td></td>
</tr>
<tr>
<td>4 Cu/Sn-Ni</td>
<td>(above 45)</td>
<td></td>
</tr>
<tr>
<td>3 Cu/Sn-Ni</td>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td>2 Cu/Sn-Ni</td>
<td>15</td>
<td>15</td>
</tr>
<tr>
<td>0 Cu/Sn-Ni</td>
<td>4</td>
<td>4</td>
</tr>
</tbody>
</table>

A An undercoating of copper 4.0 µm thick shall be applied on copper-zinc alloys to serve as a zinc diffusion barrier.

B Thickness of Sn-Ni shall be stated in a Thickness Notation. A statement of Service Condition 5 is not sufficient.

## Table 3: Tin-Nickel Coatings on Zinc Alloys

<table>
<thead>
<tr>
<th>Service Condition Number</th>
<th>Thickness Notation</th>
<th>Minimum Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>4 Zn/Cu4/Sn-Ni</td>
<td>45</td>
<td>45</td>
</tr>
<tr>
<td>3 Zn/Cu4/Sn-Ni</td>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td>2 Zn/Cu4/Sn-Ni</td>
<td>15</td>
<td>15</td>
</tr>
<tr>
<td>1 Zn/Cu4/Sn-Ni</td>
<td>8</td>
<td>8</td>
</tr>
</tbody>
</table>

A An undercoating of copper 4.0 µm thick shall be applied to prevent zinc from contaminating the Sn-Ni plating bath and to serve as a diffusion barrier.

stresses in the coating on the inside of the bend usually cause some of the coating to flake off. To provide serviceability, the coating must be deposited in a stress-free condition. In addition, it is generally inadvisable to specify tin-nickel finish for parts subject to deformation in service.

6.2 **Basis Metal**—Tin-nickel can be deposited directly on steel, copper, and copper-base alloys. However, an undercoating of copper can improve performance in some systems and shall be used under the following conditions:

6.2.1 On steel, a copper undercoating with a minimum thickness of 4 µm, shall be used for Service Conditions 3, 4, and 5.

6.2.2 On copper-zinc alloys, a copper undercoating with a minimum thickness of 4 µm shall be used for all service conditions to prevent diffusion of the zinc.

6.2.3 Zinc-base alloys shall have an undercoating of a minimum of 4 µm of copper to prevent diffusion of the zinc into the deposit and to prevent contamination of the electrolyte with zinc.

**Note 3**—Tin-nickel-coated zinc-alloy diecastings shall never be returned for remelting to prevent contamination of the zinc alloy with tin.

7. **Coating Requirements**

7.1 **Composition of Coating**—The deposit shall contain 65 ± 5 % tin, the balance nickel.

7.2 **Appearance**:

7.2.1 The coating on all readily visible surfaces shall be smooth, fine grained, continuous, adherent, free of visible blisters, pits, nodules, indications of burning, excessive buildup, staining, and other defects. All tin-nickel coated articles shall be clean and undamaged. When necessary, preliminary samples showing the finish shall be supplied for approval. Where a rack contact mark is unavoidable, its location shall be indicated on the article or its drawing.

7.2.2 Defects and variations in appearance in the coating that arise from surface conditions of the substrate (scratches, pores, roll marks, inclusions, and the like) and that persist in the coating despite the observance of good metal finishing practices shall not be cause for rejection.

**Note 4**—Coatings generally perform better in service when the substrate over which they are applied is smooth and free of torn metal, inclusions, pores, and other defects. The specifications covering the unfinished product should provide limits for these defects. A metal finisher can often remove defects through special treatments, such as grinding, polishing, abrasive blasting, chemical etches, and electropolishing. However, these are not normal in the treatment steps preceding the application of the coating. When they are desired, they are the subject of special agreement between the purchaser and the seller.

**Note 5**—Proper preparatory procedures and thorough cleaning are essential to ensure satisfactory adhesion and corrosion resistance performance of the coating. Materials used for cleaning should not damage the basis metal, for example, by causing defects such as pits, intergranular attack, stress corrosion cracking, and unwanted hydrogen embrittlement. It is recommended that the following Practices, where appropriate for cleaning, be used: B 183, B 242, B 252, B 281, and B 322.

7.3 **Thickness**:

7.3.1 The thickness of the coating everywhere on the significant surfaces shall conform to the requirements in Tables 1-3 as to minimum thickness.

**Note 6**—The thickness of electrodeposited coatings varies from point to point on the surface of the product. (See Practice B 507.) The thickness is less in interior corners and holes. Such surfaces are often exempt from thickness requirements. If the full thickness is required in those locations, the electroplater will have to use special techniques that will probably raise the cost of the process.

**Note 7**—The coating thickness requirement of this specification is a minimum. Variation in the thickness from point to point on an article and from article to article in a production lot is inherent in electroplating. Therefore, if all of the articles in a production are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the specified minimum.

7.4 **Adhesion**—The coatings shall be adherent to the basis metal when subject to either test, in accordance with 8.5.2 and 8.5.3. There shall be no separation of the coating from the substrate.

7.5 **Integrity of the Coating**:

7.5.1 **Gross Defects/Mechanical Damage**—The coatings shall be free of mechanical damage, large pores, and similar gross defects. For some applications this requirement may be relaxed to allow for a small number of such defects (per unit area), especially if they are outside the significant surfaces.

7.5.2 **Porosity**—Almost all as-plated electrodeposits contain some porosity. The amount of porosity that may be tolerable depends on the severity of the environment that the article is likely to encounter during service or storage. If the pores are few in number or away from significant surfaces, their presence can often be tolerated. Such acceptance (or pass-fail) criteria shall be part of the product specification for the particular article or coating requiring the porosity test (see 8.6 for porosity test methods).

7.6 **Pre-Treatments of Iron and Steel for Reducing the Risk of Hydrogen Embrittlement**—Parts that are made of steels with ultimate tensile strengths of 1000 MPa (hardness of 31 HRC) or greater that have been machined, ground, cold formed, or cold straightened subsequent to heat treatment shall be heat treated prior to processing according to Specification B 849. The tensile strength shall be supplied by the purchaser.

7.7 **Post-Coating Treatments of Iron and Steel for Reducing the Risk of Hydrogen Embrittlement**—Parts that are made from steels with ultimate tensile strengths equal to or greater than 1000 MPa (hardness of 31 HRC) and surface hardened parts shall require heat treatment according to Specification B 850.

7.8 **Supplementary Requirements**—Packaging—If packaging requirements are to be met under this Specification, they shall be in accordance with Practice D 3951.

8. **Test Methods**

8.1 **Special Test Specimens**:

8.1.1 The permission or the requirement to use special test specimens, the number to be used, the material from which they are to be made, and their shape and size shall be stated by the purchaser.

**Note 8**—Test specimens often are used to represent the coated articles in a test if the articles are of a size, shape, or material that is not suitable for the test, or if it is preferred not to submit articles to a destructive test because, for example, the articles are expensive or few in number. The specimen should duplicate the characteristics of the article that influence the property being tested.
8.1.2 Special test specimens used to represent articles in an adhesion, solderability, porosity, corrosion resistance, or appearance test shall be made of the same material, shall be in the same metallurgical condition, and shall have the same surface condition as the articles they represent, and they shall be placed in the production lot of and be processed along with the articles they represent.

8.1.3 Special test specimens used to represent articles in a coating thickness test may be made of a material that is suitable for the test method even if the represented article is not of the same material. For example, a low-carbon steel specimen may represent a brass article when the magnetic thickness test is used (see Test Method B 499). The thickness specimen need not be carried through the complete process with the represented article. If not, it shall be introduced into the process at the point where the coating is applied and it shall be carried through all steps that have a bearing on the coating thickness. In rack plating, the specimen shall be racked in the same way with the same distance from and orientation with the anodes and other items in the process as the article it represents.

Note 9—When special test specimens are used to represent coated articles in a thickness test, the specimens will not necessarily have the same thickness and thickness distribution as the articles unless the specimens and the articles are of the same general size and shape. Therefore, before finished articles can be accepted on the basis of a thickness test performed on special test specimens, the relationship between the thickness on the specimen and the thickness on the part needs to be established. The criterion of acceptance is that thickness on the specimen that corresponds to the required thickness on the article.

8.2 Composition of the Coating—The deposit continues to have a content of 35 ± 5 % nickel (with the balance tin) over a wide range of solution compositions and operating conditions (see 1.1). For this reason an analysis of the deposit is required infrequently, if at all. A sample of the deposit can be obtained by plating on a passivated stainless steel panel from which the deposit can be peeled. The composition of the deposit can be determined by such methods as volumetric or gravimetric analysis, density measurements, atomic adsorption, X-ray and spectrometry.

8.3 Appearance—The coating shall be examined at up to 4X magnification for conformance to the requirements for appearance.

8.4 Thickness—The coating thickness shall be measured at locations on the significant surface(s) where the thickness would appear to be a minimum. Several methods of determining the thickness are available, depending upon the thickness of coating, the shape of the article, and the basis metal. They are known as microscopical, magnetic, coulometric, and beta backscatter. X-ray spectrometry may be used, but if the basis metal is a tin-containing alloy, such as bronze, or if a nickel undercoating is present, the measurement instruments must be calibrated on the same substrate material. The following methods are acceptable for measuring local thickness of the coating: Test Methods B 487, B 499, B 504, B 567, and B 568.

8.5 Adhesion:

8.5.1 Adhesion shall be determined by either the burnishing test or the heat-quench test.

8.5.2 Burnishing Test—The adhesion of thinner deposits can be determined by the burnishing test described in Section 4 of Practice B 571.

8.5.3 Heat-Quench Test—The heat-quench test is described in Section 9 of Practice B 571. For tin-nickel alloy coatings the temperatures of test for various substrates shall be the same as those shown in Table I of the test method for chromium, nickel plus chromium, and copper coatings (see Note 11).

Note 10—This test may have an adverse effect on the mechanical properties of the article tested.

8.6 Porosity and Gross Defects Testing:

8.6.1 Coatings on articles of steel (or iron) having a local thickness of 10 µm or greater should be subjected to the test given in Appendix X2, and the results evaluated according to the procedure described.

8.6.2 For coatings on articles made from copper or copper alloy as the substrate metal, the following tests can be used.

8.6.2.1 To determine mechanical damage or gross defects as defined in Guide B 765 only, subject samples to the sodium polysulfide immersion test outlined in Specification B 246. Black spots or lines are evidence of mechanical damage or gross defects.

8.6.2.2 To determine all porosity to the copper or copper alloy substrate, the humid sulfur vapor test (see Test Method B 809) shall be used.

8.7 Post-Coating Treatment for Reducing the Risk of Hydrogen Embrittlement—If required by the purchaser, the effectiveness of the post-coating heat treatment performed under 7.7 shall be determined by the method described in 8.4 of Specification B 634 entitled “Hydrogen Embrittlement Relief.”

9. Sampling

9.1 The sampling plan used for the inspection of a quantity of the coated articles shall be as agreed upon between the purchaser and the seller.

Note 11—Usually, when a collection of coated articles (the inspection lot, see 9.2) is examined for compliance with the requirements placed on the articles, a relatively small number of the articles, the sample, is selected at random and is inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Three standards, Test Method B 602, Guide B 697, and Test Method B 762, contain sampling plans that are designed for the sampling inspection of coatings.

Test Method B 602 contains four sampling plans, three for use with tests that are non-destructive and one when they are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 identifies the plan to be used.

Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and seller need to agree on the plan to be used.

Test Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numerical value and certain statistical requirements must be met. Test Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Test Method B 762 identifies the plan to be used.

Note 12—When both destructive and non-destructive tests exist for the measurement of a characteristic, the purchaser needs to state which is to
be used so that the proper sampling plan is selected. A test may destroy the coating but in a non-critical area; or, although it may destroy the coating, a tested part can be reclaimed by stripping and recoating. The purchaser needs to state whether the test is to be considered destructive or nondestructive.

9.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specifications, that have been coated by a single seller at one time or at approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

9.3 If special test specimens are used to represent the coated articles in a test, the number used shall be that required in 8.1.1.

10. Rejection and Rehearing

10.1 Articles that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer or seller promptly and in writing. In case of dissatisfaction with the results of a test, the producer or seller may make a claim for a rehearing. Coatings that show imperfections during subsequent manufacturing operations may be rejected.

11. Certification

11.1 When specified in the purchase order or contract, the purchaser shall be furnished certification that samples representing each lot have been either tested or inspected as directed in this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

12. Keywords

12.1 alloys; electrodeposited; tin-nickel; coatings; electrodeposited; nickel alloy; coatings; electrodeposited; tin-nickel alloy

APPENDIXES

(X1. DESCRIPTION OF SERVICE CONDITIONS AND EXAMPLES OF END USES)

- SC5—Extended severe service conditions. The coating is subjected to continuous abrasion or exposure to corrosive liquids or gases. Complete coverage of tin-nickel is required. Typical applications are chemical pumps, valves, and flow control devices.

- SC4—Very severe service conditions. The coating is subjected to abrasion or exposure conditions, or both, that are less severe than those of SC5, or is exposed for intermittent periods. The applications are similar to those of SC5.

- SC3—Severe service conditions. Includes exposure to dampness and to industrial atmospheres. Typical applications are cooking utensils, analytical weights, and surgical instruments.

- SC2—Moderate service conditions. Includes dry or interior atmosphere. Typical applications are electronic parts, watch parts, and printed circuit board etch resist coating.

- SC1 and SC0—Mild service conditions. Less severe conditions than SC2. Typical applications are electronic parts, printed circuit board etch resist coating, and final coating when given a gold flash to assure solderability.

(X2. SULFUR DIOXIDE POROSITY TEST)

- Principle—Exposure to a moist atmosphere containing a low concentration of sulfur dioxide causes no corrosion of tin-nickel alloy of the correct composition, but causes spots of corrosion product to appear at discontinuities in the coating. If the sulfur dioxide concentration is too high, the corrosion product is too fluid to permit easy identification of pore sites. The method, which depends on the production of sulfur dioxide from the reaction between sodium thiosulfate and sulfuric acid within the test chamber, ensures suitable conditions for the development of immobile corrosion products at discontinuities.

- Apparatus:

  - Chamber—The test shall be performed in a chamber fitted with a lid or door and preferably made of glass or of a transparent plastic. The chamber shall be large enough to hold the test specimens with their lowest parts at least 75 mm above the surface of a solution occupying at least 2% of the chamber capacity. The chamber shall be gas-tight but need not be capable of resisting pressure. A glass plate makes an adequate joint with a lubricated ground edge of a glass tank. The chamber shall have a uniform cross section and the solution placed in it shall cover the base completely.

- Glass or Plastic Stand—to support the specimens under test inside the cabinet. The significant surfaces may be inclined at any angle, but the same inclination should be used for similar articles.

- Corrosive Medium—The corrosive medium shall be moist air containing sulfur dioxide produced by a solution occupying 2% of the capacity of the chamber and prepared by adding 1 part by volume of 0.1 N sulfuric acid to 4 parts of a solution containing 10 g of sodium thiosulfate crystals in 1 litre of water. The chemicals shall be analytical reagent grade.

- Temperature of Test—Conduct the test at 20 ± 2°C.
taking precautions against rapid temperature fluctuation in the course of the test.

X2.5 Procedure:

X2.5.1 Before testing, clean the specimens with an organic solvent (for example, 1,1,1-trichloroethane), wipe them with a lint-free cloth, and allow them to attain room temperature.

X2.5.2 Introduce into the test chamber a volume of the sodium thiosulfate solution equal to 2% of the volume of the chamber. Suspend the test specimens above this solution with the surface of the specimens at least 25 mm apart, at least 25 mm from any wall of the chamber, and at least 75 mm from the surface of the solution. Add to the solution a volume of 0.1 N sulfuric acid equal to a quarter of the volume of the thiosulfate solution and close the chamber, keeping it shielded from drafts and other causes of rapid temperature change. The addition of the sulfuric acid may be made before the test specimens are placed in position, provided that the chamber is closed within five minutes of the addition of the acid.

X2.5.3 Leave the specimen in the chamber for 24 h. After removing the specimens from the corrosive atmosphere, allow them to dry without wiping or cleaning in any way. They should then be examined and evaluated, using the options outlined in Guide B 765, Sections 6 and 7.
Standard Specification for
Autocatalytic Nickel Boron Coatings for Engineering Use

This standard is issued under the fixed designation B 607; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 Nickel boron coatings are produced by autocatalytic (electroless) deposition from aqueous solutions. These solutions contain either an alkylamineborane or sodium borohydride as a reducing agent, a source of nickel ions, a buffer, complexant, and control chemicals.

1.2 This standard describes the requirements for coatings of autocatalytic nickel boron deposited from aqueous solutions onto substrates for engineering use. The specification classifies these coatings into two types:

1.2.1 Type 1 coatings have a boron content of 0.1 to less than 3.5 mass percent with the balance nickel.

1.2.2 Type 2 coatings have a boron content of 3.5 to 6 mass percent and a minimum of 90 mass percent nickel.

1.3 The coatings are hard and uniform in thickness, even on irregular shaped parts, and used in a wide range of applications.

1.4 Process solutions formulated with an alkylamineborane usually produce coatings that contain 0.1 to 3.5 % boron. Thin coatings of this type provide bondability and solderability on electronic components such as lead frames, electrical contacts, and headers. To maintain solderability, these coatings are generally not heat treated.

1.5 Process solutions formulated with sodium borohydride are strongly alkaline and are frequently used to plate steel and titanium parts to impart surface hardness and wear resistance properties. Deposits produced from these processes can contain 3 to 5 % boron and thallium or other metals which are used to stabilize the plating solution and modify the coating properties.

1.6 The physical and mechanical properties of these deposits such as density, hardness, stress, and melting point will vary with the boron content. The variation of boron content also affects the quantity and structure of nickel boride precipitated during heat treatment. In the as-plated condition the deposit consists of a predominantly amorphous mixture of nickel and boron with a hardness of about 700 HKN. When the deposit is heated above 300°C the nickel crystallizes, forming nickel clusters of Ni (111) and boron precipitates as nickel boride, Ni2B (211) and (311), increasing the hardness to greater than 1000 HK100 for Type 2 coatings.

1.7 The nickel boron coatings are microporous and offer limited corrosion protection. Their columnar structure, however, is beneficial in reducing wear because it provides a means of trapping lubricants within the surface of the coated part.

1.8 This document describes only autocatalytic nickel boron coatings that have been produced without use of external electric sources.

1.9 The following hazards caveat pertains only to the Test Methods section of this specification: This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

Note 1—The following AMS standards are not requirements. They are referenced for information only: AMS 2399 and AMS 2433.

2. Referenced Documents

2.1 ASTM Standards:
B 374 Terminology Relating to Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section
B 567 Test Method for Measurement of Coating Thickness by Beta Backscatter Method
B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings
B 578 Test Method for Microhardness of Electroplated Coatings
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 656 Guide for Autocatalytic (Electroless) Nickel-Phosphorus Deposition on Metals for Engineering Use
B 667 Practice for Construction and Use of a Probe for Measuring Electrical Contact Resistance
B 678 Test Method for Solderability of Metallic-Coated Products


2 Annual Book of ASTM Standards, Vol 02.05.
3 Annual Book of ASTM Standards, Vol 03.04.
3. Terminology

3.1 Definitions—Many terms used in this standard are defined in Terminology B 374.

3.2 Definitions of Terms Specific to This Standard:
3.2.1 cold shut—a void on the surface which has been closed by machining and then partially opened through cleaning.

3.2.2 hot halide stress-corrosion cracking—a type of mechanical failure produced by halogenated solvents that have been absorbed onto titanium and then in the presence of heat cause microcracking, and the loss of mechanical strength.

3.2.3 lap cracks—a surface imperfection caused by cold working of steels producing a void which can be duplicated in the deposit.

3.2.4 significant surface—those substrate surfaces which the coating must protect and that are essential to the appearance.

4. Classification

4.1 The classification by type of these coatings establishes the amount of boron in the alloy.

4.1.1 Type 1—Coatings shall contain 0.1 to less than 3.5 mass percent boron with the balance nickel.

4.1.2 Type 2—Coatings shall contain 3.5 to 6 mass percent boron and a minimum of 90 mass percent nickel.

4.2 The classification by class of these coatings establishes the post treatment to be performed on the part(s). The post treatment steps are designed to reduce the potential for hydrogen embrittlement, increase the adhesion of the coating to the substrate, improve the fatigue properties of the part(s), and increase the wear resistance and hardness of the coating:

4.2.1 Class 1—Parts are supplied as plated with no post heat treatment.

4.2.2 Class 2—Parts are heat treated after plating to increase hardness. The coating is heat treated at 365 to 385°C for 90 min (see 7.2.4).

4.2.3 Class 3—Parts are heat treated after plating at 180 to 200°C for 2 to 23 h to improve coating adhesion on steel and for hydrogen embrittlement relief of steels (see 7.2.4).

4.2.4 Class 4—Parts are heat treated after plating at 120 to 130°C for a minimum of 1 h to improve adhesion on heat-treatable (age-hardened) aluminum alloys and carburized steels (see 7.2.4).

4.2.5 Class 5—Parts are heat treated after plating at 365 to 375°C for a minimum of 4 h to improve adhesion on titanium and titanium alloys (see 7.2.4).

4.3 The classification by grade establishes the minimum thickness of the coating:

4.3.1 Grade A—Parts are plated to a minimum coating thickness of 0.5 µm.

4.3.2 Grade B—Parts are plated to a minimum coating thickness of 12 µm.

4.3.3 Grade C—Parts are plated to a minimum coating thickness of 25 µm.

4.3.4 Grade D—Parts are plated to a minimum coating thickness of 75 µm.

5. Ordering Information

5.1 The purchaser should be aware of several processing considerations or options available to the processor and when ordering should supply the information described in 5.1.1 through 5.1.15 in the purchase order and drawings.

5.1.1 Title, ASTM designation, and year of issue of this specification.

5.1.2 Composition and metallurgical condition of the basis metal, assemblies of dissimilar materials must be identified.

5.1.3 Classification of the coating: type, class, and grade for this specification (see Section 4).

5.1.4 Minimum thickness required on the significant surface, and any maximum dimensions or tolerance requirements, if any (see 7.2.2).

5.1.5 Method of adhesion testing from Test Method B 571 to be used in acceptance requirements (see 8.3).

5.1.6 Requirements for certification and test reports (see Section 11).

5.1.7 Requirements for heat treatment of the part(s) for stress relief prior to plating (see 7.2.4).

5.1.8 Optional sampling plan for lot inspection of the part(s) (see 9.1 and 13.1).

5.1.9 Increased sampling frequency, if any, for qualification tests (see 7.3).

5.1.10 Supplemental requirements for shot peening of the part(s) (see 12.1).

5.1.11 Supplemental requirements for wear testing (see 12.2 and 12.3).

5.1.12 Supplemental requirements for heat treatment in vacuum or inert or reducing atmosphere (see 7.2.1 & 12.4).

5.1.13 Supplemental contact resistance requirements (see 12.5).

5.1.14 Supplemental solderability requirements (see 12.6).
6. Materials and Manufacture

6.1 Pretreatment—Parts can be processed in accordance with Practice B 656.

6.1.1 A suitable method should be used to remove surface oxides and foreign materials which can cause poor adhesion and increased porosity.

6.1.2 A suitable method should be used to condition and activate the surface so that an adherent coating will be produced.

6.2 Basis Material and Workmanship—Nickel boron coatings will replicate the surface finish of the basis material. Imperfections in the surface of the basis material including scratches, porosity, pits, inclusions, roll and die marks, lap cracks, burrs, cold shuts, and surface roughness that could adversely affect the coating should be brought to the attention of the purchaser prior to processing (see 7.2.1).

6.3 Stress Relief—Surface-hardened parts can require stress relief before plating. The stress relief heat treatment can reduce the hardness of some alloys and should therefore be reviewed by all parties before processing (see 5.1.7 and 7.2.4). Shorter times and higher temperature can be used if the resulting loss of surface hardness is acceptable to the purchaser.

6.4 Hydrogen Embrittlement Relief—Hydrogen embrittlement of high strength steels can be initiated by several different processing operations. Exposure of the parts to hydrogen sources will generally induce the condition. Care must be exercised whenever high strength steel is processed to ensure minimal exposure and timely relief treatment.

6.5 Stress-Corrosion Cracking—Titanium and titanium alloys are subject to stress-corrosion cracking after processing. Pretreatment solutions including rinses should not contain methanol, halogenated hydrocarbon, or more than 50 ppm chlorides, all of which can cause subsequent stress-corrosion cracking when the parts are heated to 260°C or higher.

7. Requirements

7.1 Process—The nickel boron coatings shall be produced by autocatalytic nickel deposition from aqueous solutions.

7.2 Acceptance Requirements—The acceptance requirements in 7.2.1 through 7.2.4 are required for all lots of part(s). Each lot of part(s) shall be sampled with the recommended procedure described in Section 9 of this specification.

7.2.1 Appearance—The coating shall have a uniform appearance without visible imperfections such as blisters, pits, pimples, and cracks.

7.2.1.1 Imperfections that arise from the surface condition of the basis metal and that cannot be removed using conventional metal finishing techniques and that persist in the coating shall not be cause for rejection.

7.2.1.2 Discoloration caused by heat treatment shall not be cause for rejection unless specified in the ordering information (see 5.1.12 and 12.4).

7.2.2 Thickness—The coating thickness shall be measured and conform to the specified grade.

7.2.3 Adhesion—The coating shall pass the adhesion test of Test Method B 571 as specified in the ordering information (see 5.1.15).

7.2.4 Heat Treatment:

7.2.4.1 All steel part(s) with a tensile strength of 1000 MPa or greater shall be heat treated at 190 ± 15°C for stress relief in accordance with Table 1 before plating and baked within 3 h after plating for hydrogen embrittlement relief.

7.2.4.2 Class 2 coated part(s) shall be heat treated after plating in accordance with Table 2 for precipitation hardening of the deposit.

7.2.4.3 Heat treatment for Class 3 coated steel part(s) shall be in accordance with Table 1.

7.2.4.4 Heat treatment for Class 4 and 5 coated part(s) other than steel basis material shall be in accordance with Table 3.

7.3 Qualification Requirements—Coating and process attributes that require testing on a monthly basis, or more frequently when specified in the ordering information by the purchaser. A test specimen or part, processed in a manner that duplicates the characteristics of production parts, shall be produced and used in these tests.

7.3.1 Hardness—The hardness of the Type 2, Class 2, Grade C and D coating shall be not less than 1000 HK100 as measured by Test Method B 578.

7.3.2 Composition—The coating composition produced from the process shall be analyzed for nickel and boron. The alloy produced shall be within the range specified for the coating type.

7.3.3 Hydrogen Embrittlement—The process and coating shall be evaluated for freedom from hydrogen embrittlement and pass requirements of Test Method F 519.

8. Test Methods

8.1 Test Specimens:

8.1.1 When separate test specimens are required, the number to be used, the material from which they are to be made, and their shape and size shall be specified by the purchaser.

8.1.2 When separate test specimens are used for acceptance or qualification testing of the coating, the specimens shall be made of the same material as the part(s), have the same metallurgical condition as the part(s), and be processed with the part(s).

8.2 Thickness—The thickness shall be measured at any place on the significant surface designated by the purchaser, and the measurement shall be made with an accuracy of better than 10 % by a method selected by the purchaser. Fig. X1.1 describes the density of these coatings in relationship to their boron content.

**TABLE 1 Heat Treatment for Stress Relief Before Plating and for Hydrogen Embrittlement Relief After Plating**

<table>
<thead>
<tr>
<th>Tensile strength steel, MPa</th>
<th>Heat treatment, minimum, at 190 ± 15°C, h</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000 to 1450</td>
<td>2</td>
</tr>
<tr>
<td>1450 to 1800</td>
<td>18</td>
</tr>
<tr>
<td>over 1800</td>
<td>23</td>
</tr>
</tbody>
</table>

Note: 1—Heat treatment for stress relief of surface hardened steels prior to plating shall be 140 ± 10°C for 5 h.
8.2.1 Weigh, Plate, Weigh Method—Using a similar substrate material, weigh to the nearest milligram before and after plating. Calculate the thickness from the increase in mass, surface area, and density of the coating.

NOTE 2—The density of the coating will decrease as the mass percent boron in the coating increases. For Type 1 coatings the density is approximately 8.7 g/cm³ and for Type 2 coatings it is approximately 8.2 g/cm³ (see Appendix X1).

8.2.1.1 Example:

\[
\text{Thickness, } \mu\text{m} = \frac{10 \times W}{A \times D}
\]  

where:

- \(W\) = mass gain, mg,
- \(A\) = area of plating, cm², and
- \(D\) = density of deposit, g/cm³.

8.2.2 Metallographic Sectioning—Plate a specimen of similar composition and metallurgical condition to the part(s) being plated, or use a sample from the lot, cross-section, mount, and polish. Using a calibrated Vernier microscope, examine the thickness of the deposit and average over 10 readings using Test Method B 487.

NOTE 3—Microscopic metallographic sectioning is dependent on the sample preparation.

8.2.3 Micrometer Method—Measure the part(s) or test a coupon in a specific area before and after plating using a suitable micrometer. Ensure that the specimen is at the same temperature for each measurement and that the surface measured is smooth.

8.2.4 Beta Backscatter Method—The coating thickness can be measured by the use of a beta backscatter device using Test Method B 567. This technique measures the mass per unit area of the coating applied over the substrate and is displayed as thickness. The use of the beta backscatter method is restricted to basis metals that have an atomic number less than 18 or greater than 40. The instrument shall be calibrated with specimens having the same basis material and coating composition as the part(s).

8.2.5 X-Ray Spectrometry—The coating thickness can be measured by X-ray spectrometry using Test Method B 568. This technique measures the mass per unit area of the coating applied over the substrate and is displayed as thickness. It can only be used for coatings of known boron content. The instrument shall be calibrated with specimens having the same basis material and coating composition as the part(s).

8.3 Adhesion—Adhesion of the coating to the basis material shall be evaluated using one of the procedures described in Test Method B 571 (see 5.1.5).

8.4 Composition:

8.4.1 Inductively Coupled Plasma—Chemical analysis by inductively coupled plasma—Dissolve approximately 50 mg of a foil sample in 50 mL of 100 % warm reagent grade nitric acid. Analyze the solution for nickel at 232.00 nm, boron at 241.77 nm, and any other alloying element at an appropriate wavelength. From the results of the analysis calculate a fraction of the boron or alloying element divided by the total of boron, nickel, and alloying element. Report the percent boron and alloying element if present in the coating.

8.4.2 Other Chemical Methods—Determine mass percent nickel content according to Methods E 39 on known weight of deposit dissolved in nitric acid. The determination of mass percent boron content may be accomplished by an appropriate analytical method (such as atomic absorption spectroscopy). Report the percent boron and alloying elements if present in the coating.

9. Sampling

9.1 The sampling plan used for the inspection of a quantity of coated parts (lot) shall be as described in Test Method B 602, unless otherwise specified by purchaser in the purchase order or contract (see 5.1.8 and 13.1).

NOTE 4—Usually, when a collection of coated parts (the inspection lot; see 9.2) is examined for compliance with the requirements placed on the parts, a relatively small number of parts—the sample—is selected at random and inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection sample. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for the sampling inspection of coatings.

Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one for use with tests that are destructive. The purchaser and producer may agree on the plan(s) to be used. If they do not, Test Method B 602 identifies the plan to be used.

Guide B 697 provides a large number of plans and also gives guidance on the selection of a plan. When Guide B 697 is specified, the purchaser and producer need to agree on the plan to be used.

Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numerical value and certain statistical requirements must be met. Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The purchaser and producer may agree on the plan(s) to be used. If they do not, Method B 762 identifies the plan to be used.

9.2 An inspection lot shall be defined as a collection of coated parts that are of the same kind, that have been produced to the same specification, that have been coated by a single producer at one time or approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

9.3 If separate test specimens are used to represent the part(s) in the test, the number shall be that required in 8.1.

10. Rejection and Rehearing

10.1 Part(s) that fail to conform to the requirements of this standard may be rejected. Rejection shall be reported to the producer promptly in writing. In the case of dissatisfaction with the results of a test, the producer may make a claim for a rehearing. Coatings that show imperfections may be rejected.
11. Certification

11.1 When specified in the purchase order or contract, the purchaser shall be furnished certification that the samples representing each lot have been processed, tested, and inspected as directed in this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

12. Supplementary Requirements

12.1 Shot Peening—When specified by the purchaser in the ordering information, the part(s) shall be shot peened prior to plating in accordance with MIL-STD-13165.

12.2 Abrasive Wear—When specified by the purchaser in the ordering information, the coating shall be tested for abrasion wear resistance using the procedure described in Appendix X2 of this specification. The coating shall meet a maximum wear rate which is specified by the purchaser and agreed to by the producer.

12.3 Adhesive Wear—When specified by the purchaser in the ordering information, the coating shall be tested for adhesive wear resistance using Test Method D 2714 or D 2670. The wear rate shall be specified by the purchaser and agreed to by the producer.

12.4 Inert Atmosphere—When specified by the purchaser in the ordering information, the coating shall be heat treated in a vacuum or an inert or reducing atmosphere to prevent surface oxidation of the coating.

12.5 Contact Resistance—When specified by the purchaser in the ordering information, the coating shall be tested for contact resistance using Practice B 667.

12.6 Solderability—When specified by the purchaser in the ordering information, the unaged coating shall pass Test Method B 678.

13. Special Government Regulations

13.1 Sampling—The producer when plating part(s) for the U.S. government and military use shall use a sampling plan from MIL-STD-105.

13.2 Shot Peening—High strength steel part(s) processed for U.S. government and military use shall be shot peened in accordance with MIL-STD-13165.

APPENDIXES

(Nonmandatory Information)

X1. DENSITY OF NICKEL BORON COATINGS

X1.1 Many thickness test methods measure the mass per unit area and display the thickness in linear units. These test methods require a density factor to calculate the thickness. Fig. X1.1 can be used to determine the density of nickel coatings with varying boron contents.

FIG. X1.1 Density of Nickel Coatings with Varying Boron Content
X2. TABER ABRASER WEAR TEST METHOD

X2.1 Scope

X2.1.1 This test method will evaluate the resistance of the coating to abrasive wear. The test is performed on a rotating plated panel that is abraded by two rotating rubber wheels. The panel is weighed before and after each 1000 cycles for weight loss determination. Duration of the test is 5000 cycles and it can be extended to 25 000 cycles.

N O T E X2.1—Variations in results have been attributed to the humidity in the laboratory and the storage conditions of the CS-10 wheels. Care should be taken to control the humidity between tests as recommended by the equipment manufacturer.

X2.1.2 The results are variable between tests and therefore three plated test specimens should be tested to 6000 cycles each. The results should be averaged without the first 1000 cycles and the abrasion wear resistance reported.

X2.2 Apparatus 9

X2.2.1 Taber Abraser Wear Testing Unit—The unit must be capable of loading with a 1000 g load and operating with a vacuum.

X2.2.2 Abrasive Wheels—Use CS-10 (resilient rubber) Taber wheels. To reface the wheels use CS-11 discs.

X2.2.3 Test Specimens—Test specimens shall be made from 20 gage CR steel 4 by 4 in. (100 mm by 100 mm by 1.3 mm) with a 0.250-in. (6.35-mm) hole in the center.

X2.2.4 Analytical Balance, Capable of weighing 150 g ± 0.1 mg.

X2.3 Procedure

X2.3.1 Plate three specimens with 0.001 in. (25 µm) of nickel boron coating.

X2.3.2 Heat treat the specimens for 375 ± 10°C for 90 min.

X2.3.3 Abrasion test the specimens. For each of the three specimens follow X2.3.3.1 through X2.3.3.6:

X2.3.3.1 Smooth the coating for 1000 cycles to remove any surface roughness. The wheels shall be loaded with 1000 g with the vacuum on high for the entire test.

X2.3.3.2 Cool and weigh the specimen to the nearest 0.1 mg.

X2.3.3.3 Dress the CS-10 wheels with a CS-11 disc for 50 cycles.

X2.3.3.4 Abrasion test the coating with a 1000 g load for 1000 cycles.

X2.3.3.5 Repeat X2.3.3.2, X2.3.3.3, and X2.3.3.4 until a total of 6000 cycles have been accomplished for each specimen.

X2.3.3.6 Determine the relative humidity in the test area and report it with the wear results.

N O T E X2.2—This equipment should be operated using manufacturer’s recommended procedures.

X2.4 Reporting

X2.4.1 Determine the average weight loss in milligrams for each specimen per 1000 cycles (Taber Wear Index) and the mean weight loss per 1000 cycles for all specimens. Report the mean and standard deviation for the coating and the humidity during testing.

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9 The Taber Abraser Wear Testing Unit, abrasive wheels and discs, and test specimens are available from Teledyne Taber, North Tonawanda, NY 14120.
Standard Test Method for Measuring Dimensional Changes of Metal Powder Specimens

This standard is issued under the fixed designation B 610; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method primarily covers a procedure for determining the change in dimensions that occurs as a result of pressing and sintering a metal powder specimen. The size change referred to is the difference between the die size and the size of the sintered specimen after it has cooled to room temperature, expressed as a percentage. This size change is a basic characteristic of the metal powder.

1.2 The general procedure of measuring a test specimen and die, then calculating the dimensional change, as described in this test method, also may be used to determine green expansion, heat treat changes, or other dimensional changes of test specimens that result from processing steps.

1.3 The values stated in inch-pound units are to be regarded as the standard. The SI equivalents are in parentheses and may be approximate.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   B 215 Practices for Sampling Finished Lots of Metal Powders
   B 243 Terminology of Powder Metallurgy
   B 312 Test Method for Green Strength for Compacted Metal Powder Specimens

3. Terminology

3.1 Definitions—For definitions of terms used in this test method, see Terminology B 243.

4. Summary of Test Method

4.1 Test specimens are made from the metal powder lot under consideration by pressing and sintering or other processing (see 1.2). The absolute dimensional change as a result of pressing and sintering is calculated from measurements taken on the sintered test specimens and from the die cavity.

4.2 When this method is used to compare metal powder lots, specimens from a reference metal powder lot agreed upon by the parties concerned are made and processed at the same time. The dimensional change of the test specimen is calculated and mathematically compared with the dimensional change of the reference specimen.

5. Significance and Use

5.1 The dimensional change obtained under specified conditions of pressing and sintering is a material characteristic inherent in the powder. It is useful to the production, testing, and use of metal powders.

5.2 The absolute dimensional change may be used to classify powders or differentiate one type or grade from another, to evaluate additions to the powder mix or measure process changes, and to guide in the design of tooling.

5.3 The comparative dimensional change is mainly used as a quality control test to measure variations between lots of metal powders of the same material composition.

5.4 Factors known to affect size change are the test specimen process conditions of green density, sinter time, sinter temperature, sinter atmosphere, level of additions to the base metal powder, lubricant type, powder grade, powder type, and powder lot.

6. Apparatus

6.1 Balance, suitable for weighing accurately to 0.01 g.

6.2 Mixer, capable of thorough and controlled mixing of metal powders and lubricants.

6.3 Compression-Testing Machine or Powder Press, capable of applying the required pressure to produce the test specimen.

6.4 Punches and Die, for producing a test specimen, are shown in Fig. 1.

6.5 Micrometers, capable of measuring the necessary dimensions to 0.0001 in. (0.0025 mm).

6.6 Sintering Furnace, capable of maintaining a specified work temperature and having controlled atmosphere and a
water-jacketed cooling zone. A vacuum furnace also is permitted.

6.7 When the dimensional change test is run to approve metal powders for production, it is preferable to use sintering conditions that approximate or simulate production conditions.

7. Test Specimen

7.1 The green test specimen, having nominal dimensions of 0.500 in. (12.70 mm) wide by 1.250 in. (31.75 mm) long by 0.250 in. (6.35 mm) thick, as shown in Fig. 2, is the recommended test specimen.

8. Procedure

8.1 Measure the long dimension of the die cavity to 0.0001 in. (0.0025 mm). Note this dimension, \( L_D \), for future reference.

8.2 Obtain a test sample of the test metal powder (and reference metal powder, if used) in accordance with Practice B 215.

8.3 Compact three test specimens (and three reference specimens, if used) in accordance with Test Method B 312.

8.4 Sinter the test specimens (with three reference specimens, if used) using a heating rate, sintering time and temperature, and cooling rate suitable for the material being tested.

8.5 When the dimensional change method is used to approve a metal powder lot, all powder and processing conditions shall be agreed upon by the parties concerned.

8.6 Measure the long dimension of the sintered specimens to 0.0001 in. (0.0025 mm). Note this dimension, \( L_T \) and \( L_R \) for reference specimens, if used, for later calculation use.

9. Calculation

9.1 Absolute Dimensional Change:

9.1.1 Calculate the percent dimensional change (\( D_T \)) for the specimens from the test powder as follows:

\[
D_T, \% = \frac{L_T - L_D}{L_D} \times 100
\]  

where:

- \( L_T \) = long dimension of sintered test specimen (see 8.6)
- \( L_D \) = long dimension of the die cavity (see 8.1)

9.1.2 Calculate the percent dimensional change (\( D_R \)) for the specimens from the reference powder as follows:

\[
D_R, \% = \frac{L_R - L_D}{L_D} \times 100
\]  

where:

- \( L_R \) = long dimension of sintered reference specimen (see 8.6).

9.2 Comparative Dimensional Change—Calculate the comparative dimensional change (\( D \)) as follows:

\[
D, \% = D_T - D_R
\]  

10. Report

10.1 Report the absolute dimensional change from die size for the specimens made from the test powder (\( D_T \)), and those from the reference powder (\( D_R \)), as the average of the three specimens, calculated to the nearest 0.01 %, including the sign of + for growth and – for shrinkage.

10.2 Report the comparative dimensional change (\( D \)), as the mathematical difference between the average absolute dimensional change from die size for the specimens made from the test powder and those from the reference powder as a percent to the nearest 0.01 %, including the sign of + or – to indicate direction of the difference.

Note 1—If the % dimensional change is divided by 100, the results then will be expressed in in./in. (mm/mm).

10.3 The following supplementary information should be reported for clarification:

10.3.1 Identification of powder, brand, grade, and lot number.

10.3.2 Chemistry of powder mix used if other than elemental powders are being tested.

10.3.3 Type, brand and percent of lubricant or the additives.

10.3.4 Compacting pressure.

10.3.5 Green density.

10.3.6 Sintering temperature.

10.3.7 Sintering time, as defined as the elapsed time for the heated specimen to be within ±15°F (±8°C) of the stated sintering temperature for iron and ±10°F (±5°C) for copper base, and
10.3.8 Furnace atmosphere.

10.4 The absolute dimensional change ($D_R$) of the specimens made from the reference metal powder shall be within the limits agreed upon between the parties concerned in order to have a valid test.

11. Precision and Bias

11.1 The precision and bias of this test method is currently under development in Subcommittee B09.02 on Base Metal Powders.

12. Keywords

12.1 die size; dimensional change; metal powders; sintering
1. Scope

1.1 This test method covers the determination of abrasive wear resistance of cemented carbides.

1.2 The values stated in inch-pound units are to be regarded as the standard. The SI equivalents of inch-pound units are in parentheses and may be approximate.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 311 Test Method for Density Determination for Powder Metallurgy (P/M) Materials Containing Less Than Two Percent Porosity

3. Terminology

3.1 Definitions:

3.1.1 abrasion resistance, \(n\)—the specimen’s unit loss in volume per revolution of the steel wheel carrying the abrasive.

3.1.2 abrasive wear, \(n\)—that wear caused by a slurred abrasive on a rotating surface, contacting the specimen surface under pressure.

3.1.3 wear number, \(n\)—the reciprocal of the specimen’s total volume loss in units of \(\text{cm}^{-3}\).

4. Significance and Use

4.1 This test method provides a guide to determine the relative abrasive wear resistance of cemented carbides under high-stress abrasion conditions. The abrasive wear is not a measure of wear characteristics of carbides under all conditions and is not to be misconstrued as indicative of wear when carbides are used for machinery. It does show the difference in resistance to abrasive use for different carbide compositions and can be used as a practical test or as a research method for sorting out these differences.

5. Apparatus

5.1 Vessel, suitable for holding the abrasive slurry and the wheel (see Fig. 1). One suitable vessel is made of \(\frac{3}{16}\)-in. (5-mm) steel. The internal dimensions are \(8\frac{3}{4}\) in. (220 mm) high, \(9\frac{3}{4}\) in. (245 mm) long, and \(2\frac{1}{4}\) in. (60 mm) wide, with a \(5\frac{1}{2}\)-in. (140-mm) radius at the bottom.

5.2 Wheel, made of annealed AISI 1020 steel, that rotates in the center of the vessel at \(100 \pm 5\) rpm. The direction of rotation is from the slurry to the specimen. Four curved vanes are affixed to either side of the wheel to agitate and mix the slurry and to propel it toward the specimen. The maximum wheel diameter shall be \(6.65\) in. (169 mm) and the width shall be \(0.500 \pm 0.005\) in. (12.7 \pm 0.1 mm). The wheel shall be discarded when its diameter has decreased to \(6.50\) in. (165 mm) minimum, after repetitive use. In use, a slight burr will form at the periphery. This burr will compensate for wheel wear by widening the wear path in the specimen; it shall not be removed.

5.3 Specimen Holder, to be pressed against the periphery of the wheel. This specimen holder shall be mounted so that not more than \(0.002\) in. (0.05 mm) of side play occurs at the line of contact between the specimen and the wheel. The specimen holder shall be so placed that the specimen is tangential to the wheel at the center line of the wheel and specimen. A 10-kg weight shall be attached to the other end of the specimen holder lever arm. With a lever advantage of two-to-one, a force of 20 kg is thus applied to the specimen at the line of contact.

6. Test Specimen

6.1 The specimen shall be at least \(\frac{3}{16}\) in. (5 mm) thick and have a surface area large enough so that the wear will be confined within its edges. The specimen may be as large as the specimen holder of the apparatus will permit. Dimensional tolerances are not important, but the specimen shall be flat within a maximum of 0.004 in./in. (mm/mm). Surface finish of the specimen is not important, except that ground blanks shall be cleaned with a suitable solvent to remove all oils or waxes, and unground blanks shall be grit blasted to remove all adhering foreign particles.

7. Procedure

7.1 Weigh the specimen on an analytical balance to the nearest 0.0001 g.
7.2 Determine the density in accordance with Test Method B 311.
7.3 Place the specimen in a specimen holder and fasten rigidly with set screws and pressure plates.
7.4 Insert the specimen holder into the abrasion wear test machine.
7.5 Release the 10-kg weight, causing the load to be applied to the specimen that is bearing against the wheel.
7.6 Plug the drain at the bottom of the vessel.
7.7 Pour aluminum oxide grit, 30 mesh, into the vessel to within 1 in. (25 mm) of the center of the wheel. If a different abrasive is used it should be so noted in the report. Add water to the aluminum oxide in the proportion of 1 cm³/4 g of grit.
7.8 Just as the water has seeped into the abrasive grit, start the rotation of the wheel and continue for 1000 revolutions (determine by means of a revolution counter). If the abrasive tends to accumulate at the end of the vessel opposite the specimen, stir and agitate with a rod to ensure that it is mixed into the slurry. Constantly check the wheel during the run to ensure that a thin film of abrasive slurry is evenly dispersed over the outside rim which is not in contact with the slurry.
7.9 Stop the rotation of the wheel after 1000 revolutions. Remove the drain block and flush the slurry out of the vessel. Do not re-use the abrasive grain; use fresh grain for every run.
7.10 Remove the sample from the specimen holder, rinse free of grit, and dry.
7.11 Weigh the specimen again to the nearest 0.0001 g.

8. Calculations
8.1 Calculate the abrasion resistance, \( A \), in cubic centimetres per revolution, as follows:

\[
A = \frac{L}{1000D} \times 10^5
\]

where:
\( L \) = weight loss, g, and
\( D \) = specimen density, g/cm³.
8.2 Calculate the wear number, \( W \), in reciprocal cubic centimetres, as follows:

\[
W = \frac{D}{L}
\]

9. Report
9.1 The manufacturer’s grade designation, the manufacturer, and the abrasion resistance or wear number shall be included in the report.

10. Precision and Bias
10.1 The precision depends upon the relative abrasive resistance of the carbide being tested and since this test method covers a broad range of materials it cannot be determined at this time.

11. Keywords
11.1 abrasion resistance; abrasive wear; abrasive wear resistance; cemented carbides; hardmetals; powder metallurgy; wear number
1. Scope

1.1 This specification covers iron-copper-tin-graphite sintered metal powder oil-impregnated bearings of one composition commonly known as diluted bronze.

1.2 The following safety hazards caveat pertains only to the test method described in this specification: This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   B 328 Test Method for Density, Oil Content, and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings
   E 9 Test Methods of Compression Testing of Metallic Materials at Room Temperature

3. Ordering Information

3.1 Orders for material under this specification shall include the following information:
   3.1.1 Dimensions and
   3.1.2 Certification (12.1).

4. Materials and Manufacture

4.1 Bearings shall be made by briquetting and sintering metal powders with an addition of graphite so as to produce finished parts conforming to the requirements of this specification.

5. Chemical Composition

5.1 The material shall conform to the requirements as to chemical composition prescribed in Table 1.

6. Physical Properties

6.1 Density—The density of bearings supplied fully impregnated with lubricant shall be 6.0 to 6.4 g/cm³.

6.2 Oil Content—Oil content of bearings shall not be less than 17 % when determined in accordance with Test Method B 328.

6.3 Radial Crushing Strength—Radial crushing strength determined as prescribed in 9.2 shall be not less than nor greater than the calculated value limits using the minimum and maximum K values given below and using the following equation:

   \[ P = \frac{KLT^2}{D - T} \]  

where:
   \( P \) = radial crushing strength (Note), lbs (or N);
   \( D \) = outside diameter of bearing, in. (or mm);
   \( T \) = wall thickness of bearing, in. (or mm);
   \( L \) = length thickness of bearing, in. (or mm); and
   \( K \) = strength constant with \( K_{\text{min}} = 22,000 \) psi (152 MPa) and \( K_{\text{max}} = 50,000 \) psi (345 MPa).

Note 1—Strengths above the maximum value indicate high combined carbon, which has a deleterious effect on bearing performance.

7. Workmanship, Finish, and Appearance

7.1 Bearings shall be uniform in composition. When cut or fractured, the exposed surface shall be of uniform appearance. The parts shall be free of defects which would affect their serviceability. Upon metallographic examination, the microstructure of the material shall be iron dispersed through a matrix of substantially alpha bronze and containing no free tin visible at 300x. To check for uniform permeability, the part shall be observed either while it is being heated or while the inside of the bearing is being subjected to air pressure. Oil shall exude uniformly from all critical surfaces.
8. Sampling

8.1 Lot—Unless otherwise specified, a lot shall consist of parts of the same form and dimensions made from powders of the same composition, formed and sintered under the same conditions, and submitted for inspection at one time.

8.2 Sample for Chemical Analysis—At least one sample for chemical analysis shall be taken from each lot. A sample shall consist of not less than 2 oz (56 g) of chips obtained by milling or drilling from at least two pieces with clean dry tools without lubrication. To obtain oil-free chips, the parts selected for test shall be extracted in accordance with Test Method B 328, if necessary.

8.3 Mechanical Tests—The manufacturer and purchaser shall agree on a representative number of test pieces.

9. Test Methods

9.1 Density—Test Method B 328.

9.2 Radial Crushing Strength—Compress the test specimens between two flat surfaces at a “no load” speed no greater than 0.2 in./min (5.0 mm/min), the direction of the load being normal to the longitudinal axis of the specimen. The point at which the load drops as a result of the first crack shall be considered the crushing strength. Apply this test to plain cylindrical bearings. Test flanged bearings by cutting off the flange and compressing the two sections separately. Each section shall meet the minimum strength requirements prescribed in 6.3.

10. Inspection

10.1 Unless otherwise specified, inspection of parts supplied on contract shall be made by the purchaser at the destination.

11. Rejection

11.1 Unless otherwise specified, rejection based on tests made in accordance with this specification shall be reported to the manufacturer within 30 days from receipt of the material by the purchaser.

12. Certification

12.1 A certification based on the manufacturer’s quality control that the material conforms to the requirements of this specification shall be the basis of shipment of the material. A certificate covering the conformance of the material to this specification shall be furnished by the manufacturer upon request of the purchaser.

13. Keywords

13.1 density; $K$ strength constant; oil content; oil-impregnated bearings; porosity

APPENDIX

(Nonmandatory Information)

X1. EXPLANATORY INFORMATION

X1.1 Design Information

X1.1.1 In calculating permissible loads, the operating conditions, housing conditions, and construction should be considered. The maximum static bearing load should not exceed 8500 psi (60 MPa) of projected bearing area (length times inside diameter of bearing) for this material. This figure is 75 % of the value for the compression deformation limit (yield strength, permanent set of 0.001 in. (0.025 mm) for specimens 1 1/8 in. (28.6 mm) in diameter and 1 in. (25.4 mm) in length) as determined in accordance with Test Methods E 9.

X1.2 Permissible Loads

X1.2.1 Permissible loads for various operating conditions are given in Table X1.1.

X1.3 Dimensional Tolerances

X1.3.1 Commercial dimensional tolerances are given in Table X1.2.

X1.4 Press Fits

X1.4.1 Plain cylindrical journal bearings are commonly installed by press fitting the bearing into a housing with an insertion arbor. For housings rigid enough to withstand the press fit without appreciable distortion and for bearings with wall thickness approximately one eighth of the bearing outside diameter, the press fits shown in Table X1.3 are recommended.

<table>
<thead>
<tr>
<th>TABLE X1.1 Permissible Loads</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shaft Velocity, ft/min (m/min)</td>
</tr>
<tr>
<td>Slow and intermittent</td>
</tr>
<tr>
<td>25 (7.6)</td>
</tr>
<tr>
<td>50 to 100 (15.2 to 30.4), incl</td>
</tr>
<tr>
<td>Over 100 to 150 (30.4 to 45.7), incl</td>
</tr>
<tr>
<td>Over 150 to 200 (45.7 to 61), incl</td>
</tr>
<tr>
<td>Over 200 (61)</td>
</tr>
</tbody>
</table>

*For shaft velocities over 200 ft/min (61 m/min), the permissible loads may be calculated as follows: $P = 40 000/V$

where:
$P$ = safe load, psi of projected area and
$V$ = shaft velocity, ft/min.

In SI units:
$P = 85/V$

where:
$P$ = is in MPa
$V$ = is in m/min and should not exceed 400 ft/min (122 m/min).

X1.5 Running Clearance

X1.5.1 Proper running clearance for sintered bearings depends to a great extent on the particular application. Therefore, only minimum recommended clearances are listed in Table
The maximum running clearances will automatically be held within good design practice for average conditions if dimensions shown in the standard size list in Table X1.5 are used. It is assumed that ground steel shafting will be used and that all bearings will be oil impregnated.

### Standard Sleeve Bearing Sizes

X1.6.1 The standard size list shown in Table X1.5 is adaptable to the basic shaft and basic hole systems through control of the insertion arbor diameter. It is based on dimensional tolerances shown in Table X1.2 and on tables of recommended press fits, Table X1.3, and running clearances, Table X1.4.

### Flange and Thrust-Bearing Specifications

X1.7.1 Diameter and thickness specifications for flange and thrust washers are shown in Table X1.6.

### Impregnating Oil

X1.8.1 It was found that a circulating-type oil containing rust and oxidation inhibitors is the most desirable type of oil to be used. The viscosity should be specified by the user in accordance with the application.
### TABLE X1.5 Standard Sleeve Bearing Size Specifications

<table>
<thead>
<tr>
<th>Inside Diameter</th>
<th>Wall Thickness, in.</th>
<th>Outside Diameter</th>
<th>Length</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fractional, in.</td>
<td>Fractional, in.</td>
<td>Decimal, in.</td>
<td>mm</td>
</tr>
<tr>
<td>1/8</td>
<td>1/32</td>
<td>3/16</td>
<td>0.127</td>
</tr>
<tr>
<td>1/4</td>
<td>1/32</td>
<td>1/16</td>
<td>0.253</td>
</tr>
<tr>
<td>5/32</td>
<td>1/32</td>
<td>1/16</td>
<td>0.315</td>
</tr>
<tr>
<td>3/16</td>
<td>1/32</td>
<td>3/16</td>
<td>0.401</td>
</tr>
<tr>
<td>5/32</td>
<td>1/16</td>
<td>5/32</td>
<td>0.440</td>
</tr>
<tr>
<td>1/4</td>
<td>1/16</td>
<td>7/32</td>
<td>0.504</td>
</tr>
<tr>
<td>5/16</td>
<td>1/16</td>
<td>7/16</td>
<td>0.687</td>
</tr>
<tr>
<td>3/8</td>
<td>1/16</td>
<td>1/2</td>
<td>0.752</td>
</tr>
<tr>
<td>7/16</td>
<td>1/16</td>
<td>1/2</td>
<td>0.879</td>
</tr>
<tr>
<td>1/2</td>
<td>1/16</td>
<td>5/8</td>
<td>0.937</td>
</tr>
<tr>
<td>5/8</td>
<td>1/16</td>
<td>3/4</td>
<td>1.126</td>
</tr>
<tr>
<td>3/4</td>
<td>1/16</td>
<td>7/8</td>
<td>1.379</td>
</tr>
<tr>
<td>1</td>
<td>1/16</td>
<td>1</td>
<td>1.630</td>
</tr>
<tr>
<td>1/2</td>
<td>1/4</td>
<td>1</td>
<td>1.879</td>
</tr>
<tr>
<td>1 1/8</td>
<td>1/4</td>
<td>1 1/2</td>
<td>2.505</td>
</tr>
<tr>
<td>1 1/2</td>
<td>1/4</td>
<td>1 3/4</td>
<td>3.006</td>
</tr>
</tbody>
</table>

### TABLE X1.6 Flange and Thrust Bearings Diameter and Thickness Tolerances

#### Flange Bearings, Flange Diameter Tolerances

<table>
<thead>
<tr>
<th>Diameter Range</th>
<th>Standard</th>
<th>Special</th>
</tr>
</thead>
<tbody>
<tr>
<td>in.</td>
<td>mm</td>
<td>in.</td>
</tr>
<tr>
<td>0 to 1 1/2</td>
<td>0 to 38</td>
<td>±0.005</td>
</tr>
<tr>
<td>Over 1 1/2 to 3</td>
<td>39 to 76</td>
<td>±0.010</td>
</tr>
<tr>
<td>Over 3 to 6</td>
<td>77 to 152</td>
<td>±0.025</td>
</tr>
</tbody>
</table>

#### Flange Bearings, Flange Thickness Tolerances

<table>
<thead>
<tr>
<th>Diameter Range</th>
<th>Standard</th>
<th>Special</th>
</tr>
</thead>
<tbody>
<tr>
<td>in.</td>
<td>mm</td>
<td>in.</td>
</tr>
<tr>
<td>0 to 1 1/2</td>
<td>0 to 38</td>
<td>±0.005</td>
</tr>
<tr>
<td>Over 1 1/2 to 3</td>
<td>39 to 76</td>
<td>±0.010</td>
</tr>
<tr>
<td>Over 3 to 6</td>
<td>77 to 152</td>
<td>±0.015</td>
</tr>
</tbody>
</table>

#### Thrust Bearings (1/4-in. (6.35-mm) Thickness, max), Thickness Tolerances for All Diameters

<table>
<thead>
<tr>
<th>Diameter Range</th>
<th>Standard</th>
<th>Special</th>
</tr>
</thead>
<tbody>
<tr>
<td>in.</td>
<td>mm</td>
<td>in.</td>
</tr>
<tr>
<td>0 to 1 1/2</td>
<td>0 to 38</td>
<td>0.003</td>
</tr>
<tr>
<td>Over 1 1/2 to 3</td>
<td>39 to 76</td>
<td>0.004</td>
</tr>
<tr>
<td>Over 3 to 6</td>
<td>77 to 152</td>
<td>0.005</td>
</tr>
</tbody>
</table>

Notes:

1. Standard and special tolerances are specified for diameters, thickness and parallelism. Special tolerances should not be specified unless required since they require additional or secondary operations and, therefore, are costlier.
2. Outside diameter tolerances are the same as for flange bearings.

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Standard Practice for Preparation of Molybdenum and Molybdenum Alloys for Electroplating

This standard is issued under the fixed designation B 629; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a procedure for preparing molybdenum and its alloys for electroplating. The procedure utilizes techniques, equipment, and chemicals that are common to electroplating. Specialized procedures such as hydrogen heat treatment and fused salt pickling are not included. These and other procedures can be found in the references listed at the end of this practice.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Significance and Use

2.1 Molybdenum is used in engineering applications where strength and rigidity of component parts are needed, especially at high temperatures. Its good strength at high temperatures, low vapor pressure, and good impact and creep resistance make it attractive for load-bearing items such as turbine blades, mechanical seals, rocket motor parts, heating elements, and electronic devices. Molybdenum, however, is easily oxidized, and the oxides are not protective. The volatility of the oxides above 800°C can lead, in the case of unprotected parts, to catastrophic oxidation and to losses in dimensions. Electroplated coatings are applied to molybdenum to prevent oxidation, to prevent seizing and galling, and to assist joining by soldering, brazing, and diffusion bonding.

3. Procedure

3.1 Precleaning:

3.1.1 The principles of electroplating on molybdenum are similar to those of other metals. The surfaces must be free of dirt, grease, and other foreign matter. Solvent, emulsion, and soak cleaners are suitable for precleaning to remove gross surface contaminants. If a molybdenum specimen is subjected to severe forming operations and retains a carbonaceous surface layer, descale the part by immersion in a 110 g/L solution of sodium hydroxide at or near boiling, or by mechanical finishing. Glass bead blasting can be used to remove uneven coatings of scale without disturbing the finish of the metal.

3.1.2 Cathodic alkaline cleaning is preferred for the final precleaning so as to avoid anodic oxidation. Proprietary formulations used for cathodic alkaline cleaning of other metals are suitable. Cathodic treatment at about 6 V for 30 to 60 s is usually adequate, using a cleaner concentration of 60 to 75 g/L and a solution temperature of 60 to 75°C.

3.2 Rinse—Here, and throughout the procedure, the water rinses should be warm (approximately 50°C).

3.3 Electropolishing—Either of the following electropolishes may be used. The choice of electropolish and time depends on the amount of material that can be removed and the degree of surface improvement desired. The cathodes may be lead or carbon. The sulfuric acid bath produces a blue film that is partly removed in the rinse and completely removed in the alkaline dip.

3.3.1 Sulfuric Acid:

<table>
<thead>
<tr>
<th>H₂SO₄ (sp gr 1.83)</th>
<th>80 mass % (755 mL of acid/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>20 to 30°C</td>
</tr>
<tr>
<td>Current density</td>
<td>1100 to 2200 A/m² anodic</td>
</tr>
<tr>
<td>Time</td>
<td>30 s</td>
</tr>
</tbody>
</table>

3.3.2 Sulfuric - Phosphoric Acid:

<table>
<thead>
<tr>
<th>H₃PO₄ (85 %)</th>
<th>50 % vol (500 mL/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>50 to 55°C</td>
</tr>
<tr>
<td>Current density</td>
<td>2500 A/m² anodic</td>
</tr>
<tr>
<td>Time</td>
<td>180 s</td>
</tr>
</tbody>
</table>

3.4 Rinse.

3.5 Alkaline Dip—Briefly immerse the specimen in a 45-g/L solution of sodium hydroxide at room temperature.

3.6 Rinse.

3.7 Acid Dip—Briefly immerse the specimen in a 10-mass % solution of sulfuric acid (58 mL of 1.83 sp gr acid/L) at room temperature.

3.8 Rinse.
3.9 Chromium Striking—Electroplate the specimen in a conventional chromium electroplating solution for 1 to 5 min at 1500 to 2500 A/m² at a temperature of 50°C minimum. In some cases, it may be necessary to first make the part anodic in the chromium solution for 1 min at 2200 A/m² in order to attain good adhesion.

3.10 Rinse.

3.11 Nickel Striking—Electroplate the specimen in the following solution at 800 A/m² for 120 s.

<table>
<thead>
<tr>
<th>NiCl₂·6H₂O</th>
<th>240 g/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>HCl (sp gr 1.18)</td>
<td>10 vol % (100 mL/L)</td>
</tr>
<tr>
<td>Temperature</td>
<td>20 to 30°C</td>
</tr>
<tr>
<td>Anode</td>
<td>nickel or carbon</td>
</tr>
</tbody>
</table>

3.12 Rinse.

3.13 Electroplate the specimen with the desired metal.

REFERENCES

(3) Domnikov, L., Metal Finishing, Vol 61, April 1963, p. 64.

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Standard Practice for Preparation of Chromium for Electroplating with Chromium

This standard is issued under the fixed designation B 630; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice is intended to serve as a process guide (not as a standard procedure) for producing adherent electrodeposits of chromium on chromium for engineering use.

1.2 Only well-known, generally practical methods using hexavalent chromium plating baths have been included. Some of the newer proprietary chromium plating processes produce chromium plating for which this process is unsuitable. Suppliers of the processes should be consulted.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 177 Guide for Chromium Electroplating on Steel for Engineering Use

3. Significance and Use

3.1 Electrodeposits of chromium on chromium are produced in cases where it is desired to obtain a better performing coating, to reclaim plated parts that are defective, or restore worn chromium coatings to original dimensions.

4. Preparation of Surface

4.1 Surface Finishing—The smoothness of the chromium coating before electroplating with chromium should closely approximate the requirements of the finished part. It is usually achieved by appropriate grinding with a diamond wheel.

4.2 Stripping and Basis Metal—With previously electroplated parts where the chromium deposit has been worn through, or where grinding has exposed the basis metal, preferred results are obtained by stripping the remaining chromium completely. However, it is possible to obtain satisfactory deposits on such substrates by careful attention to details in the pretreatment and electroplating process steps.

4.3 Stripping may be done by several methods, including proprietary processes or inhibited hydrochloric acid solution (10 to 20% by volume), or anodic treatment at 55 to 800 A/m² (5 to 75 A/ft²) in a solution of 35 to 50 g/L (5 to 7 oz/gal) of sodium hydroxide or 60 to 75 g/L (8 to 10 oz/gal) of sodium carbonate at 20 to 25°C (70 to 80°F).

4.4 Process Outline:

4.4.1 Preclean (see 5.1.1 and 5.1.2),
4.4.2 Alkaline electrolytic clean (see 5.1.3),
4.4.3 Rinse (cold water),
4.4.3.1 Rinse (hot water),
4.4.3.2 Dry,
4.4.3.3 Mask (see 5.2),
4.4.4 Activate (see 5.3),
4.4.5 Chromium electroplate,
4.4.6 Rinse (cold water),
4.4.7 Rinse (hot water), and
4.4.8 Dry.

5. Procedure

5.1 Cleaning—The following cleaning treatments may be used for all conditions and types of electrodeposited chromium. The choice of the procedures will be governed largely by the condition of the surface.

5.1.1 Precleaning—When considerable amounts of grease and oil are present on the surface, precleaning (emulsion or alkaline spray or soak) is necessary to remove the bulk of the soil.

5.1.2 Abrasive Cleaning—Oxide and other films may be removed by mechanical techniques using such abrasives as fine emery on a cloth or a wheel, pumice slurry, or greaseless buffing compounds. Grit blasting, shot blasting, wet blasting, or vapor honing may also be used. Abrasive cleaning is generally not necessary when suitable mechanical finishing procedures have been used.

5.1.3 Cathodic Alkaline Cleaning—Removal of final traces of dirt, grease, and oil is best accomplished by cathodic alkaline cleaning. Cathodic electroleaching is necessary because anodic electroleaching will strip chromium and may expose the basis metal. In cathodic cleaning, the parts are negatively charged and attract positively charged metallic ions, soaps, and other colloidal materials in the solution, causing them to “plate out” as loose smut on the surfaces being cleaned. Thus, do not allow the cleaning solution to become heavily contaminated with dirt, grease, or oil.
5.2 **Masking**—Rigid, nonconductive materials or stop-offs, which are especially compounded nonconductive tapes, waxes, lacquers, or polymeric materials, will protect areas that are to remain free of chromium. Due to the varying kinds of chromium electroplating solutions and interactions with these materials, vendor instructions should be followed.

5.3 **Activating**:

5.3.1 The procedure used for etching or activating the chromium surface is most important and usually determines the soundness of the adhesion. Chromium exhibits a strong tendency to acquire surface oxides rapidly (air passivity) that reduce the adhesion of subsequent chromium deposits. Anodic etching in a chromic acid solution, 250 g/L, at room temperature is recommended. In some instances, this procedure may be performed in the chromium electroplating solution.

5.3.2 Give the part an anodic etch for 5 to 60 s at 6 V. For effective control, confirm etch action by visual observation of uniform gassing; increase etch time if necessary. When basis metal is exposed or high-speed chromium electroplating solutions are used, keep the time as short as possible.

5.3.3 When the part to be electroplated has a large mass, allow it to reach the temperature of the electroplating bath before etching.

5.4 **Chromium Electroplating**:

5.4.1 Make the part cathodic up to 3.0 V. Slowly increase the voltage in steps of 0.25 V so that gassing and electroplating start after 30 to 60 s and full current is reached in 5 min.

5.4.2 The composition of the ordinary or "conventional" solution is 250 to 400 g/L of chromic acid (CrO₃) and sulfate radical (SO₄²⁻) to maintain a ratio by weight of CrO₃ to SO₄²⁻ of 100 to 1, although many electroplaters prefer ratios as low as 80 to 1, which facilitates electroplating chromium on chromium. The sulfate is generally added as sulfuric acid (94 to 96 mass % H₂SO₄). Refer to Guide B 177 for more information on the operation of the electroplating solution.

5.4.3 Some proprietary baths contain a supplementary catalyst in the form of fluorides or other mixed catalysts. Some of these baths are satisfactory, but some could be unsuitable. The supplier of any proprietary bath should be consulted for specific information.

6. **Keywords**

6.1 activation; chromium; cleaning; preparation; striking
Standard Specification for Electrodeposited Coatings of Zinc on Iron and Steel

This standard is issued under the fixed designation B 633; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

e1 Note—In Section 2 and Paragraph 8.1.5, Guide F 1470 was incorrectly cited as Guide F 1740. This was editorially corrected in August 2001.

1. Scope

1.1 This specification covers requirements for electrodeposited zinc coatings applied to iron or steel articles to protect them from corrosion. It does not cover electrodeposited zinc-coated steel wire or sheets (see Specification A 591/A 591M for sheets).

1.2 The coatings are provided in four standard thickness classes (4.1), in the as-plated condition or with one of three types of supplementary finish (4.2).

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
A 591/A591M Specification for Steel Sheet, Electrolytic Zinc-Coated, for Light Coating Mass Applications
B 117 Practice for Operating Salt Spray (Fog) Apparatus
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 201 Practice for Testing Chromate Coatings on Zinc and Cadmium Surfaces
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 254 Practice for Preparation of and Electroplating on Stainless Steel
B 320 Practice for Preparation of Iron Castings for Electroplating
B 322 Practice for Cleaning Metals Prior to Electroplating
B 374 Terminology Relating to Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
B 571 Test Methods for Adhesion of Metallic Coatings
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 762 Test Method of Variables Sampling of Metallic and Inorganic Coatings
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 676 Test Method of Variables Sampling of Metallic and Inorganic Coatings
D 2092 Guide for Treatment of Zinc-Coated (Galvanized) Steel Surfaces for Painting
F 1470 Guide for Fastener Sampling for Specified Mechanical Properties and Performance Inspection
MIL-STD-1312 Fastener Tests, Methods (Test 12)

2.2 Military Standard:

2.3 Terminology

3.1 Definitions—Definitions of the terms used in this specification are in accordance with Terminology B 374.

4. Classification

4.1 Thickness—The coating shall be provided in the four thickness classes defined in Table 1.

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1 This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.08.04 on Light Metals.


2 Annual Book of ASTM Standards, Vol 01.06.
3 Annual Book of ASTM Standards, Vol 03.02.
4 Annual Book of ASTM Standards, Vol 02.05.

5 Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.
coating. Type II for SC 4 and SC 3 and Type III for SC 2 and SC 1.

4.2 Finish—The coating shall have one of the finish types defined as follows:

<table>
<thead>
<tr>
<th>Type</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>As-plated without supplementary treatment</td>
</tr>
<tr>
<td>II</td>
<td>With colored chromate conversion coatings</td>
</tr>
<tr>
<td>III</td>
<td>With colorless chromate conversion coatings</td>
</tr>
<tr>
<td>IV</td>
<td>With phosphate conversion coatings</td>
</tr>
</tbody>
</table>

5. Ordering Information

5.1 When ordering the electroplating of articles, the purchaser shall state the designation number, the date of issue, the class or service condition number, and the type. (See 4.1, 4.2, and 7.1).

5.2 If necessary, the purchaser shall include on his part drawings or purchase order the following:

5.2.1 Electroplating application to high strength steel, if specified (6.4).

5.2.2 Thickness, if other than specified (4.1, 7.1).

5.2.3 Location of significant surface (7.1.1, 7.1.2).

5.2.4 Luster (7.3).

5.2.5 Corrosion resistance test, if specified (9.3, 10.3).

5.2.6 Hydrogen embrittlement test, if required (9.4, 10.4).

5.2.7 Sample size for inspection, if other than specified, and

5.2.8 Supplementary requirements, if applicable (Supplementary Requirement).

6. Materials and Manufacture

6.1 The coatings shall be essentially pure zinc produced by electrodiposition.

6.2 Defects in the surface of the basis metal, such as scratches, porosity, pits, inclusions, cracks, roll marks, and die marks, may adversely affect the appearance and performance of coatings applied thereto despite the observance of the best electroplating practices. Accordingly, the electroplater’s responsibility for defects in the coating resulting from such conditions shall be waived, except when he is the prime contractor supplying electroplated parts. In this event, the basis metal shall be subjected to such polishing or buffing operations as are necessary to yield deposits with the desired final luster and appearance. To minimize problems of this sort, the specifications covering the basis material on the item to be electroplated shall contain appropriate limitations to such basis metal conditions.

6.3 Cleaning of Basis Metal—Proper preparatory procedures and thorough cleaning of the basis metal are essential to ensure satisfactory adhesion and corrosion resistance performance of the coating. It is recommended that the following appropriate recommended practices be used: B 183, B 242, B 254, B 320, and B 322.

6.4 High-Tensile Strength Metals—Unless otherwise specified, high strength steels having a tensile strength greater than 1700 MPa shall not be electroplated.

6.5 Stress Relief—All steel parts having an ultimate tensile strength of 1000 MPa and above, and that have been machined, ground, cold formed or cold straightened, shall be heat treated at a minimum of 190°C for 3 h or more for stress relief before cleaning and electroplating.

6.6 Hydrogen Embrittlement Relief—All electroplated parts having a tensile strength of 1200 MPa or higher shall be baked at a minimum of 190°C for 3 h or more within 4 h after electroplating to provide hydrogen embrittlement relief. Electroplated springs and other parts subject to flexure shall not be flexed before the hydrogen embrittlement relief treatment. The baking treatment shall be done before the application of the supplementary treatments. Baked parts shall not crack or fail by fracture when tested in accordance with 10.4.

6.7 Reactivation Treatment—Electroplated surfaces passivated as a result of the baking operation shall be reactivated before receiving a supplementary treatment. Surfaces intended for supplementary treatments (Types II and III) may be reactivated by immersion in a dilute acid solution. Surfaces should be activated as soon as possible following baking and should be handled carefully to avoid contamination.

6.8 Supplementary Treatments—The supplementary film treatment for Types II and III shall be in accordance with Practice B 201. The treatment required for conversion to Type IV shall be in accordance with Guide D 2092.

NOTE 1—The zinc surface is attacked by supplementary treatments, thereby diminishing the amount of metallic zinc present. With Classes Fe/Zn25 and Fe/Zn13, this reduction is insignificant; but it is significant with Fe/Zn8 and Fe/Zn5. Therefore, it is recommended that supplementary treatments not be applied to zinc coatings having a nominal thickness less than 5 µm.

7. Coating Requirements

7.1 Thickness—The thickness shall be specified in accordance with 4.1 and 5.1.

7.1.1 Significant Surfaces—Significant surfaces are areas in which minimum thicknesses to be met shall be designated on the applicable drawing or by the provision of a suitably marked sample. Significant surfaces may be defined as those normally visible, directly or by reflection, which are essential to the appearance or serviceability of the article when assembled in normal position or which can be the source of corrosion products that deface visible surfaces on the assembled article.

7.1.2 Surfaces on which the specified thickness of deposit cannot readily be controlled, such as threads, holes, deep recesses, bases of angles, and similar areas, are normally exempt from minimum thickness requirements, unless they are specially designated as not exempt. When such areas are designated, and thus made subject to minimum thickness requirements, the purchaser and the manufacturer shall recognize the necessity for either thicker deposits on other areas or for special racking.

NOTE 2—The dimensional tolerance of most threaded articles, such as nuts, bolts, screws, and similar fasteners with complementary threads, normally does not permit the application of a coating thickness much
greater than 8.0 μm. If heavier coatings are required, allowance for the
deposit buildup must be made during the manufacture of the threaded
articles.

7.2 Adhesion—The adhesion of the coating shall be such
that when examined in accordance with 10.2, the coating shall
not show separation from the basis metal at the interface.

7.3 Luster—Unless otherwise specified by the purchaser, a
bright, semibright, or dull luster shall be acceptable.

7.4 Corrosion Resistance—Zinc coatings with Types II and
III treatments shall show neither corrosion products of zinc nor
basis metal corrosion products at the end of the test period, as
shown below, when tested by continuous exposure to salt spray
in accordance with 10.3. The appearance of corrosion products
visible to the unaided eye at normal reading distance shall be
cause for rejection except that white corrosion products at the
edges of specimens shall not constitute failure.

<table>
<thead>
<tr>
<th>Corrosion Resistance Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Types</td>
</tr>
<tr>
<td>II</td>
</tr>
<tr>
<td>III</td>
</tr>
</tbody>
</table>

8.1 The purchaser and producer are urged to use statistical
process control in the coating process. Properly performed,
statistical process control will assure coated products of
satisfactory quality and will reduce the amount of acceptance
inspection. The sampling plan used for the inspection of the
quality coated article shall be agreed upon between the
purchaser and producer.

8.1.1 When a collection of coated articles (inspection lot,
see 8.2) is examined for compliance with the requirements
placed on the articles, a relatively small number of the articles
(sample) is selected at random and is inspected. The inspection
lot is then classified as complying with the requirements based
on the results of the inspection of the sample. The size of the
sample and the criteria for compliance are determined by the
application of statistics. The procedure is known as sampling
inspection. Test Method B 602, Guide B 697, and Test Method
B 762 contain sampling plans that are designed for sampling
inspection of coatings.

8.1.2 Test Method B 602 contains four sampling plans, three
for use with tests that are nondestructive and one when they are
destructive. Test Method B 602 provides a default plan if one
is not specified.

8.1.3 Guide B 697 provides a large number of plans and
also gives guidance in the selection of a plan. Guide B 697
provides a default plan if one is not specified.

8.1.4 Test Method B 762 can be used only for coating
requirements that have a numerical limit, such as coating
thickness. The test must yield a numeric value and certain
statistical requirements must be met. Test Method B 762
contains several plans and also gives instructions for calculating
plans to meet special needs. Test Method B 762 provides a
default plan if one is not specified.

8.1.5 Guide F 1470 can be used for fasteners such as
internally threaded, externally threaded, and nonthreaded fasteners
and washers. This guide provides for two plans: one
designated the “detection process” and one designated the
“prevention process.” The purchaser and producer shall agree
on the plan to be used.

8.2 An inspection lot shall be defined as a collection of
coated articles that are the same kind, that have been produced
to the same specification, that have been coated by a single
supplier at one time or approximately the same time, under
essentially identical conditions, and that are submitted for
acceptance or rejection as a group.

9. Specimen Preparation

9.1 Electroplated Parts or Separate Specimens—When the
electroplated parts are of such form, shape, size, and value as
to prohibit use thereof, or are not readily adaptable to a test
specified herein, or when destructive tests of small lot sizes are
required, the test shall be made by the use of separate
specimens plated concurrently with the articles represented.
The separate specimens shall be of a basis metal equivalent to
that of the articles represented. “Equivalent” basis metal
includes chemical composition, grade, condition, and finish of
surface before electroplating. For example, a cold-rolled steel
surface should not be used to represent a hot-rolled steel
surface. Because of the impracticality of forging or casting
separate test specimens, hot-rolled steel specimens may be
used to represent forged and cast steel articles. The separate
specimens may also be cut from scrap castings when ferrous
alloy castings are being electroplated. These separate
specimens shall be introduced into a lot at regular intervals before
the cleaning operations, preliminary to electroplating, and shall
not be separated therefrom until after completion of electro-
plating. Conditions affecting the electroplating of specimens,
including the spacing, plating media, bath agitation, tempera-
ture, and so forth, in respect to other objects being electro-
plated, shall correspond as nearly as possible to those affecting
the significant surfaces of the articles represented. Unless a
need can be demonstrated, separately prepared specimens shall
not be used in place of production items for nondestructive and
visual examinations.

9.2 Thickness and Adhesion Specimens—If separate speci-
mens for thickness and adhesion tests are required, they shall
be strips approximately 25 mm wide, 100 mm long, and 1 mm
thick.

9.3 Corrosion Resistance Specimens—If separate speci-
mens for corrosion resistance tests are required, they shall be
panels not less than 150 mm long, 100 mm wide, and
approximately 1 mm thick.

9.4 Hydrogen Embrittlement Specimens—If specimens are
required, the configuration shall be that specified by the
purchaser.
10. Test Methods

10.1 Thickness:

10.1.1 The thickness of the coating may be determined by Test Methods B 487, B 499, B 504, B 567, or B 568, as applicable.

10.1.2 MIL-STD-1312, Test 12, may be used for thickness measurements of electroplated fastener hardware.

10.1.3 Other methods may be used if it can be demonstrated that the uncertainty of the measurement with these methods is less than 10%.

10.1.4 Make thickness measurements of zinc electroplatings, Types II, III, and IV, after application of the supplementary treatments. When Test Methods B 504, B 567, or B 568 are used, remove the supplementary treatment before testing. The chromate film may be removed from Type II and Type III coatings by using a very mild abrasive (a paste of levigated alumina rubbed on with the finger). The phosphate coating may be removed from Type IV coating by a concentrated (28%) ammonia solution that quickly dissolves the phosphate coating but does not attack the underlying zinc.

10.2 Adhesion—Determine adhesion by any suitable procedure in accordance with Test Method B 571.

10.3 Corrosion Resistance—When specified in the contract or purchase order, determine the corrosion resistance in accordance with Practice B 117. Subject the selected samples to the salt spray test; the length of time to be applicable for the type of supplementary coating shall be in accordance with the requirements of 7.4. To secure uniformity of results, age Types II and III supplementary coatings at room temperature for 24 h before subjection to the salt spray.

10.4 Hydrogen Embrittlement Relief—When specified in the contract or purchase order, prepare and test the satisfactory behavior of parts to indicate freedom from hydrogen embrittlement.

10.5 Visual Examination—Examine material for compliance with the requirements of luster (7.3) and workmanship (7.5) after electroplating.

11. Rejection

11.1 Coatings not conforming to this specification or to authorized modification shall be rejected. They may be reconsidered for inspection in accordance with Test Method B 602.

12. Certification

12.1 The purchaser may require in the purchase order or contract that the producer or supplier provide certification that the finish was produced and tested in accordance with this specification and found to meet the requirements. The purchaser may similarly require that a report of the test results be furnished.

13. Packaging and Packing

13.1 Preservation, packaging, and packing methods for zinc-electroplated parts or articles used by a supplier shall be such as to preclude damaging during shipment and handling.

14. Keywords

14.1 electrodeposited coatings; zinc; zinc, electrodeposited coatings

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirement shall apply only when specified by the purchaser as part of the purchaser’s order or contract and for all agencies of the United States Government.

S1. Responsibility for Inspection

S1.1 The producer or supplier shall be responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein unless disapproved by the purchaser. The purchaser retains the right to perform any of the inspections and tests set forth in this specification where such inspections and tests are deemed necessary to ensure that supplies and services conform to the prescribed requirements.
X1. SERVICE LIFE OF ZINC

X1.1 The service life of zinc coating is a function of its thickness and the type of environment to which it is exposed. While the conditions of exposure and uses of electroplated metal are so varied that it is not definitely possible to predict the exact life of articles protected by a coating of a given thickness, those using zinc coatings can draw on the wealth of practical experience at hand, supplemented by results of corrosion tests carried out over the years. As a result of large-scale and long-range tests conducted by ASTM and other organizations, there is excellent information on the corrosion behavior of zinc and zinc coatings. The following data, based on worldwide testing, may be used to compare the behavior of electrodeposited coatings of zinc in various atmospheres. The values are only relative, since individual studies in various parts of the world have resulted in figures which vary widely from these averages.

<table>
<thead>
<tr>
<th>Atmosphere</th>
<th>Mean Corrosion Rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Industrial</td>
<td>5.6 µm/year</td>
</tr>
<tr>
<td>Urban nonindustrial or marine</td>
<td>1.5 µm/year</td>
</tr>
<tr>
<td>Suburban</td>
<td>1.3 µm/year</td>
</tr>
<tr>
<td>Rural</td>
<td>0.8 µm/year</td>
</tr>
<tr>
<td>Indoors</td>
<td>considerably less than 0.5 µm/year</td>
</tr>
</tbody>
</table>

Note: X1.1—The mean corrosion rate given pertains to zinc only and does not include a corrosion rate when zinc is passivated or in contact with other materials.

X2. EXAMPLES OF APPROPRIATE SERVICE CONDITIONS AND DESCRIPTION OF SERVICE CONDITIONS

X2.1 SC 4—Very Severe—Exposure to harsh conditions, or subject to frequent exposure to moisture, cleaners, and saline solutions, plus likely damage by denting, scratching, or abrasive wear. Examples are: plumbing fixtures, pole line hardware.

X2.2 SC 3—Severe—Exposure to condensation, perspiration, infrequent wetting by rain, and cleaners. Examples are: tubular furniture, insect screens, window fittings, builder’s hardware, military hardware, washing machine parts, bicycle parts.

X2.3 SC 2—Moderate—Exposure mostly to dry indoor atmospheres but subject to occasional condensation, wear, or abrasion. Examples are: tools, zippers, pull shelves, machine parts.

X2.4 SC 1—Mild—Exposure to indoor atmospheres with rare condensation and subject to minimum wear or abrasion. Examples are: buttons, wire goods, fasteners.
Standard Specification for Electrodeposited Coatings of Rhodium for Engineering Use

This standard is issued under the fixed designation B 634; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

e1 Note—Keywords were added editorially in April 2004.

1. Scope

1.1 This specification covers requirements for electrodeposited coatings of rhodium used for engineering purposes.

1.2 Coatings of rhodium covered by this specification are usually employed for their corrosion resistance, stable electrical contact resistance, wear resistance, reflectivity, and heat resistance.

1.3 Appendix X1 covers some typical applications for electrodeposited rhodium.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 The following documents form a part of this document to the extent referenced herein.

2.2 ASTM Standards:

B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 252 Guide for Preparation of Zinc Alloy Die Castings for Electroplating and Conversion Coatings
B 254 Practice for Preparation of and Electroplating on Stainless Steel
B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings
B 322 Practice for Cleaning Metals Prior to Electroplating
B 343 Practice for Preparation of Nickel for Electroplating with Nickel
B 456 Specification for Electrodeposited Coatings of Copper Plus Nickel Plus Chromium and Nickel Plus Chromium
B 481 Practice for Preparation of Titanium and Titanium Alloys for Electroplating
B 482 Practice for Preparation of Tungsten and Tungsten Alloys for Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
B 507 Practice for Design of Articles to Be Electroplated on Racks
B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 762 Test Method of Variables Sampling of Metallic and Inorganic Coatings
E 8 Test Methods of Tension Testing of Metallic Materials
2.3 Military Standards:

MIL-R-46085 Rhodium Plating, Electrodeposited
QQ-N-290 Nickel Plating, Electrodeposited

3. Classification

3.1 Electrodeposited coatings of rhodium on the basis of thickness are classified as follows:

<table>
<thead>
<tr>
<th>Class</th>
<th>Minimum Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>0.5</td>
<td>0.5</td>
</tr>
<tr>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>2.5</td>
<td>2.5</td>
</tr>
</tbody>
</table>

This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.08.02 on Precious Metal Coatings.


3 Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.
4. Ordering Information

4.1 To make application of this standard complete, the purchaser needs to supply the following information to the seller in the purchase order or other governing document:

4.1.1 Name, designation, and year of issue of this standard,
4.1.2 Class including a maximum thickness, if appropriate (3.1),
4.1.3 Nature of substrate, for example, high strength steel, need for stress relief, undercoats, embrittlement relief (5.1, 5.2, 5.3),
4.1.4 Significant surfaces (6.2),
4.1.5 Appearance (6.3),
4.1.6 Requirements and methods of testing for one or more of the following requirements: need for and type of test specimens (8.1); thickness (8.2); adhesion (8.3); absence of embrittlement (8.4); reflectivity (8.5); and undercoats (S1.4), and
4.1.7 Sampling plans (Section 7) and quality assurance (S1.2).

5. Process Requirements

5.1 Preparatory Procedures—The basis metal shall be subjected to such cleaning procedures as are necessary to ensure a surface satisfactory for subsequent electroplating. Materials used for cleaning shall have no damaging effects on the basis metal resulting in pitting, intergranular attack, stress corrosion cracking, or hydrogen embrittlement.

NOTE 1—For basis metal preparations, the following appropriate ASTM standards are recommended: Practices B 183, B 242, B 252, B 254, B 281, B 322, B 343, B 481, and B 482.

5.2 Preplating Operations:

5.2.1 Electroplating shall be applied after all basis metal heat treatments and mechanical operations such as forming, machining, and joining of the article have been completed.

5.2.2 Stress Relief Treatment—All steel parts having an ultimate tensile strength of 1050 MPa (approximately 35 HRC) and above and that have been machined, ground, cold formed, or cold straightened shall have heat treatment to 190 ± 15°C for a minimum of 5 h before cleaning and electroplating.

5.3 Post-Plating Procedures:

5.3.1 Embrittlement Relief—Steel parts having an ultimate tensile strength of 1200 MPa (approximately 38 HRC) or greater shall be baked at 190 ± 15°C for a minimum of 8 h within 4 h after electroplating to provide hydrogen embrittlement relief. Steel parts having an ultimate tensile strength greater than 1300 MPa (approximately 40 HRC) shall be baked at 190 ± 15°C for a minimum of 23 h within 4 h after electroplating.

6. Coating Requirements

6.1 Nature of Coating—The coating shall be essentially pure rhodium produced by electrodeposition from aqueous electrolytes.

6.2 Thickness—The rhodium coating thickness on all significant surfaces shall conform to the requirements of the specified class as defined in Section 3.

6.3 Significant Surfaces—Significant surfaces are defined as those normally visible (directly or by reflection) or are essential to the serviceability or function of the article or which can be the source of corrosion products or tarnish films that interfere with the function or desirable appearance of the article. When necessary, the significant surfaces shall be indicated on the drawings of the parts, or by the provision of suitably marked samples.

NOTE 2—Variation in the coating thickness from point-to-point on a coated article is an inherent characteristic of electroplating processes. Therefore, the coating thickness will have to exceed the specified value at some points on the significant surfaces to ensure that the thickness equals or exceeds the specified value at all points. Therefore, the average coating thickness on an article will usually be greater than the specified value; how much greater is largely determined by the shape of the article (see Practice B 507) and the characteristics of the electroplating process. Additionally, the average coating thickness on an article will vary from article to article within a production lot. If all of the articles in a production lot are to meet the thickness requirement, the average coating thickness of the production lot as a whole will be greater than the average necessary to ensure that a single article meets the requirements.

6.4 Appearance:

6.4.1 Electroplated coatings shall completely cover all surfaces as specified in the manufacturing document and shall have a uniform appearance to the extent that the nature of the basis metal and good commercial practices permit.

6.4.2 Defects in the surface of the basis metal such as scratches, pits, nonconductive inclusions, and roll and die marks, may adversely affect the appearance and performance of the applied coatings. Such defects that persist in the finish despite the observance of good metal finishing practices shall not be cause for rejection.

NOTE 3—Applied finishes generally perform better in service when the substrate over which they are applied is smooth and free from torn metal, inclusions, pores, and other defects. It is recommended that the specifications covering the unfinished product provide limits for those defects. A metal finisher often can remove defects through special treatments such as grinding, polishing, abrasive blasting, chemical treatments, and electropolishing. However, these are not normal in the treatment steps preceding the application of the finish. When they are desired, they are the subject of special agreement between the purchaser and the supplier.

6.5 Adhesion—The rhodium coatings shall be free of blisters and peeled areas when tested in accordance with 8.3.

7. Sampling

7.1 A random sample of the size required by Test Methods B 602 or B 762 shall be selected from the inspection lot (see 7.2). The articles in the lot shall be inspected for conformance to the requirements of this specification and the lot shall be classified as conforming or nonconforming to each requirement according to the criteria of the sampling plans in Test Method B 602.

NOTE 4—Test Method B 602 contains four sampling plans, three for use with nondestructive test methods. The fourth is to be used with destructive test methods. The three methods for nondestructive tests differ in the quality level they require of the product. Test Method B 602 requires use of the plan with the intermediate quality level unless the purchaser specifies otherwise. It is recommended that the purchaser compare the plans with his needs and state which plan is to be used. If the plans in Test Method B 602 do not serve the needs, additional ones are
given in Guide B 697 which provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and seller need to agree on the plan to be used.

Note 5—Test Method B 762 is a variables sampling plan. Such plans can only be used when a test yields a measured quantity, such as thickness, and when the requirements are stated as a numerical limit also such as thickness. Test Method B 762 contains several plans and gives instructions for calculating plans for special needs. Buyer and seller may agree on the plan or plans to be used; if not Test Method B 762 identifies the plan to be used.

Note 6—When both destructive and nondestructive tests exist for the measurement of a characteristic, the purchaser needs to state which is to be used so that the proper sampling plan is selected. Whether or not a test is destructive may not always be clear. A test may destroy the coating but in a noncritical area. The purchaser needs to state whether the test is to be considered destructive or nondestructive. The decision is important because the plans for destructive tests are significantly less able to discriminate between acceptable and unacceptable lots. This is because fewer parts are tested.

7.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specifications, that have been coated by a single supplier at one time or at approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

7.3 If special test specimens are used to represent the coated articles in a test, the specimens shall be of the nature, size, and number, and shall be processed as required in accordance with 8.1.

8. Test Methods

8.1 Special Test Specimens:

8.1.1 Special test specimens are used to represent the coated articles in a test if the articles are of a size, shape, or material that is not suitable for the test, or if it is preferred not to submit articles to a destructive test because, for example, the articles are expensive or few in number. The permission or the requirement to use special test specimens, their number, the material from which they are to be made, and their shape and size shall be stated by the purchaser.

8.1.2 The special test specimen shall duplicate those characteristics of the article that influence the property being tested, and it shall be processed with the article through those process steps that influence the property.

8.1.2.1 The special test specimens used to represent an article in an adhesion, corrosion resistance, or appearance test shall be made of the same material, shall be in the same metallurgical condition, and shall have the same surface condition as the articles they represent; they shall be placed in the production lot of and be processed along with the articles they represent.

8.1.2.2 Special test specimens used to represent an article in a coating thickness test shall be introduced into the process at the point where the coating or coatings are applied and shall be carried through all steps that have a bearing on the coating thickness.

Note 7—When special test specimens are used to represent a coated article in a thickness test, the specimens will not necessarily have the same thickness and thickness distribution as the article unless the specimens and the article are of the same general size and shape. Therefore, before coated articles may be accepted on the basis of a thickness test performed on test specimens, the relationship between the thickness on special test specimens and the thickness on the part needs to be established. The criterion of acceptance is that thickness on the specimen that corresponds to the required thickness on the article.

8.2 Thickness:

8.2.1 The coating thickness shall be measured at locations on the significant surfaces where it would be expected to be a minimum.

8.2.2 Use Test Method B 567 for beta backscatter or Test Method B 568 for X-ray fluorescence (as applicable) for nondestructive methods.

8.2.3 Rhodium thickness can be measured destructively by Method B 487 for microscopical cross sectioning. This method shall not be used for thicknesses less than 2.5 µm.

8.3 Adhesion—Determine adhesion in accordance with one of the procedures given in Practice B 571, Bend Tests and Heat Quench Test.

8.4 Hydrogen Embrittlement Relief:

8.4.1 Steel products that are required by 5.3 to be heat treated to remove hydrogen embrittlement shall be subjected for 200 ± 8 h to a sustained tensile load equal to 75 ± 2 % of the ultimate tensile notch strength of the material. If the product fractures during the test or is found, when examined after the test, to have cracks, it does not conform to the requirement for hydrogen embrittlement relief.

8.4.2 Separate specimens for embrittlement relief test may be used. If they are used, they shall be round notched specimens with the axis of the specimen (load direction) perpendicular to the short transverse grain flow direction. The configuration shall be in accordance with the appropriate figure for round specimens of Test Methods E 8. Specimens shall have a 60° ± 10° V-notch located approximately at the center of the gage length. The cross-sectional area at the root of the vee shall be approximately equal to half the area of the full cross-sectional area of the specimen’s reduced section. The vee shall have a 254 ± 0.013 mm radius of curvature at the base of the notch. The separate specimens shall be of the same basis metal as that of the articles represented.

Note 8—The manufacturer of the basis metal parts should provide the coating facility with notched tensile specimens when required for testing.

8.5 Reflectivity—Measure in the spectral region of 0.45 ± 0.05 µm on a coated surface using a device comparable to that in Fig. 1 with angles of incidence and reflectance at 45 ± 2°. Standard for reflectance shall be specified by the purchaser. The light source shall provide uniform energy in the spectral region with beam collimation and detector size and position to collect all the reflected energy for measurement.

9. Keywords

9.1 connectors; contacts; electrical connectors; electrical contacts; engineering coatings; reflectors; relays; relay switches; rhodium; rhodium coatings; rhodium electrodeposits; rhodium platings
SUPPLEMENTARY REQUIREMENTS

S1.1 The following supplementary requirements shall apply only when specified by the purchaser in the contract or order and for all agencies of the U.S. Government.

S1.2 Quality Assurance:

S1.2.1 Inspection Responsibility—The producer or supplier shall be responsible for the performance of all inspection and test requirements specified herein. Except as otherwise specified in the contract or order, the producer or supplier may use his own or any other suitable facilities for the performance of the inspection and test requirements specified herein, unless disapproved by the purchaser. The purchaser shall have the right to perform any of the inspections and tests set forth in this specification where such inspections are deemed necessary to assure that material conforms to the prescribed requirements.

S1.3 Reflectivity—Reflectivity shall be a minimum of 75% when measured in accordance with 8.5.

S1.4 Undercoats—Surfaces other than gold, platinum, or silver shall be electroplated with nickel or with copper followed by nickel in accordance with B 456 except for the thickness requirement. Corrosion or heat resistant steels shall have a minimum of 2.5 µm of nickel or as otherwise specified by the purchaser.

NOTE S1.1—Military Standard QQ-N-290 may be consulted for reference.

S1.5 Thickness Cross Reference Table:

<table>
<thead>
<tr>
<th>Thickness Cross Reference Table:</th>
</tr>
</thead>
<tbody>
<tr>
<td>B 634 (rev.)</td>
</tr>
<tr>
<td>Class (µm)</td>
</tr>
<tr>
<td>0.05</td>
</tr>
<tr>
<td>...</td>
</tr>
<tr>
<td>0.25</td>
</tr>
<tr>
<td>0.50</td>
</tr>
<tr>
<td>1</td>
</tr>
<tr>
<td>...</td>
</tr>
<tr>
<td>2.5</td>
</tr>
<tr>
<td>...</td>
</tr>
<tr>
<td>6.25</td>
</tr>
</tbody>
</table>

APPENDIX

(Nonmandatory Information)

X1. TYPICAL APPLICATIONS FOR ELECTRODEPOSITED RHODIUM

<table>
<thead>
<tr>
<th>Application</th>
<th>Thickness Requirement, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reflectors, Tarnish Resistance</td>
<td>0.05 to 0.25</td>
</tr>
<tr>
<td>Light duty electrical contacts</td>
<td>0.25 to 0.80</td>
</tr>
<tr>
<td>Corrosion protection (mild)</td>
<td>0.25</td>
</tr>
<tr>
<td>Severe corrosion conditions</td>
<td>2.5</td>
</tr>
<tr>
<td>Severe wear electrical contacts</td>
<td>1 to 2.5</td>
</tr>
<tr>
<td>Special applications</td>
<td>to 6.25</td>
</tr>
</tbody>
</table>
Standard Specification for Coatings of Cadmium-Tin Mechanically Deposited

This standard is issued under the fixed designation B 635; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers the requirements for a coating that is a mixture of cadmium and tin mechanically deposited on metal products. The coating is provided in various thicknesses up to and including 12 µm.

1.2 Mechanical deposition greatly reduces the risk of hydrogen embrittlement and is suitable for coating bores and recesses in many parts that cannot be conveniently plated electrolytically. (See Appendix X1.)

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazards statements, see Section 7.

2. Referenced Documents

2.1 ASTM Standards:
B 117 Practice for Operating Salt Spray (Fog) Apparatus
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 201 Practice for Testing Chromate Coatings on Zinc and Cadmium Surfaces
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 322 Practice for Cleaning Metals Prior to Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
B 571 Test Methods for Adhesion of Metallic Coatings
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings

B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
E 87 Methods for Chemical Analysis of Lead, Tin, Antimony, and Their Alloys (Photometric Methods)
E 396 Test Methods for Chemical Analysis of Cadmium
F 1470 Guide for Fastener Sampling for Specified Mechanical Properties and Performance Inspection
2.2 U.S. Federal Standard:
FED-STD-141 Paint, Varnish, Lacquer, and Related Materials; Methods of Inspection, Sampling and Testing
2.3 U.S. Military Standard:
MIL-L-7808J Lubricating Oil, Aircraft Turbine Engine, Synthetic Base

3. Classification

3.1 Classes—Cadmium-tin coatings are classified on the basis of thickness, as follows:

<table>
<thead>
<tr>
<th>Class</th>
<th>Minimum Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>12</td>
<td>12</td>
</tr>
<tr>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>5</td>
<td>5</td>
</tr>
</tbody>
</table>

3.2 Types—Cadmium-tin coatings are identified by types on the basis of supplementary treatment required, as follows:

3.2.1 Type I—As coated, without supplementary chromate treatment (see X1.1).

3.2.2 Type II—With supplementary chromate treatment (see X1.2).

4. Ordering Information

4.1 To make the application of this specification complete, the purchaser needs to supply the following information to the supplier in the purchase order or other governing document:

4.1.1 Class, including a maximum thickness, if appropriate, type, and need for supplemental lubricant (3.1, 3.2, and 5.2.4.2).

4.1.2 Nature of substrate, for example, high-strength steel, need for stress-relief, and cleaning precautions to be followed (5.2.2).

4.1.3 Significant surfaces (5.3).

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1 This specification is under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.08.04 on Light Metals.

2 Annual Book of ASTM Standards, Vol 03.02.

3 Annual Book of ASTM Standards, Vol 02.05.

4 Discontinued—See 1984 Annual Book of ASTM Standards, Vol 03.05.

5 Annual Book of ASTM Standards, Vol 03.05.

6 Annual Book of ASTM Standards, Vol 01.08.

7 Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.
4.1.4 Requirements and methods of testing for one or more of the following requirements: need for and type of test specimens (8.1), composition (8.2), thickness (8.4), adhesion (8.6), and absence of hydrogen embrittlement and the waiting period prior to testing and testing loads (8.8) and lubricating resistance (8.2).

4.1.5 Sampling plan for each inspection criterion and responsibility for inspection, if necessary (Section 6 and Supplementary Requirement S1).

4.1.6 Requirements for certified report of test results (Section 10).

5. Requirements

5.1 Nature of Finish—The coating shall be 45 to 75 mass % cadmium, the remainder tin.

5.2 Process:

5.2.1 Stress Relief Treatment—All steel parts that have ultimate tensile strength of 1000 MPa and above and that contain tensile stresses caused by machining, grinding, straightening, or cold forming operation shall be given a stress relief heat treatment prior to cleaning and metal deposition. The temperature and time at temperature shall be 190 ± 15°C for a minimum of 3 h so that maximum stress relief is obtained without reducing the hardness below the specified minimum.

5.2.2 High-strength steels that have heavy oxide or scale shall be cleaned before application of the coating in accordance with Practice B 242. In general, non-electrolytic alkaline, anodic-alkaline, and some inhibited acid cleaners are preferred to avoid the risk of producing hydrogen embrittlement from the cleaning procedure.

5.2.3 For preparation of low-carbon steels, see Practice B 183. For cleaning, useful guidelines are also given in Practice B 322.

5.2.4 Supplementary Treatments:

5.2.4.1 Chromate treatment for Type II shall be done in a solution containing hexavalent chromium. This solution shall produce a bright or semi-bright continuous, smooth, protective film. This film may have a slight yellowish or iridescent color. The absence of color shall not be considered evidence of the absence of a Type II film or as a basis for rejection of the parts. Only post treatments that contain salts that yield films containing hexavalent chromium are permitted as treatments for producing Type II coatings.

5.2.4.2 Waxes, lacquers, or other organic coatings may be used to improve lubricity, and the need for them should be supplied in the purchase order or other governing document (4.1.1). Such supplemental lubrication treatments shall not be used to ensure conformance to the salt spray corrosion resistant requirements or to enhance the test results of the lead acetate spot test (8.5.2).

5.3 Thickness:

5.3.1 The thickness of the coating everywhere on the significant surfaces shall be at least that of the specified class as defined in 3.1.

5.3.2 Significant surfaces are defined as those normally visible (directly or by reflection) that are essential to the appearance or serviceability of the article when assembled in normal position; or which can be the source of corrosion products that deface visible surfaces on the assembled article. When necessary, the significant surfaces shall be indicated on the drawing of the article, or by the provision of suitably marked samples.

Note: 1—The thickness of mechanically-deposited coatings varies from point-to-point on the surface of a product, characteristically tending to be thicker on flat surfaces and thinner at exposed edges, sharp projections, shielded or recessed areas, interior corners and holes, with such thinner areas often being exempted from thickness requirement.

5.3.3 When significant surfaces are involved on which the specified thickness of deposit cannot readily be controlled, the purchaser and manufacturer should recognize the necessity for either thicker or thinner deposits. For example, to reduce buildup in thread roots, holes, deep recesses, bases of angles, and similar areas, the deposit thickness on the more accessible surfaces will have to be reduced proportionately.

Note 2—The coating thickness requirement of this specification is a minimum requirement; that is, the coating thickness is required to equal or exceed the specified thickness everywhere on the significant surfaces. Variation in the coating thickness from point to point on a coated article is an inherent characteristic of mechanical deposition processes. Therefore, the coating thickness will have to exceed the specified value at some points on the significant surfaces to ensure that the thickness equals or exceeds the specified value at all points. Thus, in most cases, the average coating thickness on an article will be greater than the specified value; how much greater is largely determined by the shape of the article and the characteristics of the deposition process. In addition, the average coating thickness on articles will vary from article to article within a production lot. Therefore, if all of the articles in a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the average necessary to ensure that a single article meets the requirement.

5.4 Adhesion—The cadmium-tin coating shall be sufficiently adherent to the basis metal to pass the tests specified in 8.6.

5.5 Corrosion Resistance:

5.5.1 The presence of corrosion products visible to the unaided eye at normal reading distance at the end of the specified test period as stated in Table 1 shall constitute failure, except that corrosion products at the edges of specimens shall not constitute failure. Slight “wisps” of white corrosion, as opposed to obvious accumulations, shall be acceptable.

Note 3—The hours given in Table 1 are the minimums required to guarantee satisfactory performance. Longer periods before the appearance of white corrosion products and rust are possible, but salt spray resistance does not vary in exact proportion with increased plating thickness. The hours given for Type II reflect the added protection of chromate treatments.

<table>
<thead>
<tr>
<th>Type</th>
<th>White Corrosion</th>
<th>Rust</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>not applicable</td>
<td>144</td>
</tr>
<tr>
<td>II</td>
<td>96</td>
<td>168</td>
</tr>
</tbody>
</table>

A Corrosion products are those visible to the unaided eye at normal reading distances after gentle washing to remove salt deposits.
shall not be cause for rejection.

5.5.2 There are no requirements for corrosion of base metals other than steels.

Note 4—Mechanical deposition is exclusively a barrel-finishing process. It is recognized that mechanical deposition on parts may therefore produce surfaces which have a different characteristic from those on parts which are finished exclusively by racking. Similarly, corrosion testing of actual parts may produce different results from those on test panels. Salt spray requirements that are appropriate to indicate the technical quality with which a process is carried out may be impractical for acceptance of actual parts. In such cases the purchaser should indicate his requirements on the purchase order (see 4.1.4).

Note 5—In many instances, there is no direct relation between the results of an accelerated corrosion test and the resistance to corrosion in other media, because several factors that influence the progress of corrosion, such as the formation of protective films, vary greatly with the conditions encountered. The results obtained in the test should not, therefore, be regarded as a direct guide to the corrosion resistance of the tested materials in all environments where these materials may be used. Also, performance of different materials in the test cannot always be taken as a direct guide to the relative corrosion resistance of these materials in service.

5.6 Absence of Hydrogen Embrittlement—Steel springs and other high-strength steel parts subject to flexure shall be held for a minimum of 48 h at room temperature after coating, before being loaded, flexed, or used. Such parts shall be free from hydrogen embrittlement. When specified in the purchase order, freedom from embrittlement shall be determined by the test specified herein (see 4.1.4 and 8.8).

5.7 Workmanship—The coating shall be uniform in appearance and substantially free of blisters, pits, nodules, flaking and other defects that can adversely affect the function of the coating. The coating shall cover all surfaces as stated in 5.3, including thread roots, thread peaks, corners, recesses, and edges. The coating shall not be stained or discolored through to an extent that would adversely affect appearance as a functional requirement. However, superficial staining that results from rinsing or drying, and variations in color or luster shall not be cause for rejection.

Note 6—The nature of the mechanical plating process is such that coatings characteristically will not be as smooth or as bright as some electroplated coatings.

5.8 Surface Defects—Defects and variations in appearance in the coating that arise from surface conditions of the substrate (scratches, pores, roll marks, inclusions, etc.) and that persist in the finish despite the observance of good metal finishing practices shall not be cause for rejection.

Note 7—Applied finishes generally perform better in service when the substrate over which they are applied is smooth and free of torn metal, inclusions, pores, and other defects. It is recommended that the specifications covering the unfinished product provide limits for these defects. A metal finisher can often remove defects through special treatments, such as grinding, polishing, abrasive blasting, chemical treatments, and electroplating. However, these are not normal in the treatment steps preceding the application of the finish. When desired, they must be specified on the purchase order (see 4.1.2).

6. Sampling

6.1 The purchaser and producer are urged to employ statistical process control in the coating process. Properly performed, statistical process control will assure coated products of satisfactory quality and will reduce the amount of acceptance inspection. The sampling plan used for the inspection of the quality coated article shall be agreed upon between the purchaser and producer.

6.1.1 When a collection of coated articles (inspection lot, see 6.2) is examined for compliance with the requirements placed on the articles, a relatively small number of the articles (sample) is selected at random and is inspected. The inspection lot is then classified as complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria for compliance are determined by the application of statistics. The procedure is known as sampling inspection. Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for sampling inspection of coatings.

6.1.2 Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one when they are destructive. Test Method B 602 provides a default plan if one is not specified.

6.1.3 Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. Guide B 697 provides a default plan if one is not specified.

6.1.4 Test Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numeric value and certain statistical requirements must be met. Test Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. Test Method B 762 provides a default plan if one is not specified.

6.1.5 Guide F 1470 can be used for fasteners such as internally threaded, externally threaded and nonthreaded fasteners and washers. This guide provides for two plans; one designated the “detection process” and one designated the “prevention process.” The purchaser and producer shall agree on the plan to be used.

6.2 An inspection lot shall be defined as a collection of coated articles that are the same kind, that have been produced to the same specification, that have been coated by a single supplier at one time or approximately the same time, under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

7. Hazards

7.1 Warning—Cadmium is toxic and must not be used in a coating for articles that can come into contact with food or beverages, or for dental or other equipment that can be inserted into the mouth. Consult appropriate agencies for regulations in this connection.

7.2 Precaution—Because of the toxicity of cadmium vapors and cadmium oxide fumes, cadmium-tin coated articles must not be used at temperatures of 165°F and above. They must not be welded, spot-welded, soldered, or otherwise strongly heated, without adequate ventilation that will efficiently remove all toxic fumes. (See X4.2.)

8. Test Methods

8.1 Test Specimens:

8.1.1 Test specimens may be used to represent the coated articles in a test if the articles are of a size, shape, or material
that is not suitable for the test, or if it is preferred not to submit articles to a destructive test because, for example, the articles are expensive or few. The permission or the requirement to use test specimens, their number, the material from which they shall be made, and their shape and size shall be stated in the purchase order or other governing document.

8.1.2 The test specimen shall duplicate those characteristics of the article that influence the property being tested, and it shall be processed with the article through those process steps that influence the property.

8.1.2.1 The test specimen used to represent an article in an adhesion, corrosion resistance, or appearance test shall be made of the same material, shall be in the same metallurgical condition, and shall have the same surface condition as the article it represents. It shall be placed in the production lot of and be processed along with the article it represents.

8.1.2.2 A test specimen used to represent an article in a coating thickness test shall be introduced into the process at the point where the coating or coatings are applied and it shall be carried through all steps that have a bearing on the coating thickness.

8.1.2.3 When a test specimen is used to represent a coated article in a thickness test, the specimen will not necessarily have the same thickness and thickness distribution as the article unless the specimen and the article are of the same general size and shape. Therefore, before coated articles may be accepted on the basis of a thickness test performed on test specimens, the relationship between the thickness on the specimen and the thickness on the part shall be established. The criterion of acceptance shall be that thickness on the specimen that corresponds to the required thickness on the article.

8.2 Composition—Chemical composition of the cadmium-tin coating shall be determined when required on the purchase order by procedures given in Methods E 87 or Test Methods E 396, or by other methods specified on the purchase order (see 4.1.4) as long as they can determine composition to within the desired limits.

8.3 Workmanship—The quality of workmanship shall be determined by the unaided eye at normal reading distance.

8.4 Thickness:

8.4.1 The thickness of the coating shall be determined by the microscopical method (Test Method B 487), the magnetic method (Test Method B 499), or the beta backscatter method (Test Method B 567), as applicable. Other methods may be used if it can be demonstrated that the uncertainty of the measurements with these methods is less than 10%.

8.4.1.1 The composition of the deposit shall be considered when selecting standards for calibrating beta backscatter instruments.

8.4.2 The thickness of the coating shall be measured at the location or locations on the significant surfaces of the product where the coating would be expected to be the thinnest or at such locations as specified on the purchase order (see 4.1.3 and 5.3.2).

8.4.3 Thickness measurements of Type II deposits shall be made after application of the supplementary treatment.

Note 8—The process by which Type II coatings are produced dissolves a small amount of the cadmium-tin. For this reason the thickness requirement to be checked refers to the thickness of the deposit after the application of the Type II coating.

Note 9—The thickest chromate film (olive drab) is approximately 1.5 µm thick. If this significantly affects the accuracy of the thickness measuring method used (for example, Test Method B 567) the Type II coating should be removed from the test area. Removal should be done by using a very mild abrasive (a paste of levigated alumina or magnesium oxide rubbed on gently with the finger).

8.5 Presence of Chromate:

8.5.1 Chromate conversion coatings of cadmium-tin both have an essentially silvery-white appearance. In the absence of a definitive method for distinguishing a Type II coating from a lacquer or other non-chromate film, the salt spray test shall be used to verify the presence of a Type II coating, in terms of hours to white corrosion product.

8.5.2 Frequently, the presence of a chromate coating on a product is tested for by a diphenyl carbazole test, and the quality of the coating is assessed by a lead acetate test. The reliability of these tests has been questioned because the diphenyl carbazole test merely tests for the presence of chromate ions, which may or may not be in the form of a coating, and because the lead acetate test will not distinguish between chromate and other kinds of coatings. Therefore, these tests have value as controls of an in-house process but are of less value in the evaluation of a purchased product whose processing is neither under the control nor the cognizance of the purchaser. The lead acetate test is described in Practice B 201. The diphenyl carbazole test is described in Annex A1 of this specification.

8.6 Adhesion—Adhesion of the cadmium-tin deposit to the basis metal shall be tested in a manner that is consistent with the service requirements of the coated article. The ability to separate the coating from the substrate by peeling, as distinct from flaking caused by rupture of the deposit or of the base metal, shall be evidence of failure. One of the following methods for determining adhesion shall be used:

8.6.1 The part shall be plastically deformed, if possible, to rupture as specified on the purchase order (see 4.1.4).

8.6.2 The surface of the coated article shall be scraped or sheared with a sharp edge, knife, or razor blade through the coating down to the basis metal and examined under 4× magnification.

Note 10—There is no single satisfactory test for evaluating the adhesion of mechanically deposited coatings. Those given in 8.6 are widely used; however, other tests may prove more applicable in specific cases. Various qualitative methods are discussed in Test Methods B 571. A review of methods of measuring adhesion is given in Polleys, R. W., “The Adhesion Testing of Electroplated Coatings,” Proceedings, American Electroplaters’ Society, APAAEA, Vol 50, 1963, p. 54. Application of pressure-sensitive tape to the coating is a method which may be misleading. Metal particles adhering to the tape may only reflect rinsing technique. Distinctions must be made between superficial surface effects which will not adversely affect other requirements, and internal coating consolidation (cohesion), or adhesion to the basis metal or undercoating.

8.7 Salt Spray Corrosion Resistance:

8.7.1 The 5% neutral salt spray (fog) test as defined in Practice B 117 shall be used.

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8.7.2 If samples with Type II coatings are to be examined both for white corrosion products and for rust, separate sets of samples shall be used to determine the 96-h end point for white corrosion and the 168-h end point for rust; this is to permit exposure for the longer period without having to wash specimens for examination of white corrosion in accordance with Practice B 117.

8.7.3 Parts with Type II supplementary chromate film shall be aged at room temperature for 24 h before subjection to the salt spray test.

8.7.4 Parts with coatings of wax, etc. shall not be used as samples for corrosion testing for conformance to the requirements of 5.5.

8.8 Absence of Hydrogen Embrittlement:

8.8.1 Coated parts to be tested for the absence of embrittlement from cleaning shall be tested for brittle failure in accordance with a suitable method to be specified on the purchase order (see 4.1.4). The description of the method shall include the means of applying a load to the part, the stress or load level to be applied, the duration of the test, and the waiting time that must elapse between deposition of the cadmium-tin and testing or use of the part and the criterion of failure.

8.8.2 Parts that must conform to U.S. Government requirements shall be subjected to loading conditions described above for at least 200 h.

Note 11—It is recommended that tests for embrittlement involve subjecting parts to the specified operating conditions for at least 100 h except as noted in 8.8.2. The stress level induced by the test and the waiting period prior to test depend upon many factors, such as shape of the part, carbon content of the steel, hardness of the part, and stress level in use. Parts with a tensile strength of over 1000 MPa for example, may require a 48-h waiting period; parts with lower tensile strength may require less than a 24-h waiting period. High-carbon steel parts or those cold-worked or heat-treated to tensile strengths of 1450 MPa minimum, where these parts will be subjected to a sustained load in use, may require testing at loads specified by the purchaser.

9. Rejection and Rehearing

9.1 Materials that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of a test, the producer or supplier may make a claim for rehearing. Finishes that show imperfections during subsequent manufacturing operations may be rejected.

10. Certification

10.1 The purchaser may require in the purchase order or contract that the producer or supplier give to the purchaser certification that the finish was produced and tested in accordance with this specification and found to meet the requirements. The purchaser may similarly require that a report of the test results be furnished (see 4.1.6).

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirements shall apply only when specified by the purchaser as part of the purchaser’s order or contract, and for all agencies of the United States Government.

S1. Responsibility for Inspection

S1.1 The producer or supplier shall be responsible for the performance of all inspection and test requirements specified herein (see 4.1.5). Except as otherwise specified in the contract or order, the producer or supplier may use his own or any other suitable facilities for the performance of the inspection and test requirements specified herein, unless disapproved by the purchaser. The purchaser shall have the right to perform any of the inspections and tests set forth in this specification where such inspections are deemed necessary to assure that material conforms to prescribed requirements.

S2. Lubricating Oil Resistance

S2.1 Procedure—The coating shall withstand immersion in diester lubricating oil conforming to MIL-L-7808 at a temperature of 121 ± 3°C for 24 h. Upon cooling to room temperature, parts or specimens shall be examined and compared with unexposed parts or specimens. The part or specimen shall then be tested in accordance with FED-STD 141, Method 6223 using a mandrel whose diameter is 14 times the basis metal thickness. The time of test shall be 2 s.

S2.2 Evaluation—After holding at 121°C and cooling to room temperature, the test pieces shall show no wrinkling, blistering, pitting, or other surface defects. Discoloration shall not be cause for rejection. After performance of the deformation test (Method 6223), if the edge of the ruptured coating can be peeled back, or if separation of the coating from the basis metal can be seen at the point of rupture when examined at 4X magnification, adhesion is not satisfactory due to poor resistance to lubricating oil.

S2.3 Separate Specimens—If separate specimens for lubricating oil resistance tests are required, they shall be ferrous alloy panels not less than 152 mm in length and 76 mm in width and they shall be approximately 1 mm thick. The ferrous alloy shall be of such composition and hardness that the specimen will withstand deformation without cracking or fracture.
A1. DIPEHYL CARBAZIDE TEST

A1.1 Hexavalent Chromium Test

A1.1.1 Dissolve 0.5 g of 1,5-diphenyl carbazide in a solvent mixture consisting of 20 mL of acetone and 20 mL of 95% ethanol by using, if necessary, a warm (50°C) water bath. Add to this a dilute phosphoric acid solution consisting of 20 mL of water and 20 mL of phosphoric acid.

A1.1.2 Place a drop on the surface to be tested. A pink to purple color will indicate the presence of chromate anions.

NOTE A1.1—The test solution is heat- and light-sensitive and must be discarded when it discolors.

A1.2 Hexavalent and Trivalent Chromium Test

A1.2.1 An alternative test solution for either tri- or hexavalent chromium is the following:

Formula A

- Distilled or deionized water, mL 40
- Sodium hypochlorite, 5%, mL 10
- Glacial acetic acid, mL 60
- Diphenyl carbazide, g 1
- Hydrochloric acid, concentrated, mL 15
- Hydrogen peroxide, 30%, mL 5

A1.2.2 Add each component to the distilled water in the order given above. The solution may turn pink in the beginning but the color will fade later. This solution may be stable for 2 weeks.

A1.2.3 Place a small piece of filter paper (3 mm²) on the part to be tested, with the part at room temperature. Put two drops of Formula A solution upon the filter paper and wait for 1 to 3 min for color to develop. It will change to pink if chemical chromium film exists.

A1.2.4 The test part may be warmed to about 65°C by a hot-air blower. Then drop Formula A solution immediately on the part to observe any change of color.

A1.3 Hexavalent or Trivalent Chromium Test

A1.3.1 Solutions to distinguish between tri- and hexavalent chromium films are the following:

Formula B

<table>
<thead>
<tr>
<th>Part 1</th>
<th>Glacial acetic acid, mL</th>
<th>60</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Diphenyl carbazide, mg</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>Hydrochloric acid, concentrated, mL</td>
<td>15</td>
</tr>
<tr>
<td></td>
<td>Distilled or deionized water, mL</td>
<td>40</td>
</tr>
<tr>
<td>Part 2</td>
<td>Sodium hypochlorite, 5%, mL</td>
<td>10</td>
</tr>
<tr>
<td></td>
<td>Hydrogen peroxide, 30%, mL</td>
<td>5</td>
</tr>
<tr>
<td></td>
<td>Distilled water, mL</td>
<td>40</td>
</tr>
</tbody>
</table>

A1.3.2 When Part 1 is applied to the part with hexavalent chromium in accordance with the procedure mentioned in A1.1.2, a change of color will be seen. On the other hand, Part 1 will give no pink color when applied to a trivalent chromium film. To confirm the presence of a trivalent film, Part 1 solution is combined with one or two drops of Part 2 solution. When a trivalent film is present, a pink color will show.

APPENDIXES

X1. CHARACTERISTICS

X1.1 Cadmium-tin coatings excel in minimizing dissimilar metals corrosion between coated articles and certain metals with which they are in contact. Such metals, particularly the aluminum alloys, can produce a high electrical potential with the coated part. Cadmium-tin deposits are widely used to minimize corrosion in automotive lamp contacts. These deposits, when treated with Type II coatings, give improved corrosion resistance over Type I, yet maintain the silvery metallic appearance of the article that is desired by many consumers.

X1.2 In addition, in some environments and in salt spray tests, corrosion protection equivalent to cadmium coatings of a given thickness may be obtained with less thickness by use of a cadmium-tin coating. The use of less cadmium by the substitution of cadmium-tin reduces pollution problems associated with cadmium.
X2. PROCESS

X2.1 Mechanical deposition of cadmium-tin coatings should consist in general, of all of the steps listed below, and in the sequence as shown:

X2.2 Preparation of the surface of the parts to be coated, by chemical (generally acidic) procedure, to an extent that permits uniformly satisfactory results from subsequent steps.

X2.3 Deposition of a thin metal coating, where applicable, by immersion in appropriate chemical solutions, without the use of electric current for ferrous basis metals this thin coating is generally copper. There are no thickness requirements for this coating.

X2.4 Tumbling of the parts that have been treated in accordance with X2.2 and X2.3 in a container with the following:

X2.4.1 The metals to be deposited, in powder form, and mixed in the desired proportions.
X2.4.2 Impact media, which may be glass or other substances that are essentially inert to the chemicals of the deposition process. The function of this media is to aid in providing mechanical forces to drive the metal powders onto the substrate parts.
X2.4.3 A “promoter” or “accelerator” that aids in the uniform deposition of the metal powders.
X2.4.4 A liquid medium, generally water.

X2.5 Separation of the parts from the solid and liquid media.

X2.6 Rinsing.

X2.7 Drying.

X3. SPECIFIC TYPES

X3.1 Type I (Plain Cadmium-Tin) is useful for lowest cost protection where early formation of white corrosion products is not detrimental. It is also used for higher temperature applications up to approximately 120°C, where the effectiveness of chromates is greatly reduced.

X3.2 Type II (Unleached Chromates)—The unleached chromates that on pure cadmium or pure zinc are colored (yellow iridescent, olive drab, bronze, etc.) are used to delay the appearance of white or red corrosion products on the cadmium-tin plated article. Mechanically deposited cadmium-tin coatings typically do not show the characteristic yellow color of pure zinc or pure cadmium treated with a colored chromate. Leached coatings are therefore unnecessary with this plating.

X4. HYDROGEN EMBRITTLEMENT

X4.1 A major advantage of mechanical plating is that it does not produce hydrogen embrittlement in hardened steel during the coating process. However, pronounced embrittlement can be produced in certain cleaning processes. The mild degree of embrittlement that might result from following proper procedures with cleaning methods permitted in this specification normally is self-relieving within a day’s time at room temperature.

X4.2 Because the mechanical cadmium-tin coatings of the compositions covered by this specification have their minimum melting point at 177°C, they may be heated to 165°C without danger of cadmium vapor, when this is desired for additional embrittlement relief. (See Section 7.)
Standard Test Method for Measurement of Internal Stress of Plated Metallic Coatings with the Spiral Contractometer

1. Scope

1.1 This test method covers the use of the spiral contractometer for measuring the internal stress of metallic coatings as produced from plating solutions on a helical cathode. The test method can be used with electrolytic and autocatalytic deposits.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Terminology

2.1 Definitions:

2.1.1 compressive stress (−)—stress that tends to cause a deposit to expand.

2.1.2 internal stress—the net stress that remains in a deposit when it is free from external forces. The internal stress tends to compress or stretch the deposits.

2.1.3 tensile stress (+)—stress that tends to cause a deposit to contract.

3. Summary of Test Method

3.1 The test method of measuring stress with the spiral contractometer is based on plating on the outside of a helix. The helix is formed by winding a strip around a cylinder, followed by annealing. In operation, one end of the helix is fixed and the other is allowed to move as stresses develop. The free end is attached to an indicating needle through gears that magnify the movement of the helix. As the helix is plated, the stress in the deposit causes the helix to wind more tightly or to unwind, depending on whether the stress is compressive (−) or tensile (+). From the amount of needle deflection and other data, the internal stress is calculated.

3.2 With instrument modifications, the movement of the helix can be measured electronically instead of mechanically as described in 3.1.

4. Significance and Use

4.1 The spiral contractometer, properly used, will give reproducible results (see 8.5) over a wide range of stress values. Internal stress limits with this method can be specified for use by both the purchaser and the producer of plated or electroformed parts.

4.2 Plating with large tensile stresses will reduce the fatigue strength of a product made from high-strength steel. Maximum stress limits can be specified to minimize this. Other properties affected by stress include corrosion resistance, dimensional stability, cracking, and peeling.

4.3 In control of electroforming solutions, the effects of stress are more widely recognized, and the control of stress is usually necessary to obtain a usable electroform. Internal stress limits can be determined and specified for production control.

4.4 Internal stress values obtained by the spiral contractometer do not necessarily reflect the internal stress values found on a part plated in the same solution. Internal stress varies with many factors, such as coating thickness, preparation of substrate, current density, and temperature, as well as the solution composition. Closer correlation is achieved when the test conditions match those used to coat the part.

5. Apparatus

5.1 The spiral contractometer is described by A. Brenner and S. Senderoff.²

NOTE 1—Spiral contractometers are available from many of the suppliers of nickel sulfamate.

5.2 Helices shall be stopped-off on the inside to prevent plating. Helices are available with or without a permanent inert coating on the inside (see Appendix X1).

5.3 The clamps holding the helix to the contractometer shall be coated with an inert nonconductive coating to prevent their plating and acting as thieves.

5.4 For testing electroplating solutions, anodes are placed equidistant from the helix and symmetrically positioned to produce even plate distribution. A minimum of four anodes is required. A concentric anode arrangement is preferred.

5.5 Laboratory tests on electroplating solutions shall utilize at least 3.7 L of solution. A 4-L beaker with an annular anode

arrangement is convenient. Use of this volume or larger will minimize solution changes due to electrolysis during the test.

5.6 Laboratory tests on autocatalytic plating solutions are done in a 1-L, tall-form beaker. Obviously, no anodes are used.

6. Factors Affecting Accuracy

6.1 Variations in the preparation of the helix may cause substantial variations in results.

6.1.1 Stop-off material shall be applied properly to the interior of the helix. The stop-off material shall be thin and flexible to permit the helix to move freely during the test. A coating weight of less than 50 mg/dm$^2$ is satisfactory.

NOTE 2—The inside must be stopped-off with some inert, flexible coating. One acceptable stop-off material is “Microstop.” One part of “Microstop” is diluted with two parts of acetone before use. Any nickel deposited on the inside of the helix will exhibit an opposing effect.

6.1.2 Helices that have been permanently coated on the inside with TFE-fluorocarbon may give variable results when testing near-zero stresses.

6.1.3 Cleaning variations and surface preparation of the helix before the test can produce varying results. For example, electrocleaning of the helix shall always be cathodic and controlled with respect to current, time, and temperature. Anodic cleaning at this stage can give wide variations. Abrasive cleaning of the helix and the use of etchants shall be avoided.

6.1.4 Very thin deposits of less than about 3 µm are influenced more by the surface conditions and preparation of the helix than are thicker deposits.

6.2 Internal stress varies with current density used in electroplating. The variation is not predictable and depends on the metal being deposited, impurities or additives, and the current density range under consideration. It is important that the current be measured and controlled closely throughout the stress test. Variations in currents shall be held to less than 2 %.

6.3 Because the temperature of the plating solution may affect the internal stress, it shall be maintained within 2°C during the test. The initial rest point of the indicator and the final rest point are both taken at the operating temperature of the plating solution to eliminate thermal stresses.

6.4 The solution composition shall not vary during the test. Usually, if the repeatability tests in 8.5 are met, the solution can be assumed to be unchanged during the test runs. Conversely, when the repeatability tests are not met, the plating solution shall be analyzed to determine if any changes in solution composition have occurred during the test.

6.4.1 Tests run on electroplating solutions using insoluble or inefficient anodes could result in significant solution changes during the test.

6.4.2 When testing autocatalytic plating solutions, the constituents of the plating solution may be significantly depleted during the test, unless replenished.

6.5 A relationship between the surface area to be plated and the volume of autocatalytic plating solutions exists that may affect the character of the deposit. In testing autocatalytic plating solutions, the ratio of plated surface area to the volume of solution that is normally used in the plating tank shall be maintained. When using proprietary solutions, the supplier’s recommendation shall be followed.

7. Calibration

7.1 Calibrate the instrument as directed in the manufacturer’s instructions.

7.2 The frequency of calibration will vary with use and extent of attack on the helices from the chemical stripping. When visible attack is noted, discard the helix.

7.3 The calibration procedure consists essentially of determining the force required per degree of dial deflection. A known mass is suspended over a small pulley on a lever arm with the helix mounted in place. The degree of deflection is read from the dial. The data required for the calibration calculations as expressed in metric units are as follows:

\[ w = \text{mass used in calibrating, kg,} \]
\[ a = \text{length of lever arm, m,} \]
\[ p = \text{pitch of helix, m,} \]
\[ t = \text{thickness of the strip used to make the helix, m,} \]
\[ \text{deg def} = \text{degree deflection; difference in dial readings} \]
\[ g = 9.8 \text{ m/s}^2(\text{acceleration of free fall}), \]
\[ Z = \text{calibration constant of the helix} \left( \frac{\text{MPa}}{m \text{deg def}} \right) \]
\[ Z = \frac{2(w)(a)(g)}{p(t)\text{deg def}} \times 10^{-6} \]

8. Procedure

8.1 The procedure will vary with the solution being tested. Follow the instructions given by the supplier carefully. Variations in the procedure can produce variations in results. Give appropriate attention to the factors in Section 6. A detailed procedure for nickel plating solutions appears in Appendix X1.

8.2 Position the spiral contractometer in electroplating solutions so that it is equidistant from the anodes. Position the anodes on at least four sides when they are used in a production tank or use a concentric anode arrangement. Do not place the spiral contractometer between the tank anodes and the work being plated in a production tank. A separate ammeter and current control are required. If the test is run on a sample of the solution, use a 4-L beaker.

8.3 When testing autocatalytic plating solutions, maintain the proper surface area-to-solution volume ratio (see 6.5).

8.4 The conditions of the test are usually chosen to match those of the production parts as closely as practicable.

8.4.1 The thickness of the deposit plated on the helix may not be required to be the same as the thickness plated on production parts. This is especially true when production part thickness requirements are greater than about 15 µm.

8.4.2 The current density used for the spiral contractometer is sometimes lower than that of production parts. Electroplating at the lower current density will often indicate the cumulative effects of solution impurities before these have much effect on the work being plated at the higher currents. This information can be used as a measure of the solution

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3 Manufactured by Michigan Chrome and Chemical Co., 8615 Grinell Ave., Detroit, MI 48174.
purity and will allow planning for corrective or purification procedures.

8.5 Repeatability:

8.5.1 Consider the procedure repeatable if the results of three consecutive tests on the same solution sample produce internal stress values that do not vary more than ±10 % from the mean of the three values.

8.5.2 For near zero stress values, −10 to +10 MPa, it is more difficult to detect small changes with the contractometer and an acceptable range for repeatability is ±5 MPa.

8.6 Solution adjustments may be made during the test, especially when testing autocatalytic solutions and electroplating solutions using insoluble anodes.

8.7 Clean the instrument after each use and before storage. This is to prevent solution that may creep up the shaft from crystallizing and binding or corroding the instrument.

9. Stress Calculations

9.1 Data Required:

\[ Z = \text{calibration constant for the helix, MPa/(m deg def)}, \]
\[ D = \text{change in dial reading during the test, deg, and} \]
\[ \tau = \text{thickness of the deposit on the helix, m}. \]

\( \tau \) can be calculated from the additional data:

\[ A = \text{plated area, m}^2, \]
\[ W = \text{mass of deposit, kg, and} \]
\[ d = \text{density of deposit, kg/m}^3. \]

Then \( \tau = W/(d)A \)

Internal stress, \( S \), in MPa = \[ Z \times D \]
\[ \tau \]

Note 3—This value is an average stress and is uncorrected for the effects of the differences in Young’s modulus between the helix and the deposit. It also assumes a relatively thin deposit on the helix. If a corrected or truer stress, \( S_T \), is desired:

\[ S_T = S \left[ 1 + \left( \frac{E_0 \times \tau}{E \times \tau} \right)^3 \right] \]

where:

\( E_0 = \text{Young’s modulus of the deposit, and} \)
\( E = \text{Young’s modulus of the helix}. \)

Normally, the correction made above is less than the experimental errors in the procedure and is infrequently used.

10. Report

10.1 When reporting internal stress values, it is necessary to include the following:

10.1.1 Metal deposited and the plating solution composition,
10.1.2 Instrument used (spiral contractometer),
10.1.3 Thickness of deposit on helix,
10.1.4 Current density (omit if autocatalytic deposits are being tested),
10.1.5 Temperature, and
10.1.6 pH (if it is a controllable variable).

10.2 The unit for expressing internal stress is the megapascal (MPa). One megapascal is one meganewton per square metre (MN/m²), approximately 145 psi.

11. Precision and Bias

11.1 Results are reproducible to about ±10 % except for the near-zero range of ±10 MPa. In the near-zero range, results are reproducible to about ±5 MPa.

12. Keywords

12.1 internal stress test; metallic deposit

APPENDIX

(Nonmandatory Information)

X1. PROCEDURE FOR STRESS DETERMINATION OF NICKEL ELECTRODEPOSITS

X1.1 Scope

X1.1.1 The following is a detailed procedure for the determination of internal stress using the spiral contractometer on nickel electroplating solutions. Some modifications can be made to adapt this procedure for use with other metal plating solutions.

X1.2 Preparation of Helix (use a stainless steel helix)

X1.2.1 Clean helix (as cathode) by electrocleaning at 4.0 to 5.0 A for 15 s in an alkaline steel cleaner at 50 ± 2°C.
X1.2.2 Rinse.
X1.2.3 Immerse in 50 %, v/v, hydrochloric acid for 3 to 5 s.
X1.2.4 Rinse.
X1.2.5 Nickel strike at 5.0 A for 1 min in a standard Wood’s nickel solution. The nickel strike is optional. If omitted, proceed to X1.2.9.

Note X1.1—The nickel strike solution contains 240 g/litre of nickel chloride hexahydrate and 12.5 % v/v of concentrated hydrochloric acid. It is operated at room temperature using nickel anodes.

X1.2.6 Rinse.
X1.2.7 Immerse in 50 %, v/v hydrochloric acid for 3 to 5 s.
X1.2.8 Rinse.
X1.2.9 Dry the helix with acetone and weigh to nearest 1 mg.
X1.2.10 Stop-off the inside of the helix by dipping in a test tube containing a stop-off lacquer solution (see Note 2). If the helix has a permanently bonded, inert coating, omit this step, the step described in X1.2.11, and the stripping of the stop-off.
X1.2.11 Remove the lacquer from the outside of the helix by wiping with acetone and then trichloroethylene. Do not touch the cleaned helix with bare fingers.
X1.2.12 Mount the helix with clamps. Stop-off the outside
of the bottom clamp or use a bottom clamp, bolt, and nut made of resistant plastic such as TFE-fluorocarbon. The top clamp need not be coated because it is not immersed.

**Note X1.2**—Since the clamp is insulated, the cathode efficiency can be calculated and even current distribution over the helix is obtained.

X1.2.13 Clean cathodically at 0.5 to 1.0 A for 5 s in the cleaner used in X1.2.1.

X1.2.14 Rinse.

X1.2.15 Immerse 50%, v/v, hydrochloric acid for 3 to 5 s.

X1.2.16 Rinse.

X1.2.17 Inspect the stop-off coating on the inside of the helix. If it is peeling, strip it with acetone and re-coat (from X1.2.10) the helix.

X1.2.18 Mount the helix on the instrument in a no-stress state. Adjust the pointer gear to near its center.

**Note X1.3**—If the instrument is used daily, calibrate the helix once a week. For calibration, see regular instructions.

**X1.3 Procedure**

X1.3.1 Immerse the assembled instrument into the plating solution so that the dial face is horizontal and the solution level is just below the top clamp. The helix is equidistant from the anode surface. Use at least four anodes surrounding the helix or a concentric anode.

**Note X1.4**—An annular basket is convenient for use in nickel solutions and can be used in either laboratory or production tank tests. The basket is constructed of titanium expanded metal or mesh filled with segmented nickel anodes. For nickel electroplating solutions containing less than about 15 g/litre of halide, sulfur depolarized nickel anode material is required.

X1.3.2 Allow a few seconds for the helix to reach the solution temperature and then record the initial dial reading. Temperature equilibrium is reached when the dial reading does not change after slightly tapping the instrument.

X1.3.3 Connect the electrical contacts and adjust the current to 2.0 A dc. Use a filtered rectifier or battery for the power source.

X1.3.4 Set the timer for 30 min.

**Note X1.5**—The thickness of the nickel plate should be 13 to 16 µm. This thickness minimizes the effect of the initial high stresses due to basis metal and basis metal preparation.

X1.3.5 Maintain the current within 0.05 A and the temperature within 2°C throughout the test. Tap the dial intermittently throughout the test to ensure that the instrument is not bound.

X1.3.6 Disconnect from the d-c power source after 30 min and record the final dial reading after tapping to ensure that the needle has reached a steady state.

X1.3.7 Remove the instrument and rinse it in hot water.

X1.3.8 Remove the helix from the instrument.

X1.3.9 Remove stop-off lacquer by immersion in acetone. Use succeeding amounts of fresh acetone until the wash solution is no longer colored.

X1.3.10 Allow the helix to dry and weigh it to the nearest 1 mg.
Standard Specification for Electrodeposited Engineering Chromium Coatings on Ferrous Substrates

This standard is issued under the fixed designation B 650; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This specification covers the requirements for electrodeposited chromium coatings applied to ferrous alloys for engineering applications.

1.2 Electrodeposited engineering chromium, which is sometimes called “functional” or “hard” chromium, is usually applied directly to the basis metal and is much thicker than decorative chromium. Engineering chromium is used for the following:

1.2.1 To increase wear and abrasion resistance,
1.2.2 To increase fretting resistance,
1.2.3 To reduce static and kinetic friction,
1.2.4 To reduce galling or seizing, or both, for various metal combinations,
1.2.5 To increase corrosion resistance, and
1.2.6 To build up undersize or worn parts.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 117 Practice for Operating Salt Spray (Fog) Apparatus
B 177 Guide for Engineering Chromium Electroplating
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Guide for Preparation of High-Carbon Steel for Electroplating
B 320 Practice for Preparation of Iron Castings for Electroplating
B 374 Terminology Relating to Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
B 507 Practice for Design of Articles to Be Electroplated on Racks
B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 762 Test Method of Variables Sampling of Metallic and Inorganic Coatings
B 849 Specification for Pre-Treatments of Iron or Steel for Reducing the Risk of Hydrogen Embrittlement
B 850 Guide for Post-Coating Treatments of Steel for Reducing Risk of Hydrogen Embrittlement
D 3951 Practice for Commercial Packaging
E 8 Test Methods for Tension Testing of Metallic Materials
F 1459 Test Method for Determination of the Susceptibility of Metallic Materials to Gaseous Hydrogen Embrittlement

2.2 Other Standard:
MIL-S-13165 Shot Peening of Metal Parts

3. Terminology

3.1 Definitions:
3.1.1 Significant surfaces—all surfaces upon which a deposit of controlled thickness is required.

3.1.1.1 Discussion—When a controlled deposit is required in holes, corners, recesses, and similar areas, special racking, auxiliary anodes or shielding, or both, will be necessary. With
the best practices there will be areas where a controlled deposit is impossible.

3.2 Definitions used in this specification are in accordance with Terminology B 374.

4. Classification

4.1 Electrodeposited chromium coatings in accordance with this specification are classified by the thickness of the coating as follows:

<table>
<thead>
<tr>
<th>Class No.</th>
<th>Chromium Thickness, µm</th>
<th>Typical Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.5 to 25</td>
<td>reduce friction; anti-galling, light wear resistance</td>
</tr>
<tr>
<td>2</td>
<td>&gt;25 as specified</td>
<td>buildup to dimension specified for salvage or as required for severe wear resistance</td>
</tr>
</tbody>
</table>

4.2 Unless otherwise specified by suitably marked drawings or samples, only those surfaces that can be touched with a 20-mm diameter ball shall be considered significant. In holes, corners, recesses, and other areas where a controlled deposit cannot be obtained under normal electroplating conditions, the thickness of the deposit may be that which results from control on the significant surfaces.

5. Ordering Information

5.1 The purchaser shall exercise the desired options of this standard. Ordering documents shall specify the following information:

5.1.1 Title, ASTM designation, and issue date of this specification.
5.1.2 Alloy and metallurgical condition of the product to be chromium plated.
5.1.3 Ultimate tensile strength of the material to be plated.
5.1.4 Heat treatment required for stress relief and whether it has been performed or is required.
5.1.5 The significant surfaces if different from the 20-mm ball rule (see 3.1.1).
5.1.6 Thickness of the deposit or class (see 4.1).
5.1.7 Control record requirements.
5.1.8 Preproduction test specimens, if required.
5.1.9 Sampling plan, if different from that specified in Test Method B 602 (see Section 8).
5.1.10 The number of test specimens for destructive testing (see 7.1).
5.1.11 Thickness, adhesion, porosity, and hydrogen embrittlement tests required (see Section 6).
5.1.12 Whether separate test specimens will be used (see 7.1 and 7.5).
5.1.13 Where required, any special requirements for parts that are subsequently ground to size.
5.1.14 Where required, the base metal finish in terms of center line average (CLA) or arithmetic average (AA), and
5.1.15 Where required, dimensional tolerances allowed for the specified coating thickness or class.
5.2 The manufacturer of the parts to be electroplated shall provide the electroplating facility with test specimens (see Section 7) to be electroplated for conformance tests as requested for preparation, control, inspection, and lot acceptance unless other arrangements have been made between the purchaser and the electroplating facility.

6. Coating Requirements

6.1 The appearance of the chromium coating on the significant surfaces of the product shall be smooth and free of visual defects such as blisters, pits, roughness, cracks, burned deposits, uncoated areas, or macrocracking of the deposit that is visible without magnification. The boundaries of electroplating that cover only a portion of the surface shall, after finishing as indicated on the drawing, be free of beads, nodules, jagged edges, or other irregularities that will interfere with the functioning of the plated part. Imperfections and variations that arise from surface conditions of the basis metal (scratches, pores, roll marks, inclusions, etc.) and that persist in the finish despite the observance of good metal finishing practices shall not be cause for rejection.

Note: 1—Applied finishes generally perform better in service when the substrate over which they are applied is smooth and free of torn metal, inclusions, pores, and other defects. It is recommended that the specifications covering the unfinished product provide limits for these defects. A metal finisher can often remove defects through special treatments such as grinding, polishing, abrasive blasting, chemical treatments, and electropolishing, which are not normal in the treatment steps preceding the application of the finish and will add to the cost. When they are desired, they are the subject of a special agreement between the purchaser and the seller.

6.2 In cases where design for maximum fatigue life is a consideration the parts should be shot peened (see MIL-S-13165C) or given an alternate mechanical treatment to compressively stress the surface.

6.3 Stress Relief Treatment (See headnote at the beginning of this specification): 6.3.1 All steel parts having an ultimate tensile strength of 1000 MPa (150 000 psi—approximately 32 HRC) or greater, that may contain residual stress caused by various fabrication operations such as machining, grinding, straightening, or cold forming, will require one of the stress relief heat treatments prescribed in Specification B 849 prior to electroplating. In all cases, the duration of heat treatment shall commence from the time at which the whole of each part attains the specified temperature.

6.3.1.1 The treatment selected, of necessity, must be based upon experience with the part or empirical test data. Therefore, Class SR-0 treatment is provided for parts that the purchaser wishes to exempt from treatment. However, many, if not most, steels with a tensile strength in excess of 1000 MPa will become embrittled when plated with chromium. The stress relief and hydrogen embrittlement relief treatments are essential for the safe performance of chromium plated items fabricated from those steels. Selection of Class SR-0 or ER-0 requires thorough knowledge of the embrittleness susceptibility of the specific steel employed. When the purchaser specifies Class SR-0 or ER-0, the purchaser assumes sole responsibility for any embrittlement failure of the part. The relative susceptibility of a steel can be determined by subjecting it to the Disk Rupture Test of Test Method F 1459. When no stress relief treatment is specified by the purchaser then Class SR-1 shall be applied.

6.3.2 Parts having surface hardened areas that would suffer an unacceptable reduction in hardness by treatment in accordance with Specification B 849 shall be heat-treated at a lower
temperature but not less than 130°C for a minimum period of 8 h. This treatment is applicable for parts made of steel with an actual tensile strength below 1400 MPa. The purchaser may require that the heat-treatment temperature shall not reduce the surface hardness. Shorter times at higher temperatures may be used, if the resulting loss of surface hardness is acceptable.

6.3.3 If stress relief is given after shot peening or other cold working processes to introduce beneficial compressive stresses, the temperature shall not exceed 230°C.

6.4 Hydrogen Embrittlement Relief:

6.4.1 Heat treatment appropriate for the tensile strength of the electroplated part (see Specification B 850) shall be performed to reduce the risk of hydrogen embrittlement. In all cases, the duration of the heat treatment shall commence from the time at which the whole part attains the specified temperature. See 6.3.1.1 for important embrittlement relief information regarding the selection of ER-0. When no embrittlement relief treatment is specified by the purchaser then Class ER-1 shall be applied.

6.4.2 Begin the embrittlement relief heat-treatment as soon as practical following the plating process but no longer than 1.5 h.

6.4.3 Parts or representative specimens shall be tested for compliance in accordance with 7.5.

6.5 Thickness—The thickness of the coating everywhere on the significant surface(s) shall conform to the requirements of the specified class as defined in Section 3 (see 7.2).

Note 2—The coating thickness requirements of this specification are a minimum requirement, that is, the coating thickness is required to equal or exceed the specified thickness everywhere on the significant surfaces (see 4.1). Variation in the coating thickness from point to point on a coated article is an inherent characteristic of electroplating processes. Therefore, the coating thickness must exceed the specified value at some point on the significant surfaces to ensure that the thickness equals or exceeds the specified value at all points. Hence, in most cases, the average coating thickness on an article will be greater than the specified value; how much greater is largely determined by the shape of the article (see Practice B 507) and the characteristics of the electroplating process. In addition, the average coating thickness on articles will vary from article to article within a production lot. Therefore, all of the articles within a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the average necessary to ensure that a single article meets the requirement. This may not apply to parts that are ground after plating.

6.6 Adhesion—The coating shall be sufficiently adherent to the basis metal to pass the adhesion test specified (see 7.3). These tests are, with the possible exception of the heat quench test, all destructive and therefore, in most cases, should be performed on test panels.

Note 3—Adhesion may be influenced by the method of pretreating the base metal and the type of steel used as a basis metal. Helpful information is given in Practices B 177, B 183, B 242, and B 320.

6.7 The coating shall be sufficiently free of pores to pass the porosity test specified (see 7.4).

6.8 Workmanship—Adding to (spotting in) or double electroplating, unless evidence of a satisfactory bond is established, shall be cause for rejection. Stripping and replating is permitted but parts having an ultimate tensile strength greater than 1000 MPa or a hardness greater than 32 HRC that are acid stripped shall be rebaked (see 6.3) before plating. Baking after stripping is not necessary if the parts are stripped anodically in an alkaline solution.

6.9 Supplemental Requirements—If parts are electroplated and subsequently ground to size, the grinding shall be done with a proper coolant, never dry, and with a sufficiently light cut to prevent cracking.8 Macrocracking, visually observed without magnification after grinding, shall be cause for rejection.

6.10 Packaging—Part(s) plated for the U.S. Government and Military, including subcontracts, shall be packaged in accordance with Practice D 3951.

7. Test Methods

7.1 Separate Specimens—When the coated articles are of such a form as not to be readily adaptable to a test specified herein, when destructive tests would unreasonably reduce the number or pieces in small lots, when the pieces are too valuable to be destroyed, and when specified by the purchaser, tests shall be made by the use of separate specimens plated concurrently with the articles represented. The separate specimens shall be of a basis metal equivalent to that of the parts represented. Equivalent basis metal includes chemical composition, grade, condition, and finish of surface prior to electroplating. The purchaser is responsible for providing these specimens (see section 5.3). These specimens shall be introduced into a lot before the cleaning operations preliminary to electroplating and shall not be separated therefrom until after completion of electroplating. Conditions affecting the electroplating of specimens, including the spacing and positioning in respect to anodes and to other objects being electroplated, shall correspond as nearly as practicable to those affecting the significant surfaces of the articles represented. Unless a need can be demonstrated, separately prepared specimens shall not be used in place of production items for nondestructive tests and visual examination.

7.2 Thickness—Measure the thickness of the chromium by one of the following methods; other methods may be used if it can be demonstrated that the uncertainty of the method is less than 10 % or unless direct physical measurement by mechanical means of the final part dimension is specified.

7.2.1 Microscopical—Test Method B 487 (this method is destructive).

7.2.2 Magnetic—Test Method B 499.

7.2.3 Coulometric—Test Method B 504 (this method destroys the coating in the test location).

7.2.4 X-Ray Spectrometry—Test Method B 568.

7.3 Adhesion:

7.3.1 The coated article or designated test specimen shall pass one of the following tests, or any special test particular to the function of the part as specified by the purchaser.

7.3.1.1 Bend Test—This method is destructive.

7.3.1.2 File Test—This method is destructive.

7.3.1.3 Heat and Quench Test—This method may affect the heat treatment of the basis metal.

7.3.1.4 Push Test—This method is destructive.

7.3.2 These and other adhesion tests are described in Test Methods B 571. The test selected should take into consideration the size, shape, or thickness of the part. Adhesion tests may at times fail to detect adhesion in the process of degradation; subsequent fabrication may reveal poor or inadequate adhesion that shall be cause for rejection.

7.4 Porosity—The coating shall pass one of the following tests as specified by the purchaser:

 NOTE 4—It is important to realize that the test duration specified for each test must be used exactly for valid results.

7.4.1 Ferroxyl Test—Conduct in accordance with the procedure described in Appendix X1. Observe the results after 10 min. The part fails if more than the number of pores specified by the purchaser per part or per unit area are found.

7.4.2 Neutral Salt Spray—Conduct in accordance with Practice B 117. Observe results after 16 h. The part fails if more than the number of pores specified by the purchaser per part or per unit area are found.

7.4.3 Copper Sulfate Test—Immerse the coated part in a 15-g/L solution of copper sulfate (CuSO4·5H2O) for 2 min. Inspect for copper spots that indicate pores. The part fails if more than the number of pores specified by the purchaser per part or per unit area are found.

7.5 Hydrogen Embrittlement—Representative sample parts or test specimens (see 7.5.1) plated concurrently with the parts, shall be subjected to a sustained load test. In the case of parts, the samples shall be subjected for 200 h to a sustained tensile load equal to 115% of the maximum design load for which the part was designed as specified by the purchaser. If test specimens are employed, they shall be subjected for 200 h to a load equal to 75% of the ultimate tensile strength of the alloy employed. Any fractures or signs of cracks shall be cause for rejection.

7.5.1 Separate specimens for embrittlement relief testing shall be round notched specimens of the alloy being plated or a metallurgically equivalent alternative specified by the purchaser. The specimens shall be prepared with the axis of the specimen perpendicular to the short traverse grain direction and shall conform to Fig. 8 of Test Methods E 8 and shall have a 60 V-notch, the bottom of which shall have a radius of curvature of 0.254 ± 0.0127 mm and the area of which shall be approximately equal to half the area of the specimen’s reduced section.

7.5.1.1 The specimen or the raw material from which they may be machined shall be provided by the purchaser or as agreed upon between the purchaser and the seller.

7.5.2 Alternative methods for the determination of the efficacy of the hydrogen embrittlement relief may be used as agreed upon between the purchaser and the seller.

8. Sampling Requirements

8.1 The use of statistical process control in the coating process is strongly recommended. Properly performed, this will help ensure coated products of satisfactory quality and will reduce the amount of acceptance inspection required.

8.2 The sampling plan shall be Test Method B 602 unless otherwise specified by the purchaser. Other sampling plans are contained in Guide B 697 and Method B 762.

8.3 Select a random sample of the size required by the test method selected from the inspection lot (see 8.2). Inspect the articles in the lot for conformance to the requirements of this specification and classify the lot as conforming or not conforming to each requirement in accordance with the criteria of the sampling plans in the method selected.

 NOTE 5—Test Method B 602 contains four sampling plans, three of which are to be used with nondestructive test methods. The fourth plan is used where the test method is destructive. The three plans for nondestructive tests differ in the quality level they require of the product. Test Method B 602 requires use of the plan with the intermediate quality level, unless the purchaser specifies otherwise. It is recommended that the purchaser compare the plans with his/her needs and state which plan is to be used. If the plans in Test Method B 602 do not serve those needs, additional ones are given in Guide B 697. Both Test Method B 602 and Guide B 697 list references where additional information on sampling inspection and additional plans are given.

 NOTE 6—When both destructive and nondestructive tests exist for the measurement of a characteristic, the purchaser needs to state which is to be used so that the proper sampling plan is selected. Whether or not a test is destructive may not always be clear. A test may destroy the coating but in a noncritical area, or, although it may destroy the coating, the part can be reclaimed by stripping and recoating. The purchaser needs to state whether or not the test is to be considered destructive or nondestructive. The decision is important because the plans for destructive tests are significantly less able to discriminate between acceptable and unacceptable lots. This is because fewer parts are tested in destructive plans.

8.4 An inspection lot shall be defined as a collection of coated parts that are of the same kind, that have been produced to the same specification, that have been coated by a single producer at the one time or approximately the same time under essentially the same conditions, and that are submitted for acceptance or rejection as a group.

8.5 All specimens used in the sampling plan shall be made of the same basis material in the same metallurgical conditions as the articles being plated to this specification.

8.6 All specimens shall be provided by the purchaser unless otherwise agreed upon by the producer.

9. Keywords

9.1 chromium plating standard; steel
APPENDIX

(Nonmandatory Information)

X1. FERROXYL TEST

X1.1 Scope—This method reveals discontinuities, such as pores, in coatings of chromium on iron or steel.

X1.2 Material—The test solution is prepared by dissolving 10 g of agar, 10 g of sodium chloride (NaCl), and 1 g of potassium ferricyanide (KFe(CN)) in 1 L of warm distilled or deionized water.

X1.3 Procedure—Clean and degrease the surface to be tested with methyl alcohol or other suitable solvent (avoid inhalation of fumes). Slowly warm the test solution to 93°C to liquify (a borosilicate glass double boiler arrangement is suggested). Apply the warmed test solution to the specimen by one of the following methods: (1) dip the specimen in the solution, (2) pour the solution over the specimen, or (3) dip a piece of filter paper into the solution, allow the excess to drain off, and then apply the wet paper to the test area and allow to remain undisturbed. (Do not apply the solution if its temperature is below 60°C.) Place the test specimen in a horizontal position allowing it to cool to between 21 and 27°C. Begin timing the 10-min test period from the time the agar solution is on the test specimen. At the end of the test period examine the coating or remove the filter paper and examine the aside that was in contact with the specimen. Blue spots indicate basis metal corrosion or porosity.

X1.4 Report:

X1.4.1 Report the following information:

X1.4.1.1 Area of the surface tested,

X1.4.1.2 Total number and diameter of all spots visible to the unaided eye, and

X1.4.1.3 The highest number of spots visible at one time through a template placed on the surface with a 25 by 25-mm opening.

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Designation: B 651 – 83 (Reapproved 2001)


This standard is issued under the fixed designation B 651; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method provides a means for measuring the average dimensions and number of corrosion sites in an electroplated decorative nickel plus chromium or copper plus nickel plus chromium coating on steel after the coating has been subjected to corrosion tests. This test method is useful for comparing the relative corrosion resistances of different electroplating systems and for comparing the relative corrosivities of different corrosive environments. The numbers and sizes of corrosion sites are related to deterioration of appearance. Penetration of the electroplated coatings leads to appearance of basis metal corrosion products.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section

3. Summary of Test Method

3.1 The depths and diameter of corrosion pits or the widths of corrosion crevices, and the number of pits per square millimetre or crevices per linear millimetre on a specimen surface, are determined using optical aids (magnifier, microscope, and interference microscope). The values are compared to dimensions and numbers of corrosion sites obtained from other specimens.

4. Significance and Use

4.1 Different electroplating systems can be corroded under the same conditions for the same length of time. Differences in the average values of the radius or half-width or of penetration into an underlying metal layer are significant measures of the relative corrosion resistance of the systems. Thus, if the pit radii are substantially higher on samples with a given electroplating system, when compared to other systems, a tendency for earlier failure of the former by formation of visible pits is indicated. If penetration into the semi-bright nickel layer is substantially higher, a tendency for earlier failure by corrosion of basis metal is evident.

5. Apparatus

5.1 Double-Beam Interference Microscope (lateral magnification about 100×), capable of producing, with white light, a visible group of interference fringes, and equipped with a calibrated fine focus and a graduated bifilar (movable cross hair) eyepiece.

5.2 Magnifier or Microscope (10× to 20×), with light source.

5.3 Rule, graduated in millimetres, and a scriber for producing visible lines on the specimen surface.

5.4 Microscope, with a magnification capability of 500×, equipped with a bifilar eyepiece, for making measurements on opaque surfaces.

5.5 Equipment for mounting and polishing of specimens for microscopical cross-sectional measurements.

6. Specimen Preparation

6.1 Clean the corroded specimen surface with an agent or agents that remove soil and corrosion products, but do not significantly change the surface of the corrosion sites. Scouring powder may be used to remove insoluble corrosion products, organic solvent to remove road tar, water accompanied by gentle abrasion with a cloth to remove lightly adherent soil, etc.

6.2 Mask with paint or tape that portion of the specimen surface on which no measurements of pits or cracks will be made. Alternatively, a gasketed cell pressed onto the surface may be used. The opening in the gasket will define the area to be stripped.

NOTE 1—If pitted, the area selected for measurement should contain at least 100 pits or be as large as 50 by 50 mm. If the area contains cracks, the location for measurement should contain at least 100 cracks, or be at least 50 mm long.

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6.3 Strip the chromium anodically at 6 to 8 V in a solution containing about 50 g/L of sodium carbonate (Na₂CO₃).

6.4 Remove masking material, if desired.

Note 2—If tape was employed for masking, its removal is recommended. When the specimen rests on tape, it will allow the specimen to settle slowly. This gradual movement interferes with measurements of penetration with the interference microscope.

7. Procedure for Determination of Average Number of Pits or Cracks

7.1 Using the 10× to 20× magnifier, count the number of pits in a known area or the number of cracks intersecting a line of known length. Where uncertainty exists as to whether localized blemishes are corrosion sites when the magnifier is employed, use the 100× microscope for verification. Extreme accuracy is not necessary; values within ±10% of the true value are adequate.

7.1.1 For surfaces where the number of pits is more than about 1000/cm², count the pits bounded by lines seen in the eyepiece reticle of the 100× microscope enclosing a known area of specimen surface (probably about 0.5 mm²).

7.1.2 For surfaces where the number of pits is less than about 1000/cm², lightly scribe lines 10 mm or less apart to form a rectilinear grid on the surface. Count the number of pits within a scribed area, by using the magnifier, or the 100× microscope, whichever has the necessary resolution to assure pit identification. Determine the area that contains about 100 pits, or, if the area exceeds 25 cm², count the number of pits in a 25-cm² area.

7.1.3 For surfaces with more than about 5 cracks per millimetre, count the number of cracks on the surface image that cross a 100× microscope reticle line of known length.

7.1.4 For a surface with fewer than about 5 cracks per millimetre, lightly scribe a straight line up to 50 mm long on the specimen surface. Using a magnifier or, if necessary, a 100× microscope, count the number of cracks in a known length of line, or all the cracks in 50-mm length, whichever comes first.

Note 3—If the cracks tend to be oriented, scribe the line approximately perpendicular to the predominant crack direction.

7.2 Calculate the number of pits as pits per square millimetre, or the number of cracks as cracks per millimetre. Enter result in Table 1 under “pit density” or “crack density.”

8. Determination of Mean Dimensions of Pits or Cracks

8.1 Observe one pit or crack with the interference microscope.

8.1.1 Using the bifilar eyepiece, count the number of eyepiece scale units occupied by the major diameter of the pit, or by the width of the crack. If the crack width varies, or if the pit outline is irregular, estimate the average. Enter “width” value in Table 1.

8.1.2 Adjust the elevation of the microscope tube so that interference fringes appear in the deepest part of the pit or the crack (the portion seen in the field of view) being measured; enter the reading on fine-focus knob under B in Table 1. Using the fine-focus knob only, raise the tube so that the fringes appear on the uncorroded surface surrounding the corroded site, and so that the center of the fringe group is aligned with the location of the penetration measurement. The best fringe orientation is perpendicular to the major pit diameter or crack direction. Enter the reading on fine-focus knob under A in Table 1. Subtract B from A to obtain penetration P and enter the value into Table 1. (Fine-focus knobs are generally calibrated directly in micrometres, necessitating no further conversion.)

Note 4—If the bottoms of the corrosion sites do not produce visible fringes, treat the specimen with a suitable agent to clean or to brighten the sites. Then repeat the steps in 8.1 on ten pits or cracks. A 15-s soak in a water solution of 5% H₂SO₄, by weight, followed immediately with a water rinse, is often helpful.

8.2 Section the specimen in the location of the above measurements. Mount, polish, etch, and measure (Note 5) the thickness of each deposit; enter the values into Table 1 under respective values of “Thickness.” Employ a microscope having a magnification capability of at least 500×.

Note 5—For a guide to the procedure for measuring the thickness of each deposit consult Test Method B 487.

Note 6—A suggested etchant is 1 part by volume glacial acetic acid, 1

---

**TABLE 1 Measurements of Corrosion Pits and Cracks in Nickel Plus Chromium or Copper Plus Nickel Plus Chromium Plated Surfaces**

<table>
<thead>
<tr>
<th>SAMPLE NO.</th>
<th>EXPOSURE MEDIUM</th>
<th>EXPOSURE TIME</th>
<th>Pit or crack density (7.2)</th>
<th>Pit or crack dimensions (8.1.1)</th>
<th>Thicknesses of deposits (8.2)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>__ pits/mm²</td>
<td>__ cracks/mm²</td>
<td>copper ____ µm</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>__ µm</td>
<td>__ µm</td>
<td>semibright nickel ____ µm</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>bright nickel ____ µm</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>other nickel layer ____ µm</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>chromium ____ µm</td>
</tr>
</tbody>
</table>

**Penetration of Crack or Pit into the Semibright Nickel or Copper Layers**

<table>
<thead>
<tr>
<th>Total Penetration (P) (8.1.2)</th>
<th>Penetration into Semibright Nickel (8.3)</th>
<th>Penetration into Copper (8.4)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>____ µm</td>
<td>____ µm</td>
</tr>
<tr>
<td>B</td>
<td>____ µm</td>
<td>____ µm</td>
</tr>
<tr>
<td>A-B</td>
<td>____ µm</td>
<td>mean penetration into semibright ____ µm</td>
</tr>
<tr>
<td></td>
<td></td>
<td>mean penetration into copper  ____ µm</td>
</tr>
</tbody>
</table>
part concentrated nitric acid, (sp gr 1.42) and 1 part glycerin. Approximate etch time is 30 s.

8.3 For the determination of penetration by corrosion into the semi-bright nickel layer, subtract the value of thickness of all deposits above the semi-bright layer (obtained in 8.2) from each value of penetration \( P \) (obtained in 8.1.2); enter each value of penetration into Table 1 into the column designated “semi-bright penetration.” Calculate the arithmetic mean of the values and enter the “Mean Penetration,” \( P_{sb} \) into Table 1.

8.4 For determination of penetration into the copper layer, subtract the thickness of all the layers above copper from the 1 penetration value \( A - B \) in Table 1. Calculate the arithmetic mean of the differences, and enter “Mean Penetration,” \( P_{cu} \) into Table 1.

9. Report

9.1 Report the following information:
9.1.1 Sample number or identification,
9.1.2 Exposure medium,
9.1.3 Exposure time,
9.1.4 Thickness of deposits,
9.1.5 Pit or crack density,
9.1.6 Pit or crack dimensions; width and penetration,

NOTE 7—Level \( A \) (Table 1) is the reading on the calibrated fine-focus knob corresponding to the specimen surface plane. Level \( B \) is the reading on the calibrated fine-focus knob corresponding to the maximum depth of the pit or crack. \( A - B \) is the depth of the pit or crack.

10. Precision and Bias

10.1 Precision of individual penetration measurements can be as good as \( \pm 1 \mu m \). It is determined by the care with which the interference fringes, which move laterally across the field of view as the focus knob is turned, are positioned (either in the corrosion site or on the top surface).

10.2 Bias of individual penetration measurements can be as good as the precision, since the method has no built-in bias; it can be even better than the precision when computed as a statistical average. Bias is determined by the precision and by the care with which the pits are cleaned of foreign material but have had no metal removed by etching of any cleaning agent.

10.3 Bias of individual pit diameter measurements and thickness measurements can be as good as \( \pm 1 \mu m \). Suggestions for obtaining measurements of best bias are found in Test Method B 487.

10.4 Biases of the means obtained for penetration, site diameter or width, and number are determined by the uniformity of distribution of pit size and density across the surface and by the number of individual pits measured that are used to obtain the mean. Values of number and size of corrosion sites need not be more accurate than \( \pm 10\% \) of the true average value.
Standard Test Method for
Metallographic Determination of Microstructure in Cemented Tungsten Carbides

This standard is issued under the fixed designation B 657; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope
1.1 This test method covers apparatus and procedures for the metallographic determination of microstructures in cemented tungsten carbides.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Precautions applying to use of hazardous laboratory chemicals should be observed for chemicals specified in Table 1.

2. Referenced Documents
2.1 ASTM Standards:
B 665 Practice for Metallographic Sample Preparation of Cemented Tungsten Carbides

2.2 ISO Standard:
ISO 4499 Hardmetals—Metallographic Determination of Microstructure

3. Terminology
3.1 Definitions of Symbols:

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>α phase</td>
<td>tungsten carbide</td>
</tr>
<tr>
<td>β phase</td>
<td>binder (for example, Co, Ni, Fe)</td>
</tr>
<tr>
<td>γ phase</td>
<td>carbide of a cubic lattice (for example, TiC, TaC, NbC) that may contain other carbides (for example, WC) in solid solution</td>
</tr>
<tr>
<td>γ-type phases</td>
<td>multiple carbides of tungsten and at least one metal of the binder</td>
</tr>
</tbody>
</table>

4. Significance and Use
4.1 The microstructure of cemented tungsten carbide affects the material’s mechanical and physical properties. This is not intended to be used as a specification for carbide grades, producers and users may use the microstructural information as a guide in developing their own specifications.

5. Apparatus
5.1 Metallographic Microscope capable of magnifications up to 1500 times.

5.2 Ordinary metallurgical laboratory equipment.

5.3 Equipment for specimen preparation as outlined in Practice B 665.

6. Specimen Preparation
6.1 A suitable procedure is described in Practice B 665.

7. Procedure
7.1 Examine the microstructure by gradual development of the phases by etching. Examples of suitable etching techniques are given in Table 1.

7.2 Determine the presence of γ-type phases by lightly etching the section with Technique 1 (see Table 1). Phases of γ-type are colored orange to brown. γ-Phase may etch lightly, while the other phases remain unetched. Etching by Technique 1 does not preclude subsequent etching by Techniques 2 or 3. Examine the entire section at low magnification and, if necessary, at magnifications up to 1500 times. Note and record the existence of γ-type phases and their distribution.

7.3 Determine the presence of γ phases by etching with Technique 2 (Table 1). This phase appears light yellowish brown and has a typically rounded shape (see Fig. 1). Examine the etched section and note and record the existence of a γ phase. Estimate and record its size according to Fig. 1 as γ–fine, γ–medium, or γ–coarse.

7.4 Determine the presence of α phase by etching the section with Technique 3, or in case γ phase is present, with Technique 2. The α phase appears gray. Examine the etched section and note and record the presence of an α phase. Estimate and record its size according to Fig. 2 as α–fine, α–medium, or α–coarse.

7.5 Identify the β phase after etching the surface by Technique 3 (Table 1). This phase remains white.

NOTE 1—This procedure follows essentially ISO 4499.

8. Report
8.1 The report shall include complete identification of the
specimen and the results obtained.

9. Precision and Bias

9.1 The nature of this test method precludes any statement of precision or bias.

10. Keywords

10.1 cemented carbides; hardmetals; microstructure; powder metallurgy

### TABLE 1 Etching Techniques

<table>
<thead>
<tr>
<th>Etching Technique</th>
<th>Composition of Etchants</th>
<th>Conditions of Etching</th>
<th>Objective of Etching</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 A</td>
<td>Freshly prepared mixture of equal quantities of 10 % (mass/mass) aqueous solutions of K₃Fe(CN)₆ (III) (potassium ferricyanide) and potassium or sodium hydroxide</td>
<td>Etch in mixture A at approximately 20°C for 2 to 10 s. Flush the test-piece section with water immediately, without removing the oxide layer. Dry the surface carefully with acetone or alcohol without wiping.</td>
<td>Identification of ( \eta ) phases</td>
</tr>
<tr>
<td>2 A B</td>
<td>Same as 1A; mixture of equal volumes of concentrated hydrochloric acid and water</td>
<td>Etch at approximately 20°C in mixture A for 3 to 4 min. Then wash in water and etch in mixture B for approximately 10 s. Next wash in water, then in alcohol and dry the test-piece section. Finally, etch in mixture A for approximately 20 s.</td>
<td>Identification of ( \gamma ) phase</td>
</tr>
<tr>
<td>3 A</td>
<td>Same as 1A</td>
<td>Etch in mixture A at approximately 20°C for 3 to 6 min.</td>
<td>Identification of ( \alpha ) phase</td>
</tr>
</tbody>
</table>

**Note:** The separate solutions of potassium hexacyanoferrate (III) and potassium or sodium hydroxide may be stored for a long time, but must be freshly mixed each day when used.
FIG. 1 $\gamma$ Phase 1500×.
FIG. 1 (1500×) (continued)

\[ \gamma - \text{coarse} \]
FIG. 2 α Phase 1500×.
FIG. 2 (1500×) (continued)

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Standard Guide for Measuring Thickness of Metallic and Inorganic Coatings

This standard is issued under the fixed designation B 659; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide outlines the methods for measuring the thickness of many metallic and inorganic coatings including electrodeposited, mechanically deposited, vacuum deposited, anodic oxide and chemical conversion coatings.

1.2 This guide is limited to tests considered in ASTM standards and does not cover certain tests that are employed for special applications.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- B 244 Test Method for Measurement of Thickness of Anodic Coatings on Aluminum and of Other Nonconductive Coatings on Nonmagnetic Basis Metals with Eddy-Current Instruments
- B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section
- B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
- B 530 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Electrodeposited Nickel Coatings on Magnetic and Nonmagnetic Substrates
- B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
- B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
- B 588 Test Method for Measurement of Thickness of Transparent or Opaque Coatings by Double-Beam Interference Microscope Technique

2.2 ISO Standards:

- 1463 Metal and Oxide Coatings—Measurement of Thickness by Microscopic Examination of Cross Sections
- 2128 Surface Treatment of Metals—Anodization (Anodic Oxidation) of Aluminum and Its Alloys—Measurement of the Thickness of Oxide Coatings—Nondestructive Measurement by Light Section Microscope
- 2176 Petroleum Products Lubricating Grease Determination of Dropping Point
- 2177 Metallic Coatings—Measurement of Coating Thickness—Coulometric Method by Anodic Solution
- 2178 Non-Magnetic Metallic and Vitreous or Porcelain Enamel Coatings on Magnetic Basis Metals, Measurement of Coating Thickness, Magnetic Method
- 2360 Non-Conductive Coatings on Non-Magnetic Basis Metals—Measurement of Coating Thickness—Eddy Current Method
- 2361 Electrodeposited Nickel Coatings on Magnetic and Non-Magnetic Substrates—Measurement of Coating Thickness—Magnetic Method
- 3497 Metallic Coatings—Measurement of Coating Thickness—X-Ray Spectrometric Methods
- 3543 Metallic and Non-Metallic Coatings—Measurement of Thickness—Beta Backscatter Method

3. Significance and Use

3.1 Most coating specifications specify the thickness of the coating because coating thickness is often an important factor in the performance of the coating in service.

3.2 The methods included in this guide are suitable for acceptance testing and are to be found in ASTM standards.

3.3 Each method has its own limitations with respect to the kind of coating and its thickness.

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1 This guide is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on Test Methods.


3 Discontinued. See 2000 Annual Book of ASTM Standards, Vol 02.05.

4 Available from American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.
4. Reliability of Methods

4.1 All methods covered by this guide are sufficiently reliable to be used for acceptance testing of many electroplated and other coatings. That is, each method is capable of yielding measurements with an uncertainty of less than 10 % of the coating thickness over a significant range of coating thicknesses when used by properly instructed personnel.

5. Nondestructive Methods

5.1 Magnetic Methods—These methods employ instruments that measure the magnetic attraction between a magnet and the coating or the substrate or both, or that measure the reluctance of a magnetic flux path passing through the coating and the substrate. These methods, in practice, are limited to nonmagnetic coatings on carbon steel (Test Method B 499 and ISO 2178) and to electrodeposited nickel coatings on carbon steel or on nonmagnetic substrates (Test Method B 530 and ISO 2361) and to nonmagnetic autocatalytically deposited nickel-phosphorus alloys on carbon steel (Test Method B 499 and ISO 2176). Coating thickness gages of this type are available commercially.

5.2 Eddy-Current Method—This method employs an instrument that generates a high-frequency current in a probe, inducing eddy currents near the surface of the test specimen. The magnitude of the eddy currents is a function of the relative conductivities of the coating and substrate materials and the coating thickness. Because variation in the electroplating process can change the electrical properties of the coating and hence, instrument response for a given thickness, the use of eddy-current instruments is usually limited to the measurement of nonconductive coatings on nonmagnetic basis metals (Test Method B 244 and ISO 2360). These instruments are, however, also suitable for the thickness measurement of high-conductivity metal (for example, copper and silver) coatings on nonconductive substrates. Coating thickness gages of this type are available commercially.

5.3 X-Ray Fluorescence Methods:

5.3.1 These methods cover the use of emission and absorption X-ray spectrometry for determining the thickness of metallic coatings up to about 15 µm. The upper limit may be significantly above or below 15 µm depending on the coating material and on the equipment used. When exposed to X rays, the intensity of the secondary radiation emitted by the coating or by the substrate followed by attenuation by the coating is measured. The intensity of the secondary radiation is a function of the coating thickness.

5.3.2 In multiple coatings the X-ray method is generally applicable to the final metal coating.

5.3.3 Suitable equipment is available commercially (Test Method B 568 and ISO 3497).

5.4 Beta Backscatter Method:

5.4.1 The beta backscatter method employs radioisotopes that emit beta radiation and a detector that measures the intensity of the beta radiation backscattered by the test specimen. Part of the beta radiation entering the material collides with atoms of the material and is scattered back towards the source. The intensity of the backscattered radiation is a function, among others, of the coating thickness. A measurement is possible if the atomic number of the coating material is sufficiently different from that of its substrate and if the beta radiation is of suitable energy and intensity. The method can be used for measuring both thin and thick coatings, the maximum thickness being a function of the atomic number of the coating. In practice, high atomic number coatings, such as gold, can be measured up to 50 µm, while low atomic number coatings, such as copper or nickel, can be measured up to about 200 µm.

5.4.2 Coating thickness gages of this type are available commercially (Test Method B 567 and ISO 3543).

6. Semidestructive Methods

6.1 Coulometric Method:

6.1.1 Coating thickness may be determined by measuring the quantity of electricity consumed in dissolving the coating from an accurately defined area when the article is made anodic in a suitable electrolyte under suitable conditions. The change in potential occurring when the substrate is exposed indicates the end point of the dissolution. The method is applicable to many coating-substrate combinations (Test Method B 504 and ISO 2177).

6.1.2 Coating thickness instruments employing this method are available commercially.

6.2 Double-Beam Interference Microscope Method—A step is formed between the coating surface and the substrate surface by dissolving a small area of coating. The height of this step is measured with a double-beam interference microscope. The method is applicable to thin coatings such as usually used for decorative chromium. It can be used to measure transparent oxide coatings without the need of forming a step (Test Method B 588).

7. Destructive Methods

7.1 Microscopical Method—In the microscopical method the thickness is measured in a magnified image of a cross section of the coating (Test Method B 487 and ISO 1463).

7.2 Gravimetric Method (Strip and Weigh):

7.2.1 The coating mass is determined by weighing the sample before and after dissolving the coating without attack of the substrate or by weighing the coating after dissolving the substrate without attack of the coating.

7.2.2 The coating thickness is given by the equation:

\[ t = \frac{m \times 10}{d \times A} \]  

where:

- \( t \) = thickness, µm,
- \( d \) = density of coating material, g/cm³,
- \( m \) = mass of coating, mg, and
- \( A \) = area covered by coating, cm².

7.2.3 Procedures for applying this method to many different coatings are given in Guide B 767.

7.2.4 A variation of this method is to weigh the item before and after electroplating or, if the current efficiency is 100 %, to measure the coulombs passed during the electroplating to determine the coating weight.
8. Other Methods

8.1 Profilometry and multiple-beam interferometry offer reliable methods of measuring coating thickness provided a step can be formed by removing a portion of the coating.

8.2 The light section microscope is used for measuring the thickness of non-opaque coatings on relatively smooth substrates (Test Method B 681 and ISO 2128).

9. Summary of Applicability of Coating Thickness Measuring Methods

9.1 The applicability and limitations of coating gages and other methods of measuring coating thickness are set forth in the pertinent ASTM and ISO standards, publications on electroplating and related finishing technology, and manufacturers’ instructions for the use of coating thickness gages. The X-ray, gravimetric, microscopical, and interference microscopical methods are applicable to almost all combinations of substrate and coatings. Table 1 indicates the substrate and coating combinations to which the beta backscatter, coulometric, eddy-current, and magnetic methods have been applied.

### TABLE 1 Applicability of Coating Thickness Measuring Methods

<table>
<thead>
<tr>
<th>Substrates</th>
<th>Copper</th>
<th>Nickel</th>
<th>Chromium</th>
<th>Auto-catalytic Nickel</th>
<th>Zinc</th>
<th>Cadmium</th>
<th>Gold</th>
<th>Palladium</th>
<th>Rhodium</th>
<th>Silver</th>
<th>Tin</th>
<th>Lead</th>
<th>Tin-Lead Alloys</th>
<th>Non-metals</th>
<th>Vitreous and Porcelain Enamels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Magnetic steel (including corrosion-resistant steel)</td>
<td>CM</td>
<td>CM*</td>
<td>CM</td>
<td>CM*</td>
<td>CM</td>
<td>BCM</td>
<td>BM</td>
<td>BM</td>
<td>BM</td>
<td>BCM</td>
<td>BCM</td>
<td>BCM</td>
<td>B*</td>
<td>C*</td>
<td>M</td>
</tr>
<tr>
<td>Nonmagnetic stainless steels</td>
<td>CE</td>
<td>CM*</td>
<td>C</td>
<td>CM*</td>
<td>C</td>
<td>BC</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>BE*</td>
<td>BC</td>
<td>BC</td>
<td>B*</td>
<td>C*</td>
<td>BE</td>
</tr>
<tr>
<td>Copper and alloys</td>
<td>C only on brass and Cu-Be</td>
<td>CM*</td>
<td>C</td>
<td>CM*</td>
<td>C</td>
<td>BC</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>BE*</td>
<td>BC</td>
<td>BC</td>
<td>B*</td>
<td>C*</td>
<td>BE</td>
</tr>
<tr>
<td>Zinc and alloys</td>
<td>C</td>
<td>M*</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B*</td>
<td>BE</td>
<td>...</td>
</tr>
<tr>
<td>Aluminum and alloys</td>
<td>BC</td>
<td>BCM*</td>
<td>BC</td>
<td>BC*</td>
<td>E</td>
<td>BC</td>
<td>BC</td>
<td>BC</td>
<td>BC</td>
<td>BC</td>
<td>BC</td>
<td>BC</td>
<td>B*</td>
<td>C*</td>
<td>E</td>
</tr>
<tr>
<td>Magnesium and alloys</td>
<td>B</td>
<td>BM*</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>E</td>
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</tr>
<tr>
<td>Nickel</td>
<td>C</td>
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<td>...</td>
<td>...</td>
<td>...</td>
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<td>BC</td>
<td>B</td>
<td>B</td>
<td>BC</td>
<td>BC</td>
<td>BC</td>
<td>B*</td>
<td>C*</td>
<td>BE</td>
</tr>
<tr>
<td>Silver</td>
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<td>...</td>
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<td>...</td>
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<td>...</td>
<td>B</td>
<td>...</td>
<td>B</td>
<td>BC</td>
<td>B*</td>
<td>BE</td>
</tr>
<tr>
<td>Glass Sealing Nickel-cobalt-iron alloys UNS No. K94610</td>
<td>M</td>
<td>CM*</td>
<td>M</td>
<td>CM*</td>
<td>M</td>
<td>BM</td>
<td>BM</td>
<td>BM</td>
<td>BM</td>
<td>BM</td>
<td>BM</td>
<td>BM</td>
<td>BCM</td>
<td>B*</td>
<td>C*</td>
</tr>
<tr>
<td>Nonmetals</td>
<td>BCE*</td>
<td>BCM*</td>
<td>BC</td>
<td>BC*</td>
<td>BC</td>
<td>BC</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B*</td>
<td>BE</td>
<td>...</td>
</tr>
<tr>
<td>Titanium</td>
<td>B</td>
<td>BM*</td>
<td>BC*</td>
<td>BE</td>
<td>BC</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B</td>
<td>B*</td>
<td>BE</td>
<td>...</td>
</tr>
</tbody>
</table>

*Method is sensitive to permeability variations of the coating.
*Method is sensitive to variations in the phosphorus content of the coating.
Method is sensitive to alloy composition.
Method is sensitive to conductivity variations of the coating.
Standard Guide for Metallographic Sample Preparation of Cemented Tungsten Carbides

1. Scope

1.1 This guide prescribes a method for preparing cemented carbides for metallographic examination.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 390 Practice for Evaluating Apparent Grain Size and Distribution of Cemented Tungsten Carbides
B 657 Test Method for Metallographic Determination of Microstructure in Cemented Tungsten Carbides

3. Significance and Use

3.1 This sample preparation procedure may be used to prepare metallographic samples for Test Method B 657 and Practice B 390. It does not include all variations of sample preparation.

4. Selection of Specimen

4.1 Cemented tungsten carbides are very often in the form of relatively small pieces; it is possible to select and mount the entire piece in such manner as to permit examination of the entire cross section. When pieces are too large for this, however, they should be sectioned, using a diamond cutoff wheel, to allow viewing as much of a representative cross section as possible. For micrographs, the area selected should represent, as nearly as possible, the entire cross section.

5. Procedure

5.1 There are several acceptable methods for preparing cemented tungsten carbide surfaces for microscopical examination. Basically, they all use diamond wheels for grinding and diamond powders for lapping. The grinding practices differ, to a minor degree, with respect to grit size of diamond. In all practices, however, the final polish is produced by extremely fine diamond powder lapping, and in all practices care must be exercised to retain the microstructure in its true form and to avoid pull-out of the softer matrix material (usually cobalt). While it is accepted that other procedures may be used successfully, this procedure has proved satisfactory in many laboratories.

5.1.1 Mounting—Where possible, specimens should be mounted in a plastic material such as phenol-formaldehyde or poly(methyl methacrylate) to facilitate polishing without rounding the edges. Larger specimens may be polished without mounting. When specimens are too large they may be sectioned using a diamond cut-off wheel or they may be fractured (appropriate safety precautions should be used when fracturing specimens). The area selected for examination should represent, as nearly as possible, the entire cross section.

5.1.2 Rough Grinding—The surface to be examined may be ground flat on a surface grinder with a resin-bonded diamond wheel (100 to 220 grit) operated at 5000 to 5500 surface feet per minute (25 to 28 m/s). After the surface is flat, several clean-up passes are required; the maximum depth of cut should be 0.0005 in. (13 µm) per pass and copious amounts of coolant should be used.

5.1.3 Polishing—Polishing in three steps using diamond powder or paste on a synthetic short-napped cloth (the reverse side of photographic paper, or manila file folders may also be used). When automatic polishing equipment is used, a resin-bonded diamond disk may be substituted in the roughing lap. For manual polishing, speeds of 500 to 600 rpm should be used; automatic polishing generally requires speeds of 100 to 200 rpm.

5.1.3.1 Roughing Lap—For the roughing lap, use NIST Grade 20 diamond powder (15 to 25 µm) dispersed in light spindle oil. Commercial diamond paste and thinner will provide similar results.

1 This guide is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.06 on Cemented Carbides.

5.1.3.2 **Second Lap**—For the second lap, use Grade 6 diamond powder (4 to 8 µm) or an equivalent paste.

5.1.3.3 **Finishing Lap**—For the finish lap, use Grade 1 diamond powder (less than 2 µm) or an equivalent paste.

**NOTE 1**—Best results are obtained by applying considerable pressure to the specimen in all lapping operations. Lack of adequate pressure will result in pulling out the softer matrix material. It is also essential that the specimen and operator’s hands be thoroughly cleaned between all grinding or polishing steps; ultrasonic cleaning is recommended.

6. **Precision and Bias**

6.1 The nature of this guide precludes any statement of precision and bias.

7. **Keywords**

7.1 cemented carbides; cemented tungsten carbides; microstructure
Standard Test Method for Solderability of Metallic-Coated Products

This standard is issued under the fixed designation B 678; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method provides a procedure for evaluating the solderability of metallic-coated products and test specimens to assure satisfactory performance in manufacturing processes requiring soldering with soft (tin-lead) solder and rosin flux. This test method is applicable only for testing coatings that are normally readily solderable such as: tin, tin-lead alloy, silver, and gold.

1.2 This test method is qualitative and broadly applicable. It is easy to perform and requires only simple equipment. There are other solderability tests not covered by this test method that are more applicable to specific situations, yield quantitative results, or both. Several are described in the literature.2 This is a “go-no-go” test and does not grade solderability as excellent, good, fair, and so forth.

1.3 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   B 32 Specification for Solder Metal
   D 509 Test Methods of Sampling and Grading Rosin
   D 1193 Specification for Reagent Water

3. Summary of Test Method

3.1 The specimen to be tested is coated with rosin flux, dipped briefly into molten tin-lead solder, and examined for complete and uniform coverage by the solder. When specifically required, the specimens are artificially aged before testing by exposure to hot, humid air.

4. Significance and Use

4.1 In order that a sound solder joint be formed simply and quickly in a production operation, the molten solder must readily wet and spread over the surfaces of the products being joined. For this to happen, the surfaces must be clean or be soiled only with contaminants that are easily removed by an appropriate flux. It often is necessary that the flux be only strong enough to remove the normally occurring soils. A more aggressive flux may corrode the product and have other harmful effects. Nonactivated rosin in alcohol is the standard flux used in this test method; however, provision is made for the use of other fluxes. Since rosin is a mild flux, it provides better discrimination between acceptable and unacceptable solderability in marginal cases than do more active fluxes.

4.2 Metallic coatings are frequently used to provide solderable surfaces. But, an improperly produced coating may not yield the required solderability. There are many coating defects that cause poor solderability including porosity, codeposited impurities, incorrect thickness, and surface contamination. It may be difficult or impractical to test a coating directly for each of the undesirable conditions. In these instances solderability is tested. Products that pass the solderability test can be expected to solder satisfactorily in production. In the case of failure to pass the test, the test results will not reveal the cause of the inadequate solderability, though, with experience, an operator may be able to identify the cause.

4.3 This test method measures the ability of a coated product to be soldered with 60/40 tin/lead solder using a nonactivated rosin flux. This solder and this flux, or an activated form of it, are generally used in the assembly of electronic products.

4.4 It is intended that the tested specimens be components of electronic products or articles with the same general shape and mass. Articles that are much more massive than this will heat up too slowly during the solder immersion. If more massive specimens are to be tested, a longer immersion time will have to be used, the time to be determined by experiment.

4.5 If the specimen tested is longer than 25 mm, its bottom end will be in the solder for significantly longer than the specified time. Therefore, if the specimen is longer than 25 mm, the results obtained at the bottom end of the specimen are invalid. This part of the specimen shall be discounted in the evaluation of the results. A second set of tests can be run on additional specimens in which the specimens are only partly immersed. These would be used to evaluate the bottom ends.

1 This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on Test Methods.


5 Annual Book of ASTM Standards, Vol 06.03.

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5. Flux

5.1 The flux shall be a 25 ± 5 mass % solution of water-white rosin, as defined by Test Methods D 509, Grade WW, dissolved in isopropyl alcohol of a minimum purity of 99 mass % (Note 1). A different flux, such as mildly activated and activated rosin fluxes, may be used if the specifying authority requires it. Such deviation from the standard shall be stated in the test report.

Note 1—Suitable fluxes are commercially available. Care must be taken that the commercial flux used is nonactivated, rosin flux. Commercial fluxes of higher concentration may be thinned with isopropyl alcohol to give the required concentration.

5.2 Solder:

5.2.1 The solder shall be an alloy of 60 mass % tin and 40 mass % lead that conforms to alloy Grade 60A of Specification B 32.

5.2.2 The composition of molten solder will gradually change because of oxidation. Also, the immersion of test specimens can introduce metallic impurities into the solder. For these reasons, the solder shall be replaced after being molten for 8 h unless chemical analysis shows it to meet the requirements of 5.2.1.

5.3 Water—The water used in the aging chamber shall be distilled or deionized water meeting the requirements for Type II or Type III reagent water as defined in Specification D 1193.

6. Sampling

6.1 The nature and the number of specimens shall be given by the specification covering the coating or the coated product or other governing document.

7. Apparatus

7.1 Solder Pot, large enough that when it is filled to its normal capacity the mass of the solder is at least 100 times the mass of the specimen that will be tested.

Note 2—If there is insufficient solder in the pot, the immersion of a room-temperature specimen will cool the solder out of the test range.

8. Procedure

8.1 Do not clean the specimens. The solderability test is to be performed on them in their as-received condition. If in the normal production process the specimens are cleaned before they are soldered, it may be preferred to clean the test specimens in the same way. If such cleaning is required, it shall be specifically called for. Avoid contaminating the specimens. Particularly do not touch the areas to be tested with bare hands. Use tweezers, forceps, cotton gloves, or other appropriate means.

8.2 For tin and tin-lead coatings, if it is required that the specimens be aged before solderability testing, proceed as directed in 8.3. If the specimens are not to be aged, start the tests with 8.5.

8.3 Suspend the specimens in a vessel above boiling water and leave them there with the water boiling continuously for 24 h. Keep the vessel covered and assure that the specimens do not touch the side of the vessel and that the lower edges of the specimens are from 50 to 100 mm above the surface of the boiling water. Arrange the cover of the vessel and the condenser, if used, so that the condensed water does not drip onto the specimens.

8.4 Remove the aged specimens from the vessel and allow them to air-dry and air-cool.

8.5 Perform the remaining steps on each specimen one at a time. Dip the specimen into the flux. Remove it and allow it to drain for 30 to 60 s.

8.6 Just before the next step, skim the surface of the molten solder with a clean stainless steel scraper, stir the solder with a clean stainless steel rod, and skim the surface again. The temperature of the solder shall be 245 ± 5°C.

8.7 Immerse the specimen into the solder at a speed of 25 ± 5 mm/s, hold it in the solder for 5 ± 0.5 s, and remove it from the solder at a speed of 25 ± 5 mm/s.

Note 3—The simplicity of the test can cause a casual attitude toward the times and rates specified. Unless the operator adheres to the times and rates, a significant variability in the results can occur. There are commercial automated dipping devices that, if used, will eliminate operator variability.

8.8 After the solder coating solidifies, remove the flux residue with isopropyl alcohol or other solvent.

8.9 Examine the solder coating on the specimen using, if necessary, up to 10× magnification. Evaluate the adherence of the coating by probing or scraping it with a sharp point or a sharp blade.

8.10 The specimen shall be judged to have passed the test if the solder coating is adherent, bright, smooth, and uniform over at least 95 % of the tested surface. The remaining 5 % may contain small pin holes, dewetted areas, and roughness. If the specimens are flat coupons, the areas within 3 mm of the edges shall be excluded from the evaluation. It is recommended that tested specimens exhibiting the worst allowable cases be retained and used as acceptance standards.

9. Report

9.1 The test report shall contain the following information:

9.1.1 The ASTM designation, including the issue date, of this method,

9.1.2 A description of the specimens tested,

9.1.3 Whether the specimens were cleaned before testing, and, if so, the process used (see 8.1),

9.1.4 Whether the specimens were aged,

9.1.5 Whether the test was performed manually or with an automatic dip tester,

9.1.6 Any deviations from the standard method (for example, nature of flux, nature of solder, time of immersion),

9.1.7 The number of specimens tested and the number of specimens that failed the test, and

9.1.8 A description of the nature of any failed specimens.

10. Precision and Bias

10.1 No statement is made either on the precision or on the bias of this test method for measuring solderability because the test results merely state whether there is conformance to the criteria for success specified in the procedure.
Standard Specification for Electrodeposited Coatings of Palladium for Engineering Use

This standard is issued under the fixed designation B 679; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε1 NOTE—Warning note updated and keywords added editorially in April 2004.

1. Scope

1.1 This specification covers requirements for electrodeposited palladium coatings containing at least 99.7 mass % of palladium metal. Composite coatings consisting of palladium with a thin gold overplate for applications involving electrical contacts are also covered.

1.2 Properties—Palladium is the lightest and least noble of the platinum group metals. It has a specific gravity of 12.0, which is substantially less than gold (19.3) and platinum (21.5). This yields a greater volume or thickness of coating and, consequently, some saving of metal weight accompanied by a small sacrifice in corrosion resistance and reflectivity. The following table compares the hardness range of electrodeposited palladium with other electrodeposited noble metals and alloys (1,2).

<table>
<thead>
<tr>
<th>Material</th>
<th>Approximate Hardness (HK25)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gold</td>
<td>50–250</td>
</tr>
<tr>
<td>Palladium</td>
<td>75–600</td>
</tr>
<tr>
<td>Platinum</td>
<td>150–550</td>
</tr>
<tr>
<td>Palladium-Nickel</td>
<td>300–650</td>
</tr>
<tr>
<td>Rhodium</td>
<td>750–1100</td>
</tr>
<tr>
<td>Ruthenium</td>
<td>600–1300</td>
</tr>
</tbody>
</table>

1.3 The values stated in SI units are the preferred values. Values provided in parentheses are for information only.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 The following standards form a part of this specification to the extent referenced herein:

2.2 ASTM Standards: 3

B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Guide for Preparation of High-Carbon Steel for Electroplating
B 254 Practice for Preparation of and Electroplating on Stainless Steel
B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings
B 322 Practice for Cleaning Metals Prior to Electroplating
B 343 Practice for Preparation of Nickel for Electroplating with Nickel
B 374 Terminology Relating to Electroplating
B 481 Practice for Preparation of Titanium and Titanium Alloys for Electroplating
B 482 Practice for Preparation of Tungsten and Tungsten Alloys for Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section
B 488 Specification for Electrodeposited Coatings of Gold for Engineering Uses
B 489 Practice for Bend Test for Ductility of Electrodeposited and Autocatalytically Deposited Metal Coatings on Metals

3 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard’s Document Summary page on the ASTM website.
B 507 Practice for Design of Articles to Be Electroplated on Racks
B 542 Terminology Relating to Electrical Contacts and Their Use
B 558 Practice for Preparation of Nickel Alloys for Electroplating
B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 689 Specification for Electroplated Engineering Nickel Coatings
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 741 Test Method for Porosity in Gold Coatings on Metal Substrates by Paper Electrography
B 742 Test Method of Variables Sampling of Metallic and Inorganic Coatings
B 745 Guide for Selection of Porosity and Gross Defect Tests for Electrodeposits and Related Metallic Coatings
B 799 Test Method for Porosity in Gold and Palladium Coatings by Sulfurous Acid/Sulfur-Dioxide Vapor
B 809 Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor (Flowers-of-Sulfur)
D 1125 Test Methods for Electrical Conductivity and Resistivity of Water

3. Terminology

3.1 Definitions—Many terms used in this specification are defined in Terminology B 374 or B 542.
3.2 Definitions of Terms Specific to This Standard:
3.2.1 underplating—a metallic coating layer between the basis metal or substrate and the topmost metallic coating. The thickness of an underplating is usually greater than 1 µm (40 µin.), in contrast to a strike or flash.

4. Classification

4.1 Orders for articles to be plated in accordance with this specification shall specify the plating system, indicating the basis metal, the thickness of the underplatings, the thickness of the palladium coating, and the grade of the gold overplating according to Tables 1 and 2.

5. Ordering Information

5.1 In order to make the application of this standard complete, the purchaser needs to supply the following information to the seller in the purchase order or other governing document:
5.1.1 The name, designation, and date of issue of this standard.

---

### TABLE 1 Thickness Class

<table>
<thead>
<tr>
<th>Thickness Class</th>
<th>Minimum Thickness of Pd (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.08</td>
<td>0.08</td>
</tr>
<tr>
<td>0.15</td>
<td>0.15</td>
</tr>
<tr>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>0.50</td>
<td>0.50</td>
</tr>
<tr>
<td>0.75</td>
<td>0.75</td>
</tr>
<tr>
<td>1.00</td>
<td>1.00</td>
</tr>
<tr>
<td>1.25</td>
<td>1.25</td>
</tr>
<tr>
<td>1.5</td>
<td>1.5</td>
</tr>
<tr>
<td>2.5</td>
<td>2.5</td>
</tr>
<tr>
<td>3.0</td>
<td>3.0</td>
</tr>
<tr>
<td>5.0</td>
<td>5.0</td>
</tr>
</tbody>
</table>

*See Appendix X4 for specific applications of the various thickness classes.*

### TABLE 2 Gold Overplate

<table>
<thead>
<tr>
<th>Grade</th>
<th>Type</th>
<th>MIL-G-45204</th>
<th>Hardness (Code)</th>
<th>Thickness Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1</td>
<td>. . . . . .</td>
<td>90 HK <em>c</em>{max} (A)</td>
<td>0.05-0.12 µm</td>
</tr>
<tr>
<td>1</td>
<td>2</td>
<td>III</td>
<td>1 130-200 HK <em>c</em>{max} (C)</td>
<td>0.05-0.25 µm</td>
</tr>
</tbody>
</table>

*See Specification B 488 and Appendix X1.*

5.1.2 The coating system including basis metal, thickness class and gold overplate grade (see 4.1 and Tables 1 and 2).
5.1.3 Presence, type, and thickness of underplating (see 3.2.3).
5.1.4 Significant surfaces shall be defined (see 3.2.2).
5.1.5 Requirements, if any, for porosity testing (see 9.5):
5.1.6 Requirement, if any, for bend ductility testing (see 9.6):
5.1.7 Sampling plan employed (see Section 8), and
5.1.8 Requirement, if any, for surface coating cleanliness (absence of residual salts). See Appendix X3.

6. Manufacture

6.1 Any process that provides an electrodeposit capable of meeting the specified requirements will be acceptable.

6.2 Substrate:

6.2.1 The surface condition of the basis metal should be specified and should meet this specification prior to the plating of the parts.
6.2.2 Defects in the surface of the basis metal, such as scratches, porosity, pits, inclusions, roll and die marks, laps, cracks, burrs, cold shuts, and roughness may adversely affect the appearance and performance of the deposit, despite the observance of the best plating practice. Any such defects on significant surfaces should be brought to the attention of the supplier and the purchaser.

6.2.3 Clean the basis metal as necessary to ensure a satisfactory surface for subsequent electroplating in accordance with Practices B 183, B 254, B 281, B 322, B 343, B 481, B 482, and B 558, and Guide B 242.

6.2.4 Proper preparatory procedures and thorough cleaning of the basis metal are essential for satisfactory adhesion and performance of these coatings. The surface must be chemically clean and continuously conductive, that is, without inclusions or other contaminants. The coatings must be smooth and as free of scratches, gouges, nicks, and similar imperfections as possible.

*NOTE 1—A metal finisher can often remove defects through special...*
treatments such as grinding, polishing, abrasive blasting, chemical treatments, and electropolishing. However, these may not be normal in the treatment steps preceding the plating, and a special agreement is indicated.

6.3 Apply the coating after all basis metal preparatory treatments and mechanical operations on significant surfaces have been completed.

6.4 Racking:
6.4.1 Position parts to allow free circulation of solution over all surfaces. The location of rack or wire marks in the coating should be agreed upon between the producer and supplier.

6.5 Plating Process:
6.5.1 Nickel Underplating—Apply a nickel underplating before the palladium when the product is made from copper or copper alloy. Nickel underplatings are also applied for other reasons. See Appendix X2.

NOTE 2—In certain instances where high frequency analog signals are employed, such as wave guides, the magnetic properties of nickel may attenuate the signal. Palladium itself is non-ferromagnetic.

6.5.2 Strikes—Standard practice calls for a gold or palladium strike to follow any underplate or substrate (other than silver or platinum) immediately prior to applying the palladium.

6.5.3 Plating—Good practice calls for the work to be electrically connected when entering the bath. A minimum of 0.5 V is suggested. During electroplating it is extremely important to maintain the voltage, current density, or both beneath the value for hydrogen evolution. (See 7.2)

6.5.4 Stress Cracking—Problems associated with the incorporation of hydrogen in the palladium, which can lead to stress cracking of the coating, shall be controlled by choosing plating baths and plating conditions that minimize the H/Pd deposition ratio (3). The presence of stress-induced microcracks that penetrate to the underlying substrate or underplating can be detected with one of the porosity tests specified in 9.5.

6.5.5 Gold Overplating—Apply a thin gold overplating after the palladium in any application in which palladium plated electrical connectors are mated together in a contact pair. This process is necessary to preserve the performance of the contact surface. See Appendix X1 for other reasons for using a gold overplate.

NOTE 3—When using Type 1 gold, the thickness of the gold overplate shall not exceed 0.12 µm (5 µin.) due to increased risk of degrading durability and increasing the coefficient of friction.

6.5.6 Residual Salts—For rack and barrel plating applications, residual plating salts can be removed from the articles by a clean, hot (50 to 100°C) water rinse. A minimum rinse time of 2.5 min (racks) or 5 min (barrel) is suggested. Best practice calls for a minimum of three dragout rinses and one running rinse with dwell times of 40 s in each station when rack plating and 80 s when barrel plating. Modern high-velocity impingement type rinses can reduce this time to a few seconds. This is particularly useful in automatic reel-to-reel applications where dwell times are significantly reduced. See Appendix X3.

7. Coating Requirements
7.1 Nature of Coating—The palladium deposit shall have minimum purity of 99.7 mass %.

7.2 Appearance—Palladium coatings shall be coherent, continuous, and have a uniform appearance to the extent that the nature of the basis metal and good commercial practices permit.

7.3 Thickness—Everywhere on the significant surface (see 5.1.4), the thickness of the palladium coating shall be equal to or exceed the specified thickness. The maximum thickness, however, shall not exceed the drawing tolerance.

NOTE 4—The coating thickness requirement of this specification is a minimum requirement; that is, the coating thickness is required to equal or exceed the specified thickness everywhere on the significant surfaces while conforming to all maximum thickness tolerances given in the engineering drawing. Variation in the coating thickness from point to point on a coated article is an inherent characteristic of electroplating processes. Therefore, the coating thickness will have to exceed the specified value at some points on the significant surfaces to ensure that the thickness equals or exceeds the specified value at all points. Hence, in most cases, the average coating thickness on an article will be greater than the specified value: how much greater is largely determined by the shape of the article (see Practice B 507) and the characteristics of the plating process.

In addition, the average coating thickness on articles will vary from article to article within a production lot. Therefore, if all of the articles in a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the average necessary to assure that a single article meets the requirement.

7.4 Adhesion—The palladium coatings shall be adherent to the substrate, when tested by one of the procedures summarized in 9.4.

7.5 Integrity of the Coating:
7.5.1 Gross Defects/Mechanical Damage—The coatings shall be free of visible mechanical damage and similar gross defects when viewed at magnifications up to 10X. For some applications this requirement may be relaxed to allow for a small number of such defects (per unit area), especially if they are outside of or on the periphery of the significant surfaces. See 7.5.2 and 6.5.4.

7.5.2 Porosity—Almost all as-plated electrodeposits contain some porosity, and the amount of porosity to be expected for any one type of coating will increase with decreasing the thickness of that particular coating type. The amount of porosity in the coating that may be tolerable depends on the severity of the environment that the article is likely to encounter during service or storage. If the pores are few in number, or away from the significant surfaces, their presence can often be tolerated. Acceptance or pass-fail criteria, if required, shall be part of the product specification for the particular article or coating requiring the porosity test. See 9.5.

NOTE 5—Extensive reviews of porosity and porosity testing can be found in the literature (4, 5).

8. Sampling
8.1 The sampling plan used for the inspection of a quality of the coated articles shall be as agreed upon between the purchaser and the supplier.

NOTE 6—Usually, when a collection of coated articles, the inspection lot (see 8.2), is examined for compliance with the requirements placed on the articles, a relatively small number of the articles—the sample—is selected at random and is inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria of
compliance are determined by the application of statistics. The procedure is known as sampling inspection. Test Method B 602, Guide B 697, and Test Method B 762 contain sampling plans that are designed for the sampling inspection of coatings.

Test Method B 602 contains four sampling plans, three for use with tests that are non-destructive and one when they are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 identifies the plan to be used.

Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and seller need to agree on the plan to be used.

Test Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numerical value and certain statistical requirements must be met. Test Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Test Method B 762 identifies the plan to be used.

8.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specifications, that have been coated by a single supplier at one time, or at approximately the same time, under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

9. Test Methods

9.1 Deposit Purity—Use any recognized method to determine qualitatively the impurities present. Atomic absorption spectrophotometry (or any other methods with demonstrated uncertainty less than 10 %) may be used to determine the metallic impurities. Initial scanning should be carried out for all elements, in order to detect any unknown or unexpected impurities. Determine deposit purity by subtracting total impurities from 100 %.

Note 7—Deposit purity is best determined on a special test specimen. One must be careful to arrange the specimen so as to electroplate at a typical density, similar to the production pieces. Palladium may be stripped by utilizing a 90 volume % (reagent grade) sulfuric, 10 % (reagent grade) nitric acid solution. The test specimen substrate should be platinum, gold, or an electrodeposit not attacked by the strip solution. The total palladium deposit should be over 100 mg and the sample weight is determined by a weigh-strip-weigh procedure. The strip solution is then used for quantitative analysis of impurities.

9.2 Appearance—The coating shall be examined at up to 10× magnification for conformance to the requirements of appearance.

9.3 Thickness—Measure thickness by methods outlined in Test Methods B 487, B 499, B 567, B 568, or B 748, or any other test method that has an uncertainty less than 10 %, or less than the test methods listed.

9.4 Adhesion—Determine adhesion by one of the following procedures (see Practice B 571 for full details):

9.4.1 Bend Test—Bend the electroplated article repeatedly through an angle of 180° on a diameter equal to the thickness of the article until fracture of the basis metal occurs. Examine the fracture at a magnification of 10×. Cracking without separation does not indicate poor adhesion unless the coating can be peeled back with a sharp instrument.

9.4.2 Heat Test—No flaking, blistering, or peeling shall be apparent at a magnification of 10× after the palladium-electroplated parts are heated to 300 to 350°C (570 to 660°F) for 30 min and allowed to cool.

9.4.3 Cutting Test—Make a cut with a sharp instrument and then probe with a sharp point and examine at a magnification of 10×. No separation of the coating from the substrate shall occur.

9.5 Plating Integrity—Porosity and microcracks shall be determined by either Test Methods B 741, B 799, or B 809 unless otherwise specified. Do not use the nitric acid vapor test (palladium can dissolve in nitric acid.)

Note the nature of the basis metal, the nature and thickness of any intermediate layers or underplate, and the shape of the palladium plated part. Guide B 765 is suitable to assist in the selection of porosity tests for electrodeposits of palladium alloys.

9.6 Ductility—When required, determine ductility in accordance with Practice B 489.

10. Special Government Requirements

10.1 The following special requirements shall apply when the ultimate purchaser is the U.S. Government or an agent of the U.S. Government.

10.1.1 Sampling—For government acceptance, the sampling plane specified in MIL-STD-105 is to be used instead of the ASTM standards specified in 8.1.

10.1.2 Thickness Testing:

10.1.2.1 In addition to the non-destructive methods outlined in Practice B 499 and Test Methods B 567 and B 568, a cross-sectioning method, such as that specified by Test Method B 487 or B 748, shall be used as a referee method to confirm the precision and bias of the particular non-destructive technique that is used.

10.1.2.2 The palladium thickness on significant surfaces shall be at least 1.3 µm (0.05 mil), unless otherwise specified on the drawings or in the contract. The coating on nonsignificant surfaces shall be of sufficient thickness to ensure plating continuity and uniform utility, appearance, and protection. The thickness of plating on nonsignificant surfaces, unless specifically exempted, shall be a minimum of 60 % of that specified for significant surfaces.

10.1.3 Packaging—The packaging and packing requirements shall be in accordance with Practice D 3951 or as specified in the contract or order. (Warning—Some contemporary packaging materials may emit fumes that are deleterious to the coating surface.)

11. Keywords

11.1 connectors; contacts; electrical connectors; electrical contacts; engineering coatings; palladium; palladium coatings; palladium electrodeposits; palladium platings
X1. SOME REASONS FOR USING A GOLD OVERPLATE

X1.1 A gold overplate is employed to enhance the performance of the palladium surface. Two types of gold are used:

X1.1.1 Type 1 gold is used in the critical areas in thickness ranges of 0.05 to 0.12 µm.

X1.1.2 Type 2 gold is used in the critical areas in thickness ranges of 0.05 to 0.25 µm or higher.

X1.2 The gold overplate offers the following performance enhancements to palladium:

X1.2.1 Durability—A gold overplate of proper thickness can reduce friction and enhance durability by providing a low shear-strength solid lubricant that reduces friction and wear (6,7). Type 1 gold should be used at a thickness no greater than 0.12 µm to maintain a low coefficient of friction. Palladium should not be mated against itself in a sliding contact pair when durability and resistance to fretting and frictional polymer formation is desired.

X1.2.2 Mating Force—Application of Type 1 or Type 2 gold can reduce friction and mating force. Type 1 should be no more than 0.12 µm thick.

X1.2.3 Fretting—Fretting occurs when two surfaces undergo low amplitude, repetitive motions. Depending on conditions and contact surface materials, fretting wear or fretting corrosion can occur. Fretting wear is loss of material along the wear track. Fretting corrosion is the formation of surface oxides at the contact surface. The addition of a Type 1 or Type 2 gold can often reduce fretting corrosion that is due to fretting motions (8). The occurrence of fretting is influenced greatly by contact design. See Terminology B 542.

X1.2.4 Frictional Polymerization—Frictional polymerization is the formation of insulating polymeric films at the contact spot. Such occurrences have been documented for palladium, palladium-nickel alloys and other metals (7). The addition of a Type 1 or Type 2 gold overplate can often reduce frictional polymer formation (8). (See Terminology B 542.)

X2. SOME REASONS FOR USING A NICKEL UNDERPLATE FOR PALLADIUM-NICKEL ELECTROPLATING

X2.1 Diffusion Barrier—To inhibit diffusion of copper from the basis metal into the palladium.

X2.2 Levelling Layer—To produce a smoother surface than the basis metal in order to ensure a lower porosity palladium top coat, for example, levelling nickel over a rough substrate.

X2.3 Pore Corrosion Inhibitor—A nickel underplate under the palladium top coat will form passive oxides at the base of pores in humid air, provided the environment does not contain significant amounts of acidic pollutants, such as SO₂ or HCl.

X2.4 Load Bearing Underlayer for Contacting Surfaces—A hard nickel underplate can serve as a load bearing foundation for the palladium top coat and reduce the wear of the precious metal during sliding of the contacting surfaces.

X2.5 For all of these purposes, the nickel underplating must be intact, that is, not cracked, and must have sufficient thickness to achieve the particular function for which it was intended. As a general rule, the minimum thickness should be 1.3 µm (50 µin.), preferably greater. For some levelling purposes, a greater thickness may be required.

X3. RESIDUAL SALTS

X3.1 Electroplated parts are placed in water of known conductivity and agitated for a specific time. The conductivity of the water extract is measured and the increase in conductivity due to residual salts and other conducting impurities is calculated. A suggested water extract conductivity test method uses a procedure in accordance with Test Methods D 1125, Method A.

X3.2 Conductivity of water for extract test shall be 1 µS/cm or less (resistivity 1 MΩ·cm or more).

X3.2.1 A sample of the coated parts having a total surface area of 30 cm² shall ordinarily be used and extracted in 100 cm³ of equilibrated water. To prepare equilibrated water, fill a clean polyethylene bottle half-way with high-purity water (X6.1), replace the bottle cap and shake the bottle vigorously for 2 min to equilibrate the water with the CO₂ in the air. CO₂ is a component of air, is soluble in water, and forms carbonic acid, which ionizes and is at equilibrium at 0.8 µS/cm. Slowly agitate the solution for 10 min before determining the conductivity of the extract. In a closed polyethylene bottle, the equilibrated water will remain in the range from 0.8 to 1 µS/cm for at least 1 week.

X3.3 Inspection under a source of ultraviolet light is often employed to determine whether electroplating salts have been removed by the rinsing following gold electroplating. The presence of salts is evidenced by a characteristic fluorescence and should not be confused with fluorescing dirt or dirt particles.

X3.4 Water or purging stains, resulting from blind holes or
from parts that were assembled before electroplating, as normally obtained in good commercial practice, are permissible except where they occur on surfaces to which electrical contact is to be made or on which subsequent soldering operations are performed.

**X4. RECOMMENDED THICKNESSES**

X4.1 Palladium thicknesses that have been recommended for specific applications are given in the following table.

<table>
<thead>
<tr>
<th>Thickness, µm</th>
<th>Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.08–0.25</td>
<td>Semiconductor Lead Frames in Integrated Circuity (9). Also solderable surfaces on Printed Wiring Boards</td>
</tr>
<tr>
<td>0.25–0.5</td>
<td>Catalysts. Also electrical contacts where little adverse environmental, electrical, or mechanical action is expected.</td>
</tr>
<tr>
<td>0.75–1.5</td>
<td>Low-energy electrical connector contacts.</td>
</tr>
<tr>
<td>2.5–5</td>
<td>Relay contacts with mechanical and electrical erosion.</td>
</tr>
</tbody>
</table>

**REFERENCES**


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1. Scope

1.1 This test method covers a test for the quality of seal of porous anodic coatings on aluminum and its alloys. It is based upon the loss in mass of the coating after immersion in a warm phosphoric-chromic acid solution.

1.2 This test method is applicable to anodic coatings intended for exposure to the weather, or for protective purposes in corrosive media, and where resistance to staining is important.

1.3 This test method is not applicable to:

1.3.1 Hard coatings, which normally are not sealed.

1.3.2 Anodic coatings that have been sealed only in dichromate solutions.

1.3.3 Anodic coatings that have undergone a treatment to render them hydrophobic.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 137 Test Method for Measurement of Coating Mass Per Unit Area on Anodically Coated Aluminum

D 1193 Specification for Reagent Water

2.2 International Standard:

ISO 3210 Anodizing of Aluminum and Its Alloys—Assessment of Sealing Quality by Measurement of the Loss of Mass After Immersion in Phosphoric-Chromic Acid Solution

3. Significance and Use

3.1 This test method describes a destructive test procedure for measuring the degree of seal of the porosity in anodic oxide coatings. Low coating mass loss is an indication of good seal quality and of the ability of the coating to resist staining and “blooming” in many types of service.

3.2 This test method is suitable for quality control purposes within manufacturing operations and for determining whether anodized parts meet seal quality requirements in applicable specifications.

4. Apparatus

4.1 Laboratory Balance, accurate to 1 mg.

4.2 Glass Container, means of heating and stirring, and a thermometer. There shall be no metal in contact with the test specimen or the solution.

5. Acid Test Solution

5.1 The test solution shall have the following makeup:

<table>
<thead>
<tr>
<th>Component</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chromic acid anhydride (CrO₃)₂</td>
<td>0.5 g</td>
</tr>
<tr>
<td>Orthophosphoric acid of 85 mass %, density 1.69</td>
<td>35 ± 0.5 mL</td>
</tr>
<tr>
<td>Distilled or deionized water conforming with Type III Reagent Water of Specification D 1193, to make up to 1000 mL</td>
<td></td>
</tr>
</tbody>
</table>

Note 1—This solution is commonly referred to as a “stripping solution” for anodic coatings and is of the same composition as that employed in Method B 137. This solution dissolves the anodic coating with no significant attack of the substrate metal.

5.2 The test solution may be used repeatedly but shall be discarded after 1 g of anodic coating has been dissolved per litre of solution.

Note 2—The solution may be used for as many as 20 test specimens per litre provided that the average mass loss does not exceed 50 mg per specimen.

6. Preparation of Test Specimen

6.1 Select an uncontaminated specimen having an area about 1 dm² of the anodized surface to be tested.

6.2 The mass of the specimen shall not exceed 200 g.

6.3 When it is desired to conduct the test on one face only of the specimen, the anodic coating on the other surface is removed by a chemical or mechanical process, leaving the significant face intact.

6.4 Bare, cut edges and other uncoated surfaces of the specimen shall be left bare and shall not be given a protective coating.
7. Procedure

7.1 Measure the area \((A)\) in square decimetres of the coating without including cut edges or other uncoated surfaces.

7.2 Weigh the specimen to the nearest 1 mg \((W_1)\).

7.3 Immerse the specimen for 15 ± 0.1 min in the stirred acid test solution maintained at a temperature of 38 ± 1°C.

7.4 Remove the specimen and wash thoroughly in deionized or distilled water conforming with Type III reagent water of Specification D 1193.

7.5 Dry the specimen with clean compressed air, or allow it to air-dry.

7.6 Reweigh the specimen to the nearest 1 mg \((W_2)\).

7.7 When it is desired to express the mass loss in the test as a percent of the total coating mass, use the above procedure followed by stripping the remainder of the oxide in the phosphoric-chromic acid solution in accordance with Method B 137.

7.8 Rinse and dry the specimen as above and reweigh it to the nearest 1 mg \((W_3)\).

8. Report

8.1 Report the mass loss per square decimetre calculated as follows:

\[
\text{Mass Loss, mg/dm}^2 = \frac{(W_1 - W_2)}{A} \quad (1)
\]

8.2 When required, report the mass loss in the test as a percent of the total coating mass, calculated as follows:

\[
\text{Coating Dissolved in Test, %} = \frac{W_1 - W_2}{W_1 - W_3} \times 100 \quad (2)
\]

9. Precision and Bias

9.1 The precision and accuracy of coating mass loss per unit area are most affected by the accuracy of measurement of the area of the anodized surface. When smooth, flat rectangular test specimens of sheet are used, the reproducibility of test results among experienced operators is usually within ±2 mg/dm², or ±10% of the arithmetic mean of the test values, whichever is greater.
Standard Specification for Electroplated Engineering Nickel Coatings

This standard is issued under the fixed designation B 689; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This specification covers the requirements for electroplated nickel coatings applied to metal products for engineering applications, for example, for use as a buildup for mismatched or worn parts, for electronic applications, including as underplates in contacts or interconnections, and in certain joining applications.

1.2 Electroplating of nickel for engineering applications (Note 1) requires technical considerations significantly different from decorative applications because the following functional properties are important:

1.2.1 Hardness, strength, and ductility,
1.2.2 Wear resistance,
1.2.3 Load bearing characteristics,
1.2.4 Corrosion resistance,
1.2.5 Heat scaling resistance,
1.2.6 Fretting resistance, and
1.2.7 Fatigue resistance.

Note 1—Functional electroplated nickel coatings usually contain about 99 % nickel, and are most frequently electrodeposited from a Watts nickel bath or a nickel sulfamate bath. Typical mechanical properties of nickel electroplated from these baths, and the combined effect of bath operation and solution composition variables on the mechanical properties of the electrodeposat are given in Guide B 832. When electroplated nickel is required to have higher hardnesses, greater wear resistance, certain residual stress values and certain leveling characteristics, sulfur and other substances are incorporated in the nickel deposit through the use of certain addition agents in the electroplating solution. For the effect of such additives, see Section 4 and Annex A3. Cobalt salts are sometimes added to the plating solution to produce harder nickel alloy deposits.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 252 Guide for Preparation of Zinc Alloy Die Castings for Electroplating and Conversion Coatings
B 253 Guide for Preparation of Aluminum Alloys for Electroplating
B 254 Practice for Preparation of and Electroplating on Stainless Steel
B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings
B 320 Practice for Preparation of Iron Castings for Electroplating
B 322 Practice for Cleaning Metals Prior to Electroplating
B 343 Practice for Preparation of Nickel for Electroplating with Nickel
B 374 Terminology Relating to Electroplating
B 480 Guide for Preparation of Magnesium and Magnesium Alloys for Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section
B 507 Practice for Design of Articles to Be Electroplated on Racks
B 530 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Electrodeposited Nickel Coatings on Magnetic and Nonmagnetic Substrates
B 558 Practice for Preparation of Nickel Alloys for Electroplating
B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 697 Guide for Selection of Sampling Plans for Inspection

Note 2 Annual Book of ASTM Standards, Vol 02.05.
of Electrodeposited Metallic and Inorganic Coatings
B 762 Test Method of Variables Sampling of Metallic and Inorganic Coatings
B 765 Guide for Selection of Porosity Tests for Electrodeposits and Related Metal Coatings
B 809 Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor ("Flowers of Sulfur")
B 832 Guide for Electroforming with Nickel and Copper
B 849 Specification for Pre-Treatments of Iron or Steel for Reducing Risk of Hydrogen Embrittlement
B 850 Guide for Post-Coating Treatments Steel for the Reducing Risk of Hydrogen Embrittlement
B 851 Specification for Automated Controlled Shot Peening of Metallic Articles Prior to Nickel, Autocatalytic Nickel, or Chromium Plating, or as Final Finish
D 1193 Specification for Reagent Water
D 3951 Practice for Commercial Packaging
F 519 Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments
2.2 Military Standards:
MIL-R-81841 Rotary Flap Peening of Metal Parts
MIL-S-13165 Shot Peening of Metal Parts
MIL-W-81840 Rotary Flap Peening Wheels
3. Terminology
3.1 Definitions:
3.1.1 significant surfaces—those surfaces normally visible (directly or by reflection) that are essential to the appearance or serviceability of the article when assembled in normal position; or that can be the source of corrosion products that deface visible surfaces on the assembled article. When necessary, the significant surfaces shall be indicated on the drawing for the article, or by the provision of suitably marked samples.

Note 2—The thickness of the electrodeposited nickel in holes, corners, recesses, and other areas where thickness cannot be controlled under normal electroplating conditions shall be specified by the buyer (see Note 5).

Note 3—When a deposit of controlled thickness is required in holes, corners, recesses, and similar areas, special racking, auxiliary anodes or shielding will be necessary.

3.2 Terminology B 374 contains most of the terms used in this specification.

4. Classification
4.1 Electroplated nickel shall be provided in any one of the following three types (Note 4):

4.1.1 Type 1—Nickel electroplated from solutions not containing hardeners, brighteners, or stress control additives.

4.1.2 Type 2—Nickel electrodeposits used at moderate temperatures and containing sulfur or other codeposited elements or compounds that are present to increase the hardness, to refine the grain structure, or to control the internal stress of the electrodeposited nickel.

4.1.3 Type 3—Electrodeposited nickel containing dispersed submicron particles, such as silicon carbide, tungsten carbide, and aluminum oxide that are present to increase hardness and wear resistance at temperatures above 325°C (618°F).

Note 4—Good adhesion of electroplated nickel to stainless steels and high alloy steels usually requires a preliminary strike of electrodeposited nickel. The recommended practices for the preparation of and electroplating on stainless steels and nickel alloys are given in Practices B 254 and B 558, respectively.

4.2 Thickness Classification—The electroplated nickel thickness, in view of the wide variety for industrial uses, shall be specified according to the following classes (Note 5):

<table>
<thead>
<tr>
<th>Class</th>
<th>Minimum Nickel Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>200</td>
<td>200</td>
</tr>
<tr>
<td>X</td>
<td>thickness as specified</td>
</tr>
</tbody>
</table>

Note 5—There is no technical limit to the nickel thickness that can be electroplated. There are practical limits to nickel thickness and uniformity of thickness distribution caused by the size and geometric configuration of the parts. (See 3.1.)

5. Ordering Information
5.1 The buyer shall supply the following information to the seller in either the purchase order or engineering drawings, marked samples, or other governing documents.

5.1.1 Title, ASTM designation number, and year of the standard.

5.1.2 Classification type and thickness classification of electroplated nickel to be applied (see 4.1 and 4.2).

5.1.3 Significant surfaces (see 3.1).

5.1.4 Sampling plan (see Section 8).

5.1.5 Number of test specimens for destructive testing (see 7.1). Identify the substrate material by alloy identification, such as by ASTM, AISI, or SAE numbers, or by equivalent composition information.

5.1.6 The thickness, adhesion, porosity, and hydrogen embrittlement tests required. See 6.3-6.7.

5.1.7 The required grinding or polishing operations of the basis metal as are necessary to yield deposit with the desired properties.

5.1.8 Where required, the basis metal finish shall be specified in terms of centerline average (CLA), or arithmetical average (AA).

5.1.9 Appearance: whether superficial staining from final rinsing or discoloration after baking is acceptable.

5.1.10 Where required, post-treatment grinding or machining shall be specified for parts which are to be electroplated and subsequently ground or machined to size.

5.1.11 Where required dimensional tolerances allowed for the specified electroplated nickel thickness or class shall be specified.

5.1.12 Where required, microhardness ranges shall be specified for the nickel deposit.

5.1.13 The buyer of the parts to be electroplated shall provide the electroplater with the following information as required:

5.1.13.1 Ultimate tensile strength of the parts.

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7 Annual Book of ASTM Standards, Vol 15.03.
8 Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn. NPODS.
5.1.13.2 Rockwell C hardness of the parts.
5.1.13.3 Heat treatment for stress relief, whether it has been performed or is required (see 6.2).
5.1.13.4 Heat treatment for hydrogen embrittlement relief (see 6.3 and Test Method F 519).
5.1.13.5 Tensile loads required for the embrittlement relief test, if applicable.
5.1.13.6 Procedures and requirements for peening to induce residual compressive stress in specified surfaces (see Note 6 and 6.4).

Note 6—Electroplating on hardened (high alloy and high carbon) steels can reduce the fatigue strength of the metal parts. This must be considered if the parts will be subjected to repeated applications of complex load patterns in service. Shot peening of significant surfaces before electroplating can reduce the loss of fatigue strength.7 Rotary flap peening, a manual method, can also be used in the repair of components in the field where conventional shot peening equipment is not available. If rotary flap peening is used, extreme care should be taken to ensure that the entire surface to be treated has been peened. Also, reduction in the fatigue life of nickel-electroplated steels can be reduced by considering the relations among the variables that influence fatigue life of nickel-electroplated, hardened steels.8

5.1.13.7 What, if any, mechanical treatment was applied by the manufacturer to the significant surface; that is, particulate blasting, grinding, polishing, or peening.
5.1.14 The manufacturer of the parts to be electroplated shall provide the electroplating facility with test specimens (see Section 7) to be electroplated for conformance tests as requested for preparation, control, inspection, and lot acceptance.

6. Coating Requirements

6.1 Appearance:
6.1.1 The coating on the significant surfaces of the product shall be smooth and free of visual defects such as blisters, pits, roughness, cracks, flaking, burned deposits, and uncoated areas. Visual defects are defined as those visible, unmagnified, to the unaided eye, 20/20 vision, or vision corrected to 20/20. The boundaries of electroplating that cover only a portion of the surface shall, after finishing as indicated in the drawing, be free of beads, nodules, jagged edges, and other detrimental irregularities. Imperfections and variations in appearance in the coating that arise from surface conditions of the basis metal (scratches, pores, roll marks, inclusions, etc.) and that persist in the finish despite the observance of good metal finishing practices shall not be cause for rejection (Note 7).

6.1.2 For parts that are electroplated and subsequently ground to size, the grinding shall be done with a sulfur-free liquid coolant, never dry, and with a sufficiently light cut to prevent cracking.

Note 7—Applied finishes generally perform better in service when the substrate over which they are applied is smooth and free of torn metal, inclusions, pores, and other defects. It is recommended that the specifications covering the unfinished product provide limits for these defects. A metal finisher can often remove defects through special treatments, such as grinding, polishing, abrasive blasting, chemical treatments, and electro-polishing. However, these are not normal in the treatment steps preceding the application of the finish. When they are desired they must be stated in the purchase order (see 5.1.7).

6.2 Pretreatment of Iron and Steel for Reducing the Risk of Hydrogen Embrittlement—Parts for critical applications that are made of steels with ultimate tensile strengths of 1000 MPa, hardness of 31 HRC or greater, that have been machined, ground, cold formed, or cold straightened subsequent to heat treatment, shall require stress relief heat treatment when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specification B 849 may be consulted for a list of pretreatments that are used widely.

6.3 Post-Coating Treatments of Iron and Steel for Reducing the Risk of Hydrogen Embrittlement—Parts for critical applications that are made of steels with ultimate tensile strengths of 1000 MPa, hardness of 31 HRC or greater, as well as surface hardened parts, shall require post coating hydrogen embrittlement relief baking when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specification B 850 may be consulted for a list of post treatments that are used widely.

6.4 Peening of Metal Parts—If peening is required before electroplating to induce residual compressive stress to increase fatigue strength and resistance to stress corrosion cracking of the metal parts, refer to Specification B 851 and to MIL-S-13165, MIL-R-81841, and MIL-W-81840.

6.5 Thickness—The thickness of the coating everywhere on the significant surface shall conform to the requirements of the specified class as defined in 3.2 (see Note 8 and 7.2).

Note 8—The coating thickness requirements of this specification are minimum requirements; that is, the coating thickness is required to equal or exceed the specified thickness on any significant surface (see 4.1). Variation in the coating thickness from point to point on a coated article is an inherent characteristic of the electroplating process. Therefore, the coating thickness will have to exceed the specified value at some points on the significant surfaces to ensure that the thickness equals or exceeds the minimum specified value at all points. Hence, in most cases, the average coating thickness on an article will be greater than the specified value; how much greater is largely determined by the shape of the article (see Practice B 507) and the characteristics of the electroplating process. In addition, the average coating thickness on articles will vary from article to article within a production lot. Therefore, if all of the articles in a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the average necessary to assure that a single article meets the requirement.

6.6 Adhesion—The coating shall be sufficiently adherent to the basis metal to pass the adhesion test specified (see 7.3).

6.7 Porosity—The coating shall be sufficiently free of pores to pass the porosity test specified (see 7.4).

6.8 Workmanship—Adding to (spotting-in) or double electroplating, unless evidence of a satisfactory bond is established, shall be cause for rejection (see 7.3). Parts having a hardness greater than 35 HRC (equivalent to a tensile strength of 1200 MPa or greater) that have been acid-stripped for recoating shall be rebaked for embrittlement relief (see 6.2) before electroplating. Stress relieving after stripping is not necessary if the stripping is done anodically in an alkaline solution. Within the

areas designated as significant surfaces there shall be no uncoated (or bare) areas (see 4.1). Contact marks shall be minimized in size and frequency. When contacts must be located on significant areas, they shall be placed in areas of minimum exposure to service or environmental conditions as designated by the purchaser. Superficial staining resulting from rinsing, or slight discoloration resulting from baking operations to relieve embrittlement shall not be cause for rejection unless specified to the contrary by the purchaser. (See 5.1.9.) Electrodeposited nickel that is to be finished by machining may have slight surface blemishes in the as-electroplated condition provided these can be eliminated by the machining operation.

6.9 Supplementary Requirements:
6.9.1 Packaging—If packaging requirements are to be met under this Specification, they shall be in accordance with Practice D 3951, or as specified in the contract or order.

NOTE 9—Some contemporary packaging materials may emit fumes that are deleterious to the surface of the coating.

7. Test Methods
7.1 Special Test Specimens:
7.1.1 The permission or the requirement to use special test specimens, the number to be used, the material from which they are to be made, and their shape and size shall be stated by the purchaser.

NOTE 10—Test specimens often are used to represent the coated articles in a test if the articles are of a size, shape, or material that is not suitable for the test, or if it is preferred not to submit articles to a destructive test because, for example, the articles are expensive or few in number. The specimen should duplicate the characteristics of the article that influence the property being tested.

7.1.2 Special test specimens used to represent articles in an adhesion, solderability, porosity, corrosion resistance, or appearance test shall be made of the same material, shall be in the same metallurgical condition, and shall have the same surface condition as the articles they represent, and they shall be placed in the production lot of and be processed along with the articles they represent.

7.1.3 Special test specimens used to represent articles in a coating thickness test may be made of a material that is suitable for the test method even if the represented article is not of the same material. For example, a low-carbon steel specimen may represent a brass article when the magnetic thickness test is used (Test Method B 499). The thickness specimen need not be carried through the complete process with the represented article. If not, it shall be introduced into the process at the point where the coating is applied and it shall be carried through all steps that have a bearing on the coating thickness. In rack plating, the specimen shall be racked in the same way with the same distance from and orientation with the anodes and other items in the process as the article it represents.

NOTE 11—When special test specimens are used to represent coated articles in a thickness test, the specimens will not necessarily have the same thickness and thickness distribution as the articles unless the specimens and the articles are of the same general size and shape. Therefore, before finished articles can be accepted on the basis of a thickness test performed on special test specimens, the relationship between the thickness on the specimen and the thickness on the part needs to be established. The criterion of acceptance is that thickness on the specimen that corresponds to the required thickness on the article.

7.2 Thickness—The thickness of the electroplated nickel shall be measured by one of the following methods.

Destructive Methods:
- Microscopical method B 487
- Coulometric method B 504

Nondestructive Methods:
- Magnetic method B 530
- X-Ray method B 568

If the accuracy of the thickness measurement is to be 10 % or less, then Test Method B 487 (Microscopical) should not be used for thicknesses less than 10 µm. Test Method B 530 (Magnetic) should not be used for thicknesses less than 10 µm. Test Method B 568 (X-ray) is instrument-dependent with regard to accuracy with increasing nickel thicknesses.

NOTE 12—Since many factors influence the accuracy of each thickness test method, the buyer is advised to review the test method being selected. Type 2 nickel coatings produced with certain organic additives can exhibit significant leveling properties on rough surfaces where less nickel is deposited on sharper points and more in depressions of a microprofile. Thickness measurements with the microscopical method can show large differences on these rough profiles.

7.3 Adhesion:
7.3.1 The coated article or designated test specimen shall pass one of the following tests, or any special test particular to the function of the part as specified by the purchaser:
7.3.1.1 Bend test,
7.3.1.2 File test,
7.3.1.3 Heat and quench test, or
7.3.1.4 Push test.

7.3.2 These and other adhesion tests are described in Test Methods B 571. The test selected should take into consideration the size, shape, or thickness of the part. Adhesion tests may at times fail to detect adhesion failure; subsequent fabrication may reveal poor or inadequate adhesion, which shall be cause for rejection.

NOTE 13—Adhesion is influenced by the method of pretreating the basis metal and the type of basis metal used. Helpful information is given in Practices B 183, B 242, B 252, B 253, B 254, B 281, B 320, B 322, and B 343, and Guide B 480.

7.4 Porosity—The coating shall pass one of the following tests as specified by the purchaser; aid in the selection is contained in Guide B 765.
7.4.1 Hot Water Porosity Test—Conduct according to the procedure described in Annex A1; observe the results after 60 min. The part fails if more than the number of pores specified by the purchaser per part or per unit area is found.
7.4.2 Ferroxyl Test—Conduct in accordance with the procedure described in Annex A2; observe the results after 10 min. The part fails if more than the number of pores specified by the purchaser per part of per unit area is found.
7.4.3 Flowers of Sulfur Test—The tests described in 7.4.1-7.4.3 are used for ferrous bases. Test Method B 809, the Flowers-of-Sulfur (or Humid Sulfur Vapor) test, can be used for nickel on copper and copper alloy substrates. Also refer to Guide B 765 for more information.
7.5 Hydrogen Embrittlement Relief—Parts shall be examined for cracks indicating embrittlement failure, or the effectiveness of the hydrogen embrittlement relief shall be determined by a procedure specified by the purchaser.

NOTE 14—When both destructive and non-destructive tests exist for the measurement of a characteristic, the purchaser needs to state which is to be used so that the proper sampling plan is selected. A test may destroy the coating but in a non-critical area; or, although it may destroy the coating, a tested part can be reclaimed by stripping and recoating. The purchaser needs to state whether the test is to be considered destructive or non-destructive.

8. Sampling Requirements

8.1 The sampling plan used for the inspection of a quantity of the coated articles shall be as agreed upon between the purchaser and the supplier.

NOTE 15—Usually, when a collection of coated articles, the inspection lot (8.2), is examined for compliance with the requirements placed on the articles, a relatively small number of the articles, the sample, is selected at random and is inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Three standards, Test Method B 602, Guide B 697, and Methods B 762 contain sampling plans that are designed for the sampling inspection of coatings.

Test Method B 602 contains four sampling plans, three for use with tests that are non-destructive and one when they are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 identifies the plan to be used.

Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and seller need to agree on the plan to be used.

9. Rejection and Rehearing

9.1 Articles that fail to conform to the requirements of this standard shall be rejected. Rejection shall be reported to the producer or supplier promptly, and in writing. In case of dissatisfaction with the results of a test, the producer or supplier may make a claim for a rehearing. Finishes that show imperfections during subsequent manufacturing operations may be rejected.

10. Certification

10.1 The purchaser may require in the purchase order or contract, that the producer or supplier give to the purchaser, certification that the finish was produced and tested in accordance with this standard and met the requirements. The purchaser may similarly require that a report of the test results be furnished.

ANNEXES

A1. HOT WATER POROSITY TEST

A1.1 General

A1.1.1 This method reveals discontinuities, such as pores, in electroplated nickel on iron or steel. It is noncorrosive to nickel.

A1.2 Materials

A1.2.1 A stainless steel (Type 304 or 316) or rubber-lined or glass vessel equipped to suspend the part that should be insulated from contact with metal vessels. The significant electroplated areas should be totally immersed in clean water that meets Specification D 1193, Type IV water standard or another type of water approved by the purchaser. The pH of the water shall be maintained between 6.0 and 7.5. Additives required for pH control shall be noncorrosive to nickel and shall be approved by the purchaser, for example, pH can be adjusted by introducing CO2 or by additions of H2SO4 or acetic acid, or NaOH. A source of oil-free air shall be available to aerate the water with agitation vigorous enough to prevent air bubbles from clinging to significant surfaces of the part.

NOTE A1.1—Ordinarily, common factory air supply does not meet the oil-free requirement.

Methods B 762 can be used only for coating requirements that have numerical limit, such as coating thickness. The test must yield a numerical value and certain statistical requirements must be met. Methods B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Methods B 762 identifies the plan to be used.

8.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specifications, that have been coated by a single supplier at one time, or at approximately the same time, under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

8.3 If special test specimens are used to represent the coated articles in a test, the number used shall be that required in 7.1.1.

A1.3 Procedure

A1.3.1 Clean and degrease the electroplated surface to be tested to provide a water break free surface. Totally immerse the electroplated areas of the part in the water which has been heated to 85°C. The 60-min test period starts when the water temperature is in equilibrium with the immersed part at 85 ± 3°C. This temperature shall be maintained during the 60-min test period. At the end of the test period, remove the part from the hot water, and allow the part to drain and dry. Oil-free air pressure may be used to speed the drying. Black spots and red rust indicate basis metal corrosion or porosity.

A1.4 Report

A1.4.1 The following information shall be included in the report:

A1.4.1.1 The area of surface tested.

A1.4.1.2 The total number and diameter of all spots visible to the unaided eye, and

A1.4.1.3 The highest number of spots visible within a square area as defined and specified by the purchaser.
A2. MODIFIED FERROXYL TEST

A2.1 General

A2.1.1 This method reveals discontinuities such as pores, in electroplated nickel on iron or steel.

Note A2.1—This test is slightly corrosive to nickel particularly if the test period is extended appreciably (3 min or more) beyond the 10-min period. The test is very sensitive to the superficial presence of iron, that is, blue spots can occur on an electrodeposited nickel surface that has been in sufficient contact with a piece of iron to leave a trace of the iron on the nickel surface.

A2.2 Materials

A2.2.1 Three solutions are required. Strips of “wet strength” filter are required.

A2.2.1.1 Solution A is prepared by dissolving 50 g of white gelatine and 50 g of sodium chloride in 1 L of warm (45°C) distilled water.

A2.2.1.2 Solution B is prepared by dissolving 50 g of sodium chloride and 1 g of a non-ionic wetting agent in 1 L of distilled water.

A2.2.1.3 Solution C is prepared by dissolving 10 g of potassium ferricyanide in 1 L of distilled water.

A2.3 Procedure

A2.3.1 Filter paper strips are immersed in solution A, which is kept sufficiently warm to keep the gelatine dissolved, and then allowed to dry. Just before use, immerse the dry filter paper strips in solution B just long enough to thoroughly wet all of the filter paper. Firmly press the filter paper against the thoroughly cleaned and degreased electroplated nickel surface to be tested. Allow 10 min contact time for the test period (see Annex A2). If the filter paper should become dry during the test, moisten again with solution B. Remove the papers at the end of the contact period, and place at once into solution C. Sharply defined blue markings will appear on the papers indicating basis metal corrosion or porosity.

A2.4 Report

A2.4.1 The following information shall be included in the report:

A2.4.1.1 The area of surface tested.

A2.4.1.2 The total number and diameter of all spots on the filter paper oriented to surface area tested.

A2.4.1.3 The highest number of spots visible within a square area as defined and specified by the purchaser.

A3. APPLICATIONS OF INDUSTRIAL ELECTROPLATED NICKEL

### TABLE A3.1 Improving Corrosion Resistance

<table>
<thead>
<tr>
<th>Nickel Electroplating Process and Deposit Thickness</th>
<th>Industry Sector or Specific Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Watts or sulfamate nickel used with or without additives to control deposit stress, ductility, porosity. Nickel thickness generally exceeds 5 µm. (^{10}) (^{(1, 2)})</td>
<td>As an undercoat for precious metals on electroplated parts. Electronic industry and optical reflectors</td>
</tr>
<tr>
<td>Nickel thickness generally exceeds 75 µm.</td>
<td>To maintain product purity of mildly corrosive materials and prevent staining from ferrous metal containers</td>
</tr>
<tr>
<td>Nickel thickness generally exceeds 125 µm.</td>
<td>Food processing equipment</td>
</tr>
<tr>
<td></td>
<td>Paper and pulp—drying cylinders and rolls</td>
</tr>
<tr>
<td></td>
<td>Textile—condenser and calender rolls</td>
</tr>
<tr>
<td></td>
<td>Soap and caustic processing—coils, pumps and pipe</td>
</tr>
<tr>
<td></td>
<td>Glass processing—Lehr rolls</td>
</tr>
<tr>
<td></td>
<td>Chemical and nuclear—externally and internally plated pipe, fittings, and other components</td>
</tr>
<tr>
<td></td>
<td>Automotive—hydraulic Rams, Cylinder liners, shock absorbers, etc.</td>
</tr>
<tr>
<td></td>
<td>Other—washers, fasteners, plastic extrusion disks, shaft, gears</td>
</tr>
</tbody>
</table>

\(^{10}\) The boldface numbers in parentheses refer to the list of references at the end of this specification.

### TABLE A3.2 Improving Wear Resistance and Fretting Corrosion

<table>
<thead>
<tr>
<th>Nickel Electroplating Process and Deposit Thickness</th>
<th>Industry Sector or Specific Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Processes proved useful include all-chloride, hard nickel, all-sulfate, and nickel sulfamate. Alloy processes include nickel-phosphorus and nickel-cobalt. Codeposition of particles in nickel matrices, for example, silicon carbide, mica, thorium ((1, 3, 4, 5))</td>
<td>Automotive—coatings on pistons, cylinder walls, rotary engine housing liners, gear shafts, drive shafts, pump rods, hydraulic pistons</td>
</tr>
<tr>
<td>Nickel thickness generally exceeds 125 µm.</td>
<td>Printing—cylinder rolls</td>
</tr>
<tr>
<td></td>
<td>Mining equipment—hydraulic pistons, shafts, pump rods, and cylinders</td>
</tr>
</tbody>
</table>
TABLE A3.3 Salvage and Repair

<table>
<thead>
<tr>
<th>Nickel Electroplating Processes and Deposit Thickness</th>
<th>Industry Sector or Specific Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Watts, modified Watts, or sulfamate nickel with anti-pitting controls</td>
<td>Heavy-duty machinery and tools—worn or mis-machined parts, shafts, splines, etc.</td>
</tr>
<tr>
<td>Nickel thickness determined by extent of repair or salvage required</td>
<td>Molds, dies, shafts, housings, and precision fitting of bearings, rings, collars, etc.</td>
</tr>
<tr>
<td>Selective electroplating</td>
<td></td>
</tr>
<tr>
<td>Nickel thickness tolerance of 1.2 µm achievable by experienced operators</td>
<td></td>
</tr>
</tbody>
</table>

REFERENCES


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Standard Specification for Coatings of Zinc Mechanically Deposited on Iron and Steel

This standard is issued under the fixed designation B 695; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This specification covers the requirements for a coating of zinc mechanically deposited on iron and steel basis metals. The coating is provided in several thicknesses up to and including 107 µm. The seven thickest classes are usually referred to as “mechanically galvanized.”

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

NOTE 1—The performance of this coating complies with the requirements of Specification A 153 and MIL-C-81562.

1.3 The values stated in SI units are to be regarded as the standard. The inch-pound equivalents of SI units may be approximate.

2. Referenced Documents

2.1 ASTM Standards:
- A 153 Specification for Zinc Coating (Hot–Dip) on Iron and Steel Hardware
- A 194/A194M Specification for Carbon and Alloy Steel Nuts for Bolts for High-Pressure and High-Temperature Service
- A 325 Specification for Structural Bolts, Steel, Heat Treated, 120/105 ksi Minimum Tensile Strength
- A 490 Specification for Heat-Treated, Steel Structural Bolts, 150 ksi Minimum Tensile Strength
- A 563 Specification for Carbon and Alloy Steel Nuts
- B 117 Practice for Operating Salt Spray (Fog) Apparatus
- B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
- B 242 Practice for Preparation of High-Carbon Steel for Electroplating
- B 322 Practice for Cleaning Metals Prior to Electroplating
- B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
- B 571 Test Methods for Adhesion of Metallic Coatings
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
- B 117 Practice for Operating Salt Spray (Fog) Apparatus
- B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
- B 242 Practice for Preparation of High-Carbon Steel for Electroplating
- B 322 Practice for Cleaning Metals Prior to Electroplating
- B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
- B 571 Test Methods for Adhesion of Metallic Coatings
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
- F 1470 Guide for Fastener Sampling for Specified Mechanical Properties and Performance Inspection

2.2 Military Standard:
- MIL-C-81562 Coating, Cadmium, Tin Cadmium and Zinc (Mechanically Deposited)

2.3 AISC Standard:
- Specifications for Structural Joints Using ASTM A 325 or A 490 Bolts

3. Classification

3.1 Classes—Zinc coatings are classified on the basis of thickness, as follows:

<table>
<thead>
<tr>
<th>Class</th>
<th>Minimum Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>110</td>
<td>107</td>
</tr>
<tr>
<td>80</td>
<td>81</td>
</tr>
<tr>
<td>70</td>
<td>69</td>
</tr>
<tr>
<td>65</td>
<td>66</td>
</tr>
<tr>
<td>55</td>
<td>53</td>
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<tr>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>40</td>
<td>40</td>
</tr>
<tr>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td>12</td>
<td>12</td>
</tr>
<tr>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>5</td>
<td>5</td>
</tr>
</tbody>
</table>

3.2 Types—Zinc coatings are identified by types on the basis of supplementary treatment required, as follows:

Type I—As coated, without supplementary treatment (Appendix X2.1).
Type II—With colored chromate conversion treatment (Appendix X2.2).

4. Ordering Information

4.1 To make the application of this standard complete, the purchaser should supply the following information to the seller in the purchase order or other governing document:

4.1.1 Class, including a maximum thickness, if appropriate, type, and for Type II, color and need for supplemental lubricant (3.1, 3.2, and 6.2.5),

4.1.2 Nature of substrate (for example, high-strength steel), need for stress relief (6.2.1), and cleaning precautions to be followed (6.2.2 and 6.2.3),

4.1.3 Significant surfaces (6.3),

4.1.4 Requirements for and methods of testing for one or more of the following, if required: need for and type of test specimens (8.1), thickness (6.3 and 8.3), adhesion (6.4 and 8.4), corrosion resistance (6.5 and 8.5), absence of hydrogen embrittlement, and the waiting period before testing and testing loads (6.6 and 8.6),

4.1.5 Inspection responsibility (Section 11) and sampling plan for each inspection criterion (Section 7), and

4.1.6 Requirements for certified report of test results (Section 10).

5. Workmanship

5.1 The coating shall be uniform in appearance and substantially free of blisters, pits, nodules, flaking, and other defects that can adversely affect the function of the coating. The coating shall cover all surfaces as stated in 6.3 including roots of threads, thread peaks, corners, recesses, and edges. The coating shall not be stained or discolored throughout to an extent that would adversely affect appearance as a functional requirement. However, superficial staining, that results from rinsing or drying, and variations in color or luster shall not be cause for rejection.

Note 2—The nature of the mechanical plating process is such that coatings characteristically will not be as smooth or as bright as some electroplated coatings.

6. Requirements

6.1 Appearance—The coating as deposited shall have a uniform silvery appearance, and a matte to medium-bright luster.

6.2 Process:

6.2.1 Stress-Relief Treatment—All steel parts that have an ultimate tensile strength of 1000 MPa and above and that contain tensile stresses caused by machining, grinding, straightening, or cold-forming operation shall be given a stress-relief heat treatment prior to cleaning and metal deposition. The temperature and time at temperature shall be 190 ± 15°C for a minimum of 3 h so that maximum stress relief is obtained without reducing the hardness below the specified minimum.

6.2.2 High-strength steels (which become embrittled when charged with hydrogen) and that have heavy oxide or scale shall be cleaned before application of the coating in accordance with Practice B 242. In general, nonelectrolytic alkaline, anodic-alkaline, and some inhibited acid cleaners are preferred to avoid the risk of producing hydrogen embrittlement from the cleaning procedure.

6.2.3 For low-carbon steels, see Practice B 183. Useful guidelines are also given in Practice B 322.

6.2.4 Mechanical deposition of zinc coatings shall consist, in general, of all of the steps listed below, and in the sequence as shown:

6.2.4.1 Preparation of the surface of the parts to be coated, by chemical (generally acidic) procedure to an extent that permits uniformly satisfactory results from subsequent steps.

6.2.4.2 Deposition of a thin metal coating, generally of copper, by immersion in appropriate chemical solutions, without the use of electric current. There are no thickness requirements for this coating.

6.2.4.3 Tumbling of the parts that have been treated according to 6.2.4.1 and 6.2.4.2 in a container with the following:

(i) the zinc metal to be deposited, in powder form;

(ii) impact media, which may be glass or other substances that are essentially inert to the chemicals of the deposition process. The function of this media is to aid in providing mechanical forces to drive the metal powder onto the substrate parts;

(iii) a “promoter” or “accelerator” which aids in the uniform deposition of the metal powder;

(iv) a liquid medium, generally water.

6.2.4.4 Separation of the parts from the solid and liquid media.

6.2.4.5 Rinsing.

6.2.4.6 Drying.

6.2.5 Supplementary Treatments:

6.2.5.1 Colored Chromate Conversion Treatments (Type II)—Colored chromate conversion treatment for Type II shall be done in a solution containing hexavalent chromium ions. This solution shall produce a bright or semi-bright continuous, smooth, protective film with a uniform color that may range from yellow through bronze and olive-drab to brown and black and that may be dyed to a desired color. Bright dips that do not contain salts that yield films containing hexavalent chromium ions are not permitted as treatments for producing Type II coatings.

6.2.5.2 Waxes, lacquers, or other organic coatings may be used to improve lubricity, and the need for them shall be supplied in the purchase order or other governing document (see 4.1.1). Supplemental lubrication treatments shall not be used to ensure conformance to the salt spray corrosion resistance requirements (see 8.5.4).

6.2.5.3 Lubrication of grade DH nuts processed in accordance with this specification and used with Specification A 325 high-strength bolts is a requirement of paragraph 6.5 of Specification A 325 and paragraph 4.8 of Specification A 563.

Note 3—Although not included in Specification A 194/A 194M, this provision should apply to mechanically galvanized A 194 2H nuts when supplied for use with Specification A 325 bolts.

Note 4—Specifications for structural joints using Specification A 325 or A 490 bolts references the use of lubricants on nuts to be used with Specification A 325 high-strength bolts and is found in the commentary on this RCSC (Research Council on Structural Connections of the Engineering Foundation) Specification, within the paragraphs entitled “Effect Of
Galvanizing Upon Torque Involved In Tightening” and “Shipping Requirements For Galvanized Bolts and Nuts,” published November 1985, page 30.¹

6.2.6 Surface Defects—Defects and variations in appearance in the coating that arise from surface conditions of the substrate (scratches, pores, roll marks, inclusions, etc.) and that persist in the finish despite the observance of good metal finishing practices shall not be cause for rejection.

NOTE 5—Applied finishes generally perform better in service when the substrate over which they are applied is smooth and free of torn metal, inclusions, pores, and other defects. It is recommended that the specifications covering the unfinished product provide limits for these defects. A metal finisher can often remove defects through special treatments, such as grinding, polishing, abrasive blasting, chemical treatments, and electropolishing. However, these are not normal in the treatment steps preceding the application of the finish. When desired they must be specified on the purchase order (4.1.2).

6.3 Thickness:

6.3.1 The thickness of the coating everywhere on the significant surfaces shall be at least that of the specified class as defined in 3.1.

6.3.2 Significant surfaces are defined as those normally visible (directly or by reflection) that are essential to the appearance or serviceability of the article when assembled in normal position; or that can be the source of corrosion products that deface visible surfaces on the assembled article. When necessary, the significant surfaces shall be indicated on the drawing for the article, or by the provision of suitably marked samples.

NOTE 6—The thickness of mechanically-deposited coatings varies from point-to-point on the surface of a product, characteristically tending to be thicker on flat surfaces and thinner at exposed edges, sharp projections, shielded or recessed areas, interior corners and holes, with such thinner areas often being exempted from thickness requirements.

6.3.3 When significant surfaces are involved on which the specified thickness of deposit cannot readily be controlled, the purchaser and manufacturer should recognize the necessity for either thicker or thinner deposits. For example, to reduce buildup in thread roots, holes, deep recesses, bases of angles, and similar areas, the deposit thickness on the more accessible surfaces will have to be reduced proportionately.

NOTE 7—The coating thickness requirement of this specification is a minimum requirement; that is, the coating thickness is required to equal or exceed the specified thickness everywhere on the significant surfaces. Variation in the coating thickness from point to point on a coated article is an inherent characteristic of mechanical deposition processes. Therefore, the coating thickness will have to exceed the specified value at some points on the significant surfaces to ensure that the thickness equals or exceeds the specified value at all points. Hence, in most cases, the average coating thickness on an article will be greater than the specified value; how much greater is largely determined by the shape of the article and the characteristics of the deposition process.

In addition, the average coating thickness on articles will vary from article to article within a production lot. Therefore, if all of the articles in a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the average necessary to ensure that a single article meets the requirement.

6.4 Adhesion—The zinc coating shall be sufficiently adherent to the basis metal to pass the tests specified in 8.4.

6.5 Corrosion Resistance:

6.5.1 The presence of corrosion products visible to the unaided eye at normal reading distance at the end of the specified test periods stated in Table 1 shall constitute failure, except that corrosion products at edges of specimens shall not constitute failure. Slight “whips” of white corrosion, as opposed to obvious accumulations, shall be acceptable.

NOTE 8—Mechanical deposition is exclusively a barrel-finishing process. It is recognized that mechanical deposition on parts may therefore produce surfaces that have a different characteristic from those on parts that are finished exclusively by racking. Similarly, corrosion testing of actual parts may produce different results from those on test panels. Salt spray requirements that are appropriate to indicate the technical quality with which a process is carried out may be impractical for acceptance of actual parts. In such cases the purchaser shall indicate his requirements on the purchase order (4.1.4).

NOTE 9—In many instances, there is no direct relation between the results of an accelerated corrosion test and the resistance to corrosion in other media, because several factors that influence the progress of corrosion, such as the formation of protective films, vary greatly with the conditions encountered. Therefore, the results obtained in the test should not, therefore, be regarded as a direct guide to the corrosion resistance of the tested materials in all environments where these materials may be used. Also, performance of different materials in the test cannot always be taken as a direct guide to the relative corrosion resistance of these materials in service.

6.5.2 On parts with Type II coatings, the greater number of hours for either white corrosion products or rust shall apply. For example, for Type II, Class 8, the test shall be continued until the 72-h requirement is met for white corrosion products; similarly, for Type II, Class 25, if no white corrosion products appear before 72 h, test shall be continued until the 192-h requirement for basis metal corrosion is met (8.5.2).

6.6 Absence of Hydrogen Embrittlement—Springs and other high-strength parts subject to flexure shall be held for a minimum of 48 h at room temperature after coating before being loaded, flexed, or used. Such high-strength steel parts shall be free of hydrogen embrittlement. When specified in the purchase order, freedom from embrittlement shall be determined by the test specified herein (4.1.4 and 8.6).

7. Sampling

7.1 The purchaser and producer are urged to employ statistical process control in the coating process. Properly performed, statistical process control will assure coated products of satisfactory quality and will reduce the amount of acceptance inspection. The sampling plan used for the inspection of the quality coated article shall be agreed upon between the purchaser and producer.

7.1.1 When a collection of coated articles (inspection lot, see 7.2) is examined for compliance with the requirements of the specification, the minimum requirements of Table 1 shall constitute acceptance. It is recognized that mechanical deposition on parts may therefore produce surfaces that have a different characteristic from those on parts that are finished exclusively by racking. Similarly, corrosion testing of actual parts may produce different results from those on test panels. Salt spray requirements that are appropriate to indicate the technical quality with which a process is carried out may be impractical for acceptance of actual parts. In such cases the purchaser shall indicate his requirements on the purchase order (4.1.4).

TABLE 1 Minimum Hours to Failure (White Corrosion Products and Red Rust for Mechanically Deposited Zinc Coatings on Iron and Steel)

<table>
<thead>
<tr>
<th>Type</th>
<th>White Corrosion</th>
<th>Red Rust</th>
</tr>
</thead>
<tbody>
<tr>
<td>Class</td>
<td>55–110</td>
<td>50</td>
</tr>
<tr>
<td>I</td>
<td></td>
<td></td>
</tr>
<tr>
<td>II</td>
<td>72</td>
<td>72</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Class</th>
<th>55–110</th>
<th>50</th>
<th>40</th>
<th>25</th>
<th>12</th>
<th>8</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>II</td>
<td>no requirement</td>
<td>300</td>
<td>250</td>
<td>192</td>
<td>96</td>
<td>56</td>
<td>38</td>
</tr>
</tbody>
</table>

¹ No requirement.
placed on the articles, a relatively small number of the articles (sample) is selected at random and is inspected. The inspection lot is then classified as complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria for compliance are determined by the application of statistics. The procedure is known as sampling inspection. Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for sampling inspection of coatings.

7.1.2 Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one when they are destructive. Test Method B 602 provides a default plan if one is not specified.

7.1.3 Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. Guide B 697 provides a default plan if one is not specified.

7.1.4 Test Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numeric value and certain statistical requirements must be met. Test Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. Test Method B 762 provides a default plan if one is not specified.

7.1.5 Guide F 1470 can be used for fasteners such as internally threaded, externally threaded and nonthreaded fasteners and washers. This guide provides for two plans: one designated the “detection process” and one designated the “prevention process.” The purchaser and producer shall agree on the plan to be used.

7.2 An inspection lot shall be defined as a collection of coated articles that are the same kind, that have been produced to the same specification, that have been coated by a single supplier at one time or approximately the same time, under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

8. Test Methods

8.1 Test Specimens:

8.1.1 Test specimens may be used to represent the coated articles in a test if the articles are of a size, shape, or material that is not suitable for the test, or if it is preferred not to submit articles to a destructive test because, for example, the articles are expensive or few. The permission or the requirement to use test specimens, their number, the material from which they shall be made, and their shape and size shall be stated in the purchase order or other governing document.

8.1.2 The test specimen shall duplicate those characteristics of the article that influence the property being tested, and it shall be processed with the article through those process steps that influence the property.

8.1.2.1 The test specimen used to represent an article in an adhesion, corrosion resistance, or appearance test shall be made of the same material, shall be in the same metallurgical condition, and shall have the same surface condition as the article it represents, and it shall be placed in the production lot of, and be processed along with, the article it represents.

8.1.2.2 A test specimen used to represent an article in a coating thickness test shall be introduced into the process at the point where the coating or coatings are applied and it shall be carried through all steps that have a bearing on the coating thickness.

8.1.2.3 When a test specimen is used to represent a coated article in a thickness test, the specimen will not necessarily have the same thickness and thickness distribution as the article unless the specimen and the article are of the same general size and shape. Therefore, before coated articles may be accepted on the basis of a thickness test performed on representative test specimens, the relationship between the thickness on the specimen and the thickness on the part shall be established. The criterion of acceptance shall be that thickness on the specimen that corresponds to the required thickness on the article.

8.2 Workmanship—Quality of workmanship shall be determined by the unaided eye at normal reading distance.

8.3 Thickness:

8.3.1 The thickness of the coating shall be determined by the microscopical method (Test Method B 487) or the magnetic method (Test Method B 499), as applicable. Other methods may be used if it can be demonstrated that the measurement uncertainty with these methods is less than 10%.

8.3.2 The thickness of the coating shall be measured at the location or locations for both significant and nonsignificant surfaces of the product where the coating would be expected to be the thinnest or at such locations as specified on the purchase order (4.1.3 and 6.3).

8.3.3 Thickness measurements of Type II deposits shall be made after application of the supplementary treatment. The Type II chromate conversion coatings shall be removed from the test area before the thickness is measured. Removal shall be done by using a very mild abrasive (such as a paste of levigated alumina or magnesium oxide) rubbed on gently with the finger.

NOTE 10—The process by which Type II coatings are produced dissolves a small amount of the zinc. For this reason the thickness requirement to be checked refers to the thickness of the deposit after the application of the Type II coatings.

8.4 Adhesion—Adhesion of the zinc deposit to the basis metal shall be tested in a manner that is consistent with the service requirements of the coated article. The ability to separate the coating from the substrate by peeling, as distinct from flaking caused by rupture of the deposit or of the basis metal, shall be evidence of failure. One of the following methods for determining adhesion shall be used:

8.4.1 The part shall be plastically deformed, if possible, to rupture as specified on the purchase order (4.1.4).

8.4.2 The surface of the coated article shall be scraped or sheared with a sharp edge, knife, or razor blade through the coating down to the basis metal and examined under 4× magnification.

NOTE 11—There is no single satisfactory test for evaluating the adhesion of mechanically deposited coatings. Those given above are widely used; however, other tests may prove more applicable in specific cases. Various qualitative methods are discussed in Test Methods B 571. A review of methods of measuring adhesion is given in the Proceedings, Amer. Electroplaters’ Soc., Vol 50.

8.5 Salt Spray Corrosion Resistance:  

10 For availability of this publication, contact American Electroplaters’ Society, 12644 Research Parkway, Orlando, FL 32826.
8.5.1 The 5% neutral salt spray (fog) test as defined in Practice B 117 shall be used.

8.5.2 If samples with Type II coatings are to be examined both for white corrosion products and for rust, separate samples may be used to determine the end point for white corrosion and for rust. This is to permit uninterrupted exposure for the longer of the two test periods required without having to wash specimens for examination, in accordance with Practice B 117.

8.5.3 Parts with Type II supplementary chromate film shall be aged at room temperature for 24 h before subjection to the salt spray test.

8.5.4 Parts with coatings of wax, etc., shall not be used as samples for corrosion testing for conformance to the requirements of 6.5.

8.6 Absence of Hydrogen Embrittlement:

8.6.1 Coated parts to be tested for the absence of embrittlement from cleaning shall be tested for brittle failure in accordance with a suitable method to be specified on the purchase order (4.1.4). The description of the method shall include the means of applying a load to the part, the stress or load level to be applied, the duration of the test, the waiting time that must elapse between deposition of the zinc and testing or use of the part, and the criterion of failure.

8.6.2 Parts that must conform to U.S. Government requirements shall be subjected to such loading conditions described above for 200 h minimum.

NOTE 12—It is recommended that tests for embrittlement involve subjecting parts to the specified operating conditions for at least 100 h (except as noted in 8.6.2). The stress level induced by the test and the waiting period prior to test depend upon many factors such as shape of the part, carbon content of the steel, hardness of the part, and stress level in use. Parts with a tensile strength of over 1000 MPa, for example, may require a 48-h waiting period; parts with lower tensile strength may require less than a 24-h waiting period. High-carbon steel parts or those cold-worked or heat-treated to tensile strengths of 1450 MPa or more, where these parts will be subjected to a sustained load in use, may require testing at loads specified by the purchaser or at 75% of the ultimate tensile strength.

9. Rejection and Rehearing

9.1 Materials that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of a test, the producer or supplier may make a claim for rehearing. Finishes that show imperfections during subsequent manufacturing operations may be rejected.

10. Certification

10.1 The purchaser may require in the purchase order or contract that the producer or supplier give to the purchaser certification that the finish was produced and tested in accordance with this specification and found to meet the requirements. The purchaser may similarly require that a report of the test results be furnished.


11.1 For parts processed for U.S. Government procurement, the producer or supplier shall be responsible for the performance of all inspection and test requirements specified herein. Except as otherwise specified in the contract or order, the producer or supplier may use his own or any other suitable facilities for the performance of the inspection and test requirements specified herein, unless disapproved by the purchaser. The purchaser shall have the right to perform any of the inspections and tests set forth in this specification where such inspections are deemed necessary to assure that material conforms to the prescribed requirements.

APPENDIXES

(Nonmandatory Information)

XI. CHARACTERISTICS

X1.1 Mechanical deposition in itself greatly reduces the risk of hydrogen embrittlement and is suitable for coating bores and recesses in many parts that cannot be conveniently electroplated (see Appendix X3).

X1.2 Zinc coatings are usually applied to provide corrosion resistance. The performance of a zinc coating depends largely on its thickness, the supplementary treatment if any, and the kind of environment to which it is exposed. The seven heaviest classes of coatings offer suitable alternatives to hot-dip galvanizing. The following data, based on widespread testing, may be used to compare the behavior of zinc in various atmospheres. The values are only indicative, because individual studies in various parts of the world have resulted in figures that vary widely from these averages.

<table>
<thead>
<tr>
<th>Atmosphere</th>
<th>Mean Corrosion Rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Industrial</td>
<td>5.6 µm (0.22 mil/year)</td>
</tr>
<tr>
<td>Urban nonindustrial or marine</td>
<td>1.5 µm (0.06 mil/year)</td>
</tr>
<tr>
<td>Suburban</td>
<td>1.3 µm (0.05 mil/year)</td>
</tr>
<tr>
<td>Rural</td>
<td>0.8 µm (0.03 mil/year)</td>
</tr>
<tr>
<td>Indoors</td>
<td>considerably less than 0.5 µm (0.01 mil/year)</td>
</tr>
</tbody>
</table>
X2. SPECIFIC TYPES

X2.1 Type I (plain zinc) is useful for lowest cost protection where early formation of white corrosion products is not detrimental. It is also used for higher temperature applications up to approximately 120°C where the effectiveness of chromates is greatly reduced.

X2.2 Type II (colored chromates)—Chromates that have a color (yellow, olive drab, bronze, etc.) are used to delay the appearance of white or red corrosion products on the plated article, or to provide a color desired by a customer for a specific purpose.

X3. HYDROGEN EMBRITTLEMENT

X3.1 A major advantage of mechanical deposition is that it does not produce hydrogen embrittlement in hardened steel during the coating process. However, pronounced embrittlement can be produced in certain cleaning processes. The mild degree of embrittlement that might result from following proper procedures with cleaning methods permitted in this specification normally is self-relieving within a day’s time at room temperature.
Standard Specification for Coatings of Cadmium Mechanically Deposited

This standard is issued under the fixed designation B 696; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 General—This specification covers the requirements for a coating of cadmium mechanically deposited on metal products. The coating is provided in various thicknesses up to and including 12 µm.

1.2 Mechanical deposition greatly reduces the risk of hydrogen embrittlement and is suitable for coating bores and recesses in many parts that cannot be conveniently electroplated (see Appendix X3).

1.3 Cadmium coatings are usually applied to provide engineering properties and corrosion resistance. The performance of a cadmium coating depends largely on its thickness and the kind of environment to which it is exposed. Without proof of satisfactory correlation, accelerated tests such as the salt spray (fog) test cannot be relied upon to predict performance in other environments, nor will these serve as comparative measures of the corrosion resistance afforded by coatings of different metals. Thus, although there is a marked superiority of cadmium coatings over zinc coatings of equal thickness in the salt spray test, this is often not the case under conditions of use, so that further testing in the service environment should be conducted.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific precautionary statements, see 1.5 and 1.6.

1.5 Precaution: Cadmium is toxic and must not be used in a coating for articles that can come into contact with food or beverages, or for dental or other equipment that can be inserted into the mouth. Consult appropriate agencies for regulations in this connection.

1.6 Precaution: Because of the toxicity of cadmium vapors and cadmium oxide fumes, cadmium-coated articles must not be used at temperatures of 320°C and above. They must not be welded, spot-welded, soldered, or otherwise strongly heated without adequate ventilation that will efficiently remove all toxic fumes.

2. Referenced Documents

2.1 ASTM Standards:
B 117 Practice for Operating Salt Spray (Fog) Apparatus
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 322 Practice for Cleaning Metals Prior to Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
F 1470 Guide for Fastener Sampling for Specified Mechanical Properties and Performance Inspection

3. Classification

3.1 Classes—Cadmium coatings are classified on the basis of thickness, as follows:

<table>
<thead>
<tr>
<th>Class</th>
<th>Minimum Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>12</td>
<td>12</td>
</tr>
<tr>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>5</td>
<td>5</td>
</tr>
</tbody>
</table>

3.2 Types—Cadmium coatings are identified by types on the basis of supplementary treatment required, as follows:

Type I—As coated without supplementary chromate treatment (Appendix X2.1).

Type II—With colored chromate conversion treatment (Appendix X2.2).

4. Ordering Information

4.1 To make the application of this standard complete, the purchaser should supply the following information to the seller in the purchase order or other governing document:

4.1.1 Class, including a maximum thickness, if appropriate,
Type, and for Type II, color and need for supplemental lubricant (see 3.1, 3.2, and 6.2.4.2),

4.1.2 Nature of substrate (for example, high-strength steel), needed for stress relief (6.2.1), and cleaning precautions to be followed 6.2.2 and 6.2.3),

4.1.3 Significant surfaces (6.3),

4.1.4 Requirements for and methods of testing for one or more of the following, if required: need for and type of test specimens (8.1), thickness (6.3 and 8.3), adhesion (6.4 and 8.4), corrosion resistance (6.5 and 8.5), absence of hydrogen embrittlement, and the waiting period before testing and testing loads (6.6 and 8.6),

4.1.5 Inspection responsibility (Supplementary Requirement SI) and sampling plan for each inspection criterion (Section 7).

4.1.6 Requirements for certified report of test results (Section 10).

5. Workmanship

5.1 The coating shall be uniform in appearance and free of blisters, pits, nodules, flaking, and other defects that can adversely affect the function of the coating. The coating shall cover all surfaces as stated in 6.3 including roots of threads, thread peaks, corners, recesses, and edges. The coating shall not be stained or discolored throughout to an extent that would adversely affect appearance as a functional requirement. However, superficial staining that results from rinsing or drying and variations in color or luster shall not be cause for rejection.

NOTE 1—The nature of the mechanical plating process is such that coatings characteristically will not be as smooth or as bright as some electroplated coatings.

6. Requirements

6.1 Appearance—The coating as deposited shall have a uniform silvery appearance, and a matte to medium-bright luster.

6.2 Process:

6.2.1 Stress-Relief Treatment—All steel parts that have ultimate tensile strength of 1000 MPa and above and that contain tensile stresses caused by machining, grinding, straightening, or cold-forming operation shall be given a stress relief heat treatment prior to cleaning and metal deposition. The temperature and time at temperature shall be 190±15°C for a minimum of 3 h so that maximum stress relief is obtained without reducing the hardness below the specified minimum. Generally, nonelectrolytic alkaline, anodic-alkaline, and some inhibited acid cleaners are preferred to avoid the risk of producing hydrogen embrittlement from the cleaning procedure. Useful guidelines are also given in Practice B 242.

6.2.2 High-strength steels that have heavy oxide or scale shall be cleaned before application of the coating in accordance with Practice B 242. In general, nonelectrolytic alkaline, anodic-alkaline, and some inhibited acid cleaners are preferred to avoid the risk of producing hydrogen embrittlement from the cleaning procedure.

6.2.3 For low-carbon steels see Practice B 183. Useful guidelines are also given in Practice B 322.

6.2.4 Supplementary Treatments:

6.2.4.1 Colored Chromate Conversion Treatments (Type II)—Chromate treatment for Type II shall be done in a solution containing hexavalent chromium. This solution shall produce a bright or semi-bright continuous, smooth, protective film with a uniform color that may range from yellow through bronze and olive drab to brown and black including olive drab and that may be dyed to a desired color. Post treatments that do not contain salts that yield films containing hexavalent chromium are not permitted as treatments for producing Type II coatings.

6.2.4.2 Waxes, lacquers, or other organic coatings may be used to improve lubricity, and the need for them shall be supplied in the purchase order or other governing document (4.1.1). Supplemental lubrication treatment shall not be used to ensure conformance to the salt spray corrosion resistance requirements.

6.2.5 Surface Defects—Defects and variations in appearance in the coating that arise from surface conditions of the substrate (scratches, pores, roll marks, inclusions, etc.) and that persist in the finish despite the observance of good metal finishing practices shall not be cause for rejection.

NOTE 2—Applied finishes generally perform better in service when the substrate over which they are applied is smooth and free of torn metal, inclusions, pores, and other defects. It is recommended that the specifications covering the unfinished product provide limits for these defects. A metal finisher can often remove defects through special treatments, such as grinding, polishing, abrasive blasting, chemical treatments, and electropolishing. However, these are not normal in the treatment steps preceding the application of the finish. When desired they must be specified on the purchase order (4.1.2).

6.3 Thickness:

6.3.1 The thickness of the coating everywhere on the significant surfaces shall be at least that of the specified class as defined in 3.1.

6.3.2 Significant surfaces are defined as those normally visible (directly or by reflection) that are essential to the appearance or serviceability of the article when assembled in normal position; or that can be the source of corrosion products that deface visible surfaces on the assembled article. When necessary, the significant surfaces shall be indicated on the drawing for the article, or by the provision of suitably marked samples.

NOTE 3—The thickness of mechanically-deposited coatings varies from point-to-point on the surface of a product, characteristically tending to be thicker on flat surfaces and thinner at exposed edges, sharp projections, shielded or recessed areas, interior corners and holes, with such thinner areas often being exempted from thickness requirements.

6.3.3 When significant surfaces are involved on which the specified thickness of deposit cannot readily be controlled, the purchaser and manufacturer should recognize the necessity for either thicker or thinner deposits. For example, to reduce buildup in thread roots, holes, deep recesses, bases of angles, and similar areas, the deposit thickness on the more accessible surfaces will have to be reduced proportionately.

NOTE 4—The coating thickness requirement of this specification is a minimum requirement; that is, the coating thickness is required to equal or exceed the specified thickness everywhere on the significant surfaces. Variation in the coating thickness from point to point on a coated article is an inherent characteristic of mechanical deposition processes. Therefore, the coating thickness will have to exceed the specified value at some points on the significant surfaces to ensure that the thickness equals or exceeds the specified value at all points. Hence, in most cases, the average coating thickness on an article will be greater than the specified value; how much greater is largely determined by the shape of the article and the characteristics of the deposition process. In addition, the average coating thickness on articles will vary from article to article within a production
6.4 Adhesion—The cadmium coating shall be sufficiently adherent to the basis metal to pass the tests specified in 8.4.

6.5 Corrosion Resistance:

6.5.1 The presence of corrosion products visible to the unaided eye at normal reading distance at the end of the specified test periods stated in Table 1 shall constitute failure, except that corrosion products at edges of specimens shall not constitute failure. Slight “wisps” of white corrosion, as opposed to obvious accumulations, shall be acceptable.

**NOTE 5—**The hours given in Table 1 are the minimums required to guarantee satisfactory performance. Longer periods before the appearance of white corrosion products and rust are possible, but salt spray resistance does not vary in exact proportion with increased plating thickness. The hours given for Type II reflect the added protection of chromate treatments without requiring impractical testing periods.

6.5.2 There are no requirements for corrosion of basis metals other than steels.

**NOTE 6—**Mechanical deposition is exclusively a barrel-finishing process. It is recognized that mechanical deposition on parts may therefore produce surfaces that have a different characteristic from those on parts that are finished exclusively by racking. Similarly, corrosion testing of actual parts may produce different results from those on test panels. Salt spray requirements that are appropriate to indicate the technical quality with which a process is carried out may be impractical for acceptance of actual parts. In such cases the purchaser should indicate his requirements on the purchaser order (4.1.4).

**NOTE 7—**In many instances, there is no direct relation between the results of an accelerated corrosion test and the resistance to corrosion in other media, because several factors that influence the progress of corrosion, such as the formation of protective films, vary greatly with the conditions encountered. The results obtained in the test should not, therefore, be regarded as a direct guide to the corrosion resistance of the tested materials in all environments where these materials may be used. Also, performance of different materials in the test cannot always be taken as a direct guide to the relative corrosion resistance of these materials in service.

6.5.3 On parts with Type II coatings, the greater number of hours for either white corrosion products or rust shall apply. For example, for Type II, Class 5, the test shall be continued until the 72-h requirement is met for white corrosion products; similarly, for Type II, Class 8, if no white corrosion products appear before 72 h, the test shall be continued until the 96-h requirement for basis metal corrosion is met (8.5.2).

6.6 Absence of Hydrogen Embrittlement—Steel springs and other high-strength steel parts subject to flexure shall be held for a minimum of 48 h at room temperature after coating before being loaded, flexed, or used. Such high-strength steel parts shall be free of hydrogen embrittlement. When specified in the purchase order, freedom from embrittlement shall be determined by the test specified herein (4.1.4 and 8.6).

7. Sampling

7.1 The purchaser and producer are urged to employ statistical process control in the coating process. Properly performed, statistical process control will assure coated products of satisfactory quality and will reduce the amount of acceptance inspection. The sampling plan used for the inspection of the quality coated article shall be agreed upon between the purchaser and producer.

7.1.1 When a collection of coated articles (inspection lot, see 7.2) is examined for compliance with the requirements placed on the articles, a relatively small number of the articles (sample) is selected at random and is inspected. The inspection lot is then classified as complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria for compliance are determined by the application of statistics. The procedure is known as sampling inspection. Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for sampling inspection of coatings.

7.1.2 Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one when they are destructive. Test Method B 602 provides a default plan if one is not specified.

7.1.3 Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. Guide B 697 provides a default plan if one is not specified.

7.1.4 Test Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numeric value and certain statistical requirements must be met. Test Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. Test Method B 762 provides a default plan if one is not specified.

7.1.5 Guide F 1470 can be used for fasteners such as internally threaded, externally threaded and nonthreaded fasteners and washers. This guide provides for two plans: one designated the “detection process” and one designated the “prevention process.” The purchaser and producer shall agree on the plan to be used.

7.2 An inspection lot shall be defined as a collection of coated articles that are the same kind, that have been produced to the same specification, that have been coated by a single supplier at one time or approximately the same time, under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

8. Test Methods

8.1 Test Specimens:

8.1.1 Test specimens may be used to represent the coated articles in a test if the articles are of a size, shape, or material that is not suitable for the test, or if it is preferred not to submit articles to a destructive test because, for example, the articles are expensive or few. The permission or the requirement to use...
test specimens, their number, the material from which they shall be made, and their shape and size shall be stated in the purchase order or other governing document.

8.1.2 The test specimen shall duplicate those characteristics of the article that influence the property being tested, and it shall be processed with the article through those process steps that influence the property.

8.1.2.1 The test specimen used to represent an article in an adhesion, corrosion resistance, or appearance test shall be made of the same material, shall be in the same metallurgical condition, and shall have the same surface condition as the article it represents, and it shall be placed in the production lot of, and be processed along with, the article it represents.

8.1.2.2 A test specimen used to represent an article in a coating thickness test shall be introduced into the process at the point where the coating or coatings are applied and it shall be carried through all steps that have a bearing on the coating thickness.

8.1.2.3 When a test specimen is used to represent a coated article in a thickness test, the specimen will not necessarily have the same thickness and thickness distribution as the article unless the specimen and the article are of the same general size and shape. Therefore, before coated articles may be accepted on the basis of a thickness test performed on representative test specimens, the relationship between the thickness on the specimen and the thickness on the part shall be established. The criterion of acceptance shall be that thickness on the specimen that corresponds to the required thickness on the article.

8.2 Workmanship—Quality of workmanship shall be determined by the unaided eye at normal reading distance.

8.3 Thickness:

8.3.1 The thickness of the coating shall be determined by the microscopical method (Test Method B 487) or the magnetic method (Test Method B 499) or the beta backscatter method (Test Method B 567), as applicable. Other methods may be used if it can be demonstrated that the measurement uncertainty with these methods is less than 10%.

8.3.2 The thickness of the coating shall be measured at the location or locations of the product where the coating would be expected to be the thinnest or at such locations as specified on the purchase order (4.1.3 and 6.3).

8.3.3 Thickness measurements of Type II deposits shall be made after application of the supplementary treatment.

8.4 Adhesion—Adhesion of the cadmium deposit to the basis metal shall be tested in a manner that is consistent with the service requirements of the coated article. The ability to separate the coating from the substrate by peeling, as distinct from flaking caused by rupture of the deposit or of the basis metal shall be evidence of failure. One of the following methods for determining adhesion shall be used:

8.4.1 The part shall be plastically deformed, if possible, to rupture as specified on the purchase order (4.1.4).

8.4.2 The surface of the coated article shall be scraped or sheared with a sharp edge, knife, or razor blade through the coating down to the basis metal and examined under 4× magnification.

8.5 Salt Spray Corrosion Resistance:

8.5.1 The 5% neutral salt spray (fog) test as defined in Test Method B 117 shall be used.

8.5.2 If samples with Type II coatings are to be examined both for white corrosion products and for rust, separate samples may be used to determine the end point for white corrosion and for rust. This is to permit uninterrupted exposure for the longer of the two test periods required without having to wash specimens for examination, in accordance with Practice B 117.

8.5.3 Parts with Type II supplementary chromate film shall be aged at room temperature for 24 h before subjection to the salt spray test.

8.5.4 Parts with coatings of wax, etc., shall not be used as samples for corrosion testing for conformance to the requirements of 6.5.

8.6 Absence of Hydrogen Embrittlement:

8.6.1 Coated parts to be tested for the absence of embrittlement from cleaning shall be tested for brittle failure in accordance with a suitable method to be specified on the purchase order (4.1.4). The description of the method shall include the means of applying a load to the part, the stress or load level to be applied, the duration of the test, the waiting time that must elapse between deposition of the cadmium and testing or use of the part, and the criterion of failure.

8.6.2 Parts that must conform to government requirements shall be subjected to conditions described above for 200 h minimum.

8.7 Adhesion Tests:

8.7.1 The test shall be performed by shearing a strip from the coated part and from the basis metal to which the coating was applied. The test shall be carried through all steps that have a bearing on the coating thickness.

8.7.2 Adhesion shall be determined by one of the methods given in the following notes, and shall have the same surface condition as the article from which it is taken.

NOTE 8—The process by which Type II coatings are produced dissolves a small amount of cadmium. For this reason the thickness requirement to be checked refers to the thickness of the deposit after the application of the Type II coatings.

NOTE 9—The thickest chromate film (olive drab) is approximately 1.5 µm thick. If this significantly affects the accuracy of the thickness measuring method used (for example Test Method B 567), the Type II coatings shall be removed from the test area before the thickness is measured. Removal shall be done by using a very mild abrasive (such as a paste of levigated alumina or magnesium oxide) rubbed gently with the finger.

8.4.2 The surface of the coated article shall be scraped or sheared with a sharp edge, knife, or razor blade through the coating down to the basis metal and examined under 4× magnification.

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8.4 Adhesion—Adhesion of the cadmium deposit to the basis metal shall be tested in a manner that is consistent with

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8.5.3 Parts with Type II supplementary chromate film shall be aged at room temperature for 24 h before subjection to the salt spray test.

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8.6.1 Coated parts to be tested for the absence of embrittlement from cleaning shall be tested for brittle failure in accordance with a suitable method to be specified on the purchase order (4.1.4). The description of the method shall include the means of applying a load to the part, the stress or load level to be applied, the duration of the test, the waiting time that must elapse between deposition of the cadmium and testing or use of the part, and the criterion of failure.

8.6.2 Parts that must conform to government requirements shall be subjected to conditions described above for 200 h minimum.

NOTE 10—There is no single satisfactory test for evaluating the adhesion of mechanically deposited coatings. Those given above are widely used; however, other tests may prove more applicable in specific cases. Various qualitative methods are discussed in Polleys, R. W., ‘‘The Adhesion Testing of Electroplated Coatings. A review of methods of measuring adhesion is given in the Proceedings, Amer. Electroplaters’ Soc., Vol. 50, 1963, p. 54.’’ Application of pressure-sensitive tape to the coating is a method which may be misleading. Metal particles adhering to the tape may only reflect rinsing technique. Distinctions must be made between superficial surface effects which will not adversely affect other requirements, and internal coating consolidation (cohesion), or adhesion to the basis metal or undercoating.

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8.5.1 The 5% neutral salt spray (fog) test as defined in Test Method B 117 shall be used.

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8.6.2 Parts that must conform to government requirements shall be subjected to conditions described above for 200 h minimum.

NOTE 11—It is recommended that tests for embrittlement involve subjecting parts to the specified operating conditions for at least 100 h (except as noted in 8.6.2). The stress level induced by the test and the waiting period prior to test depend upon many factors such as shape of the part, carbon content of the steel, hardness of the part, and stress level in


6 For availability of this publication, contact American Electroplaters’ Society, 12644 Research Parkway, Orlando, FL 32826.
use. Parts with a tensile strength of over 1000 MPa, for example, may require a 48-h waiting period; parts with lower tensile strength may require less than a 24-h waiting period. High-carbon steel parts or those cold-worked or heat-treated to tensile strengths of 1450 MPa or more, where these parts will be subjected to a sustained load in use, may require testing at loads specified by the purchaser or at 75% of the ultimate tensile strength.

9. Rejection and Rehearing

9.1 Materials that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of a test, the producer or supplier may make a claim for rehearing. Finishes that show imperfections during subsequent manufacturing operations may be rejected.

10. Certification

10.1 The purchaser may require in the purchase order or contract that the producer or supplier give to the purchaser certification that the finish was produced and tested in accordance with this specification and found to meet the requirements. The purchaser may similarly require that a report of the test results be furnished.

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirement shall apply only when specified by the purchaser as part of the purchaser’s order or contract, and for all agencies of the United States Government.

S1. Responsibility for Inspection

S1.1 The producer or supplier is responsible for the performance of all inspection and test requirements specified herein. Except as otherwise specified in the contract or order, the producer or supplier may use his own or any other suitable facilities for the performance of the inspection and test requirements specified herein, unless disapproved by the purchaser. The purchaser shall have the right to perform any of the inspections and tests set forth in this specification where such inspections are deemed necessary to assure that material conforms to prescribed requirements.

APPENDIXES

(Nonmandatory Information)

X1. PROCESS

X1.1 Mechanical deposition of cadmium coatings should consist, in general, of all of the steps listed below, and in the sequence as shown:

X1.2 Preparation of the surface of the parts to be coated, by chemical (generally acidic) means, to an extent that permits uniformly satisfactory results from subsequent steps.

X1.3 Deposition of a thin metal coating where applicable, generally of copper, by immersion in appropriate chemical solutions, without the use of electric current. For ferrous-based metals this thin coating is generally copper. There are no thickness requirements for this coating.

X1.4 Tumbling of the parts that have been treated according to X1.2 and X1.3 in a container with the following:

(i) the cadmium metal to be deposited, in powder form;
(ii) impact media, which may be glass or other substances that are essentially inert to the chemicals of the deposition process. The function of this media is to aid in providing mechanical forces to drive the metal powder onto the substrate parts;
(iii) a “promotor” or “accelerator” which aids in the uniform deposition of the metal powder;
(iv) a liquid medium, generally water.

X1.5 Separation of the parts from the solid and liquid media.

X1.6 Rinsing.

X1.7 Drying.
X2. SPECIFIC TYPES

X2.1 *Type I (Plain Cadmium)* is useful for lowest cost protection where early formation of white corrosion products is not detrimental. It is also used for higher temperature applications up to approximately 120°C where the effectiveness of chromates is greatly reduced.

X2.2 *Type II (Colored Chromates)*—Chromates that have a color (yellow, olive drab, bronze, etc.) are used to delay the appearance of white corrosion and thereby to prolong the protection afforded by the cadmium, or to provide a color desired by a customer for a specific purpose.

X3. HYDROGEN EMBRITTLEMENT

X3.1 A major advantage of mechanical deposition is that it does not produce hydrogen embrittlement in hardened steel during the coating process. However, pronounced embrittlement can be produced in certain cleaning processes. The mild degree of embrittlement that might result from following proper procedures with cleaning methods permitted in this specification normally is self-relieving within a day’s time at room temperature.
Standard Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings

This standard is issued under the fixed designation B 697; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This standard gives guidance in the selection of sampling plans to be used in the inspection of electrodeposited and related coatings on products for the purpose of deciding whether submitted lots of coated products comply with the specifications applicable to the coatings. This supplements Test Method B 602 by giving more information on sampling inspection and by providing additional sampling plans for the user who finds the limited choice of plans in Test Method B 602 to be inadequate.

1.2 When using a sampling plan, a relatively small part of the articles in an inspection lot is selected and inspected. Based on the results, a decision is made that the inspection lot either does or does not satisfactorily conform to the specification.

1.3 This standard also contains several sampling plans. The plans are attribute plans, that is, in the application of the plans each inspected article is classified as either conforming or nonconforming to each of the coating requirements. The number of nonconforming articles is compared to a maximum allowable number. The plans are simple and relatively few. Additional plans and more complex plans that cover more situations are given in the Refs (1-7) at the end of this guide and in MIL-STD 105.

1.4 Acceptance sampling plans are used:

1.4.1 When the cost of inspection is high and the consequences of accepting a nonconforming article are not serious.

1.4.2 When 100 % inspection is fatiguing and boring and, therefore, likely to result in errors. In these cases a sampling plan may provide greater protection than 100 % inspection.

1.4.3 When inspection requires a destructive test. Here, sampling inspection must be used.

1.5 Another general type of acceptance sampling plan that is not covered in these guidelines is the variables plan in which measured values of characteristics are analyzed by statistical procedures. Such plans, when applicable, can reduce inspection cost and increase quality protection. Information on variables plans is given in Method B 762, MIL-STD-414, ANSI/ASQC Z1.9-1979, and Refs (1-2).

2. Referenced Documents

2.1 ASTM Standards:

B 602 Test Method of Attribute Sampling of Metallic and Inorganic Coatings

B 762 Method of Variables Sampling of Metallic and Inorganic Coatings

2.2 ANSI Standard:

ANSI/ASQC Z1.9–1979 Sampling Procedures and Tables for Inspection by Variables for Percent Nonconformance

2.3 Military Standards:

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes

MIL-STD-414 Sampling Procedures and Tables for Inspection by Variables for Percent Defective

3. General

3.1 Procedure—The use of acceptance sampling consists of a series of decisions and actions. These are listed in order below and are discussed in this standard.

3.1.1 Select characteristics to be inspected,

3.1.2 Select type of sampling plan,

3.1.3 Select quality level,

3.1.4 Define inspection lot,

3.1.5 Select sample,

3.1.6 Inspect sample,

3.1.7 Classify inspection lot, and

3.1.8 Dispose of inspection lot.

3.2 The need for acceptance sampling arises when a decision must be made about what to do with a quantity of articles. This quantity (called the inspection lot in this guide) may be a shipment from a supplier, may be articles that are ready for a subsequent manufacturing operation, or may be articles ready for shipment to a customer.

3.3 When acceptance sampling is done, several of the articles in the inspection lot are selected at random (see Section 7). These articles constitute the sample. Each article in the sample is inspected for conformance to the requirements placed on it. If an article meets a requirement, it is classified as conforming. If not, it is classified as nonconforming. If the...
number of nonconforming articles in the sample is no more than a predetermined number (called the acceptance number), the inspection lot is accepted. If it exceeds the acceptance number, the inspection lot is rejected.

3.4 The disposition of rejected inspection lots is beyond the scope of this guide because, depending on the circumstances, lots may be returned to the supplier, kept and used, put to a different use, scrapped, reworked, or dealt with in some other way. An exception is rectifying inspection (3.11) in which rejected lots are screened and used.

3.5 Because the decision about the disposition of an inspection lot is based on the inspection of a sample, and because there is a chance that a sample will not be representative of an inspection lot, some inspection lots that have the desired quality level (Note 1) will be rejected and some inspection lots that do not have the desired quality level will be accepted. There are only two situations in which the results of acceptance sampling are totally predictable (Note 2). One is when there are no nonconforming articles in the inspection lot. There, of course, will be no nonconforming articles in the sample and the decision to accept the lot will always be made. The other situation is when no article in the inspection lot conforms. All of the articles in the sample will be nonconforming and the decision to reject the lot will always be made (Note 3).

NOTE 1—In this guide the term “quality level” means the percentage of nonconforming articles in an inspection lot or it means the average percentage of nonconforming articles in a series of inspection lots received from a single source. Terms such as high quality, increased quality, and better quality mean a relatively smaller percentage of nonconforming articles, while terms such as low quality, decreased quality, and poorer quality mean a relatively larger percentage of nonconforming articles.

NOTE 2—In this discussion and elsewhere in this guide, it is assumed that no errors are made.

NOTE 3—To be strictly correct, lots that contain no more nonconforming articles than the acceptance number will always be accepted, and lots that contain fewer conforming articles than the sample size minus the acceptance number will always be rejected.

3.6 The discussion in 3.5 leads to two important points: (1) acceptance sampling plans will permit the acceptance of inspection lots that contain nonconforming articles and (2) in a series of inspection lots, each containing the same percentage of nonconforming articles, some will be accepted and some will be rejected, and the percentage of nonconforming articles in the accepted inspection lots will be the same as in the rejected lots. In other words, acceptance sampling does not, by itself, result in higher quality. Rectifying inspection (3.11) will result in higher average quality in the product leaving inspection.

3.7 Because acceptance sampling plans permit the acceptance of inspection lots that contain nonconforming articles, basic to the selection of a sampling plan is a decision about the percentage of nonconforming articles that is acceptable. If the function of the article is so important that no nonconformers can be tolerated, acceptance sampling cannot be used. In these cases, every article must be inspected, and, to guard against error, may have to be inspected twice.

3.8 The protection that an attributes sampling plan provides against accepting an undesirable number of nonconforming articles is determined by the size of the sample and by the acceptance number. The protection provided by a plan is usually expressed in the form of an operating characteristic (OC) curve. Fig. 1 is the OC curve for the plan that calls for a sample of 55 articles and an acceptance number of two. Plotted along the horizontal axis is the quality level of an inspection lot expressed as the percentage of the articles in the lot that are nonconforming (Note 1). The vertical axis is the probability, as a percentage, that an inspection lot will be accepted by the plan (Note 4). Inspection lots with zero percent nonconforming articles will be accepted 100 % of the time (Note 2). As the percentage of nonconforming articles in the inspection lot increases, the probability of acceptance decreases. For example, as shown in Fig. 1, an inspection lot containing 1.5 % nonconforming articles has a 95 % chance of being accepted, while one containing 9.6 % nonconforming articles has only a 10 % chance of being accepted.

NOTE 4—The vertical axis of the OC curve can have two meanings. One is the probability that a particular inspection lot will be accepted. The other meaning is the percentage of a series of lots of a given quality level that will be accepted. The latter meaning is the one that is strictly correct mathematically. The former meaning is also correct, as long as the inspection lot is at least ten times bigger than the sample.

3.9 The characteristics of a sampling plan are often expressed in terms of the Acceptable Quality Level (AQL) and the Limiting Quality Level (LQL). The AQL is the quality level that will result in the acceptance of a high percentage of incoming inspection lots; usually it is the quality level that will result in the acceptance of 95 % of the incoming inspection lots. In Fig. 1, the AQL is 1.5 %. The LQL is the quality level that will result in the rejection of a high percentage of incoming inspection lots; usually it is the quality level that will result in the rejection of 90 % of the incoming inspection lots. In Fig. 1 the LQL is 9.6 %. In this standard, AQL and LQL are defined as the quality levels that will be accepted 95 and rejected 90 % of the time, respectively.

3.10 Another characteristic of sampling plans that is used in this standard is the 50/50 point. This is the quality level that will result in the acceptance of half of the incoming inspection lots. In Fig. 1 the 50/50 point is 4.8 %.

![FIG. 1 Operating Characteristic Curve for Single Sample, Attributes Sampling Plan, Sample Size = 55, Acceptance Number = 2](image-url)
3.11 Rectifying Inspection:

3.11.1 As stated in 3.4, one of the options when an inspection lot is rejected is screening of the lot. In this procedure, called rectifying inspection, all of the articles in a rejected lot are inspected and the nonconforming ones are removed and replaced with conforming articles. The now 100 % conforming inspection lot is accepted and is passed along with the inspection lots that were accepted on the basis of acceptance sampling. The addition of these 100 % conforming inspection lots improves the average quality level of all the inspection lots taken together. The amount the quality level is improved can be calculated if the average quality level of incoming inspection lots is known. The calculations reveal that if the incoming quality level is high, few inspection lots will be rejected and screened and so the average quality of the outgoing lots will be only slightly improved over the incoming. If the quality level of the incoming inspection lots is low, many of the inspection lots will be rejected and screened. The addition of this large number of 100 % conforming lots will result in a high outgoing quality level. At intermediate incoming quality levels, the outgoing quality will be poorer than these two extremes, and there will be a particular incoming quality level for which the outgoing level will be the poorest.

3.11.2 When rectifying inspection is used the average quality level of a series of outgoing lots is called the Average Outgoing Quality (AOQ) and the worst possible AOQ for a given plan is called the Average Outgoing Quality Limit (AOQL). Fig. 2 is a plot of the AOQ for the sampling plan of Fig. 1 (Note 5). This shows that the worst AOQ, the AOQL, is 2.5 % and occurs only if the average incoming quality level is 4.2 %. Fig. 2 also shows that when the quality level of incoming lots is high, the improvement caused by inspection is small. For example, if the incoming lots are of AQL quality, 1.5 %, the AOQ is 1.4 %. At lower incoming quality levels the relative improvement is greater; for example, at an incoming quality level of 3 %, the AOQ is 2.3 %.

NOTE 5—The AOQs and AOQLs in this guide are calculated on the basis that when rejected lots are screened the nonconforming articles found are replaced with conforming articles. If the discarded nonconforming articles are not replaced, a practice that is frequently done, the AOQs and AOQLs will be somewhat different from those in this guide. Chapter 16 of Ref (4) discusses this point.

3.11.3 Use of rectifying inspection will assure that with a continuous series of inspection lots the average quality level of all the accepted articles, considered as a whole, will not be worse than the AOQL of the sampling plan used. However, rectifying inspection can significantly increase inspection costs since every rejected inspection lot is 100 % inspected. The lower the quality of incoming lots, the more of them that will be rejected and then 100 % inspected. Fig. 3 shows how, for the sampling plan of Fig. 1 and lots of 550, the average number of articles inspected per inspection lot increases as the quality levels of incoming lots decrease. In lots containing up to about 1.5 % nonconforming articles the increase in inspection is moderate. Beyond that point the average amount of inspection increases rapidly. At an incoming quality level of 2.1 % the amount of inspection is doubled. And with incoming quality levels of 15 % virtually every inspection lot is 100 % inspected.

3.11.4 Because the cost of inspection using rectifying inspection plans is so greatly influenced by the quality level of incoming inspection lots, past information of that level is necessary before choosing an AOQL. The AOQL plans in Table 1 give the range of incoming quality level for which each plan is recommended. The cost of the inspection is also determined by the size of the inspection lot and by the size of the sample. If rectifying inspection is to be used on a large scale, it is recommended that the user refer to Ref (3). It contains plans that yield the lowest total inspection for each combination of AOQL, incoming quality level, and inspection lot size.

3.11.5 Whether the 100 % inspection of rejected lots is done by the purchaser or the supplier is a business decision of the purchaser. Having the supplier do the inspection provides an incentive to improve the quality of future lots. However, if the supplier does the 100 % inspection, the purchaser may want to do sampling inspection of screened lots. This adds even more to the cost of inspection.

3.11.6 Rectifying inspection, of course, cannot be used if the inspection methods destroy the inspected articles.

3.12 AQL and LQL plans with an acceptance number of zero are not included in this guide because their operating characteristics are different from plans that have acceptance numbers of one or more. They can provide LQL protection.
against the bad inspection lot but only at the cost of rejection of a large number of good lots. Or, if they are selected on a basis of the AQL, they will allow the acceptance of a large number of bad lots. Fig. 4 illustrates this. The OC curves of three plans are shown. Curve number 2 is the plan shown in Fig. 1, a sample of 55 and an acceptance number of two. Curve number 1 is the zero acceptance number plan that has the same AQL, 1.5 %, as curve number 2. The sample size is 3. But this plan has an LQL of 54 % as compared to 9.7 % for curve number 2. Curve number 3 is the zero acceptance number plan that has the same LQL as curve number 2. The sample size is 23. With this plan the AQL is 0.2 % as compared to 1.5 % for curve number 2.

4. Selection of the Type of Sampling Plan

4.1 The sampling plans of this guide are given in Table 1, Table 2, and Table 3. All are single sampling plans, that is, the decision is based on the results with a single sample. Each table contains several sets of plans. Within each set the plans have one characteristic in common. In Table 1 all of the plans in a set have the same AQL. In Table 2 they have the same LQL. And in Table 3 the plans in each set have the same AOQL.

4.2 Plans based on the AQL (Table 1) are usually selected when there is a continuing series of inspection lots from a single source. The AQL value selected is the quality level that the purchaser considers to be satisfactory. The supplier knows that if he operates his finishing process so that the average quality level of his output is as good as or better than the AQL, 95 % or more of his submitted lots will be accepted. The cost to the supplier of high rejection rates if the quality of his output is much worse than the AQL provides motivation to improve the quality. When AQL plans are used, the specific AQL chosen is often a mutual decision of the purchaser and the seller.

4.3 Plans are based on the LQL (Table 3) when the purchaser considers it important to be protected against accepting an individual bad inspection lot, the bad inspection lot being defined as one of LQL quality.

4.4 AOQL plans are usually used when a series of lots from

### TABLE 1 Constant AQL Plans

<table>
<thead>
<tr>
<th>AQL</th>
<th>Sample Size</th>
<th>Acceptance Number</th>
<th>50/50 Point</th>
<th>LQL</th>
<th>AOQL</th>
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<tbody>
<tr>
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<tr>
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<td>1.7</td>
<td>3.2</td>
<td>0.92</td>
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<td>1.3</td>
<td>1.9</td>
<td>0.73</td>
</tr>
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<td>15</td>
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</tr>
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<td>84</td>
<td>6</td>
<td>7.9</td>
<td>12</td>
<td>4.5</td>
</tr>
</tbody>
</table>

Note 1—The values listed in columns headed AQL, 50/50 Point, LQL, and AOQL are the percentages of nonconforming articles in the inspection lot.

Note 2—The AQL values are calculated for inspection lots that are very large compared to the sample. The values can be corrected for cases where this is not true by multiplying them by:

\[
1 - \frac{\text{sample size}}{\text{lot size}}
\]

### TABLE 2 Constant LQL Plans

<table>
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<tr>
<th>LQL</th>
<th>Sample Size</th>
<th>Acceptance Number</th>
<th>AQL</th>
<th>50/50 Point</th>
<th>AOQL</th>
</tr>
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<td>2.8</td>
<td>1.5</td>
</tr>
<tr>
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<td>0.97</td>
<td>4.5</td>
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<td>7.0</td>
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<tr>
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<td>2.4</td>
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<td>6.7</td>
</tr>
</tbody>
</table>

Note 1—The values listed in columns headed AQL, 50/50 Point, LQL, and AOQL are the percentages of nonconforming articles in the inspection lot.

Note 2—The AOQL values are calculated for inspection lots that are very large compared to the sample. The values can be corrected for cases where this is not true by multiplying them by:

\[
1 - \frac{\text{sample size}}{\text{lot size}}
\]
a single source are accumulated to form a large stock of product. The AQL plans assure that the quality level of the accumulated stock is not worse than the AOQL. Such plans are more commonly used internally by a manufacturer to inspect the output of a manufacturing process.

5. Selection of a Specific Plan

5.1 After the type of plan (AQL, LQL, or AOQL) is selected, a specific plan must be selected. Several factors need be taken into consideration.

5.2 The cost of inspection should be balanced against the cost that results from the acceptance of nonconforming product. In the absence of other considerations the plan chosen should be the one that yields the lowest total cost. Often a smaller sample will be used when a test method is destructive because destruction of the tested articles increases the cost of inspection. The fact that a test is destructive does not by itself, however, justify using smaller samples. It is just one of the factors in the analysis leading to the selection of the sampling plan.

5.3 Often larger samples are used with larger inspection lots because, even though the absolute cost of inspection is higher with larger samples, the inspection cost per unit of product is less (6.5). The larger samples give better discrimination between acceptable and unacceptable inspection lots. This better discrimination may be desirable with large lots because the consequences of the decision are greater. It must be kept in mind that changing the sample size changes the characteristics of the plan. With a constant AQL plan (Table 1), increasing the sample size decreases the 50/50 point and the LQL, yielding better overall protection to the purchaser. But with a constant LQL plan (Table 2) increasing the sample size increases the AQL and the 50/50 point, yielding poorer overall protection to the purchaser.

5.4 The quality history of a supplier is a consideration. If an AQL plan is used and experience shows that a supplier consistently produces at the AQL then the AQL plan with the smallest sample size may be the best choice. For example, from Table 1 using an AQL of 1.5 %, the plan with a sample size of 24 and acceptance number of one would be appropriate. With a new supplier an intermediate plan might be initially selected and then adjusted up or down the table based on the experience with the first several shipments. If a supplier has a very good quality history, it may be safe to inspect only some of the inspection lots. Whether to inspect a specific lot should be decided by chance, for example, by a coin flip.

5.5 It may be safe to accept incoming lots without inspection if a nonconforming article will be recognized at a subsequent step in manufacturing, at which step as the operator finds nonconforming articles, he sets them aside for disposition. An example is the appearance of a decorative finish. Such a practice should be provided for in the purchase contract.

5.6 Different sample size and acceptance number plans can be used for the several requirements that a finish must meet. For example, in some applications conformance to a thickness requirement may be more important than conformance to an appearance requirement. In such a case a tighter plan might be used for thickness and a looser one for appearance. However, the increased administrative cost of using two or more sample sizes may be greater than the savings.

5.7 The inherent quality level of the finishing process must be considered so that unreasonable quality demands are not made.

5.8 The selection of the sampling plan or sampling plans should be a part of the contract between the purchaser and the seller. When a sampling plan is used by an organization to inspect the output of its captive finishing department, the plans used should be arrived at in consultation with the management of the finishing department.

6. Selection of the Inspection Lot

6.1 It is important that the inspection lot be homogeneous. This means that the articles in the inspection lot are from a single supplier, from a single finishing line, and ones that were finished at the same time in one batch or produced during a single period of time.

6.2 There are several reasons homogeneity is necessary in inspection lots:

6.2.1 If lots of different quality levels are mixed before inspection, the resulting larger lot will have an average quality level that is between the best and the worst of the lots before they were mixed. This average quality will be the best that will be realized if the lot is accepted. If, however, the individual lots are inspected separately, the better ones will probably be accepted and the poorer ones rejected, so the average quality level of the accepted lots will be higher than had they been mixed.

6.2.2 If lots from different sources are combined before inspection and the combined lot is then rejected, it cannot be

---

### TABLE 3 Constant AOQL Plans

<table>
<thead>
<tr>
<th>AOQL</th>
<th>Quality Level of Process</th>
<th>Sample Size</th>
<th>Acceptance Number</th>
<th>AQL</th>
<th>50/50 Point</th>
<th>LQL</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.65</td>
<td>0.0–0.1</td>
<td>57</td>
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<td>0.09</td>
<td>1.2</td>
<td>4.1</td>
</tr>
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<td></td>
<td>0.1–0.4</td>
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<td>0.28</td>
<td>1.3</td>
<td>3.0</td>
</tr>
<tr>
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<td>0.4–0.6</td>
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<td>6.2</td>
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<td>0.42</td>
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</tr>
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<td>15</td>
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<td>11</td>
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</tbody>
</table>
6.3 It is generally best for inspection lots to be as large as possible provided they are homogeneous. With larger lots larger samples can be used. This gives better discrimination between acceptable and unacceptable lots at lower unit costs. For example, assume a 1.5 % AQL plan is used (Table 1). If inspection lots contain 1000 articles and a sample of 55 is selected, the LQL will be 9.4 %, and 5.5 % of the articles will be inspected. If the inspection lots are larger, say 2000 articles, and a sample of 92 is used, the LQL is reduced to 7.1 % and the percentage of articles inspected is reduced to 4.6 % (5.3).

7. Sampling

7.1 Once the sampling plan is selected and the inspection lot is formed, the sample is drawn from the lot. It is essential that the articles in the sample be selected at random. This means that the selection of an article is purely by chance and that the chance of selection is the same for every article regardless of whether it is at the top or at the bottom of the lot, whether it looks good or looks bad, whether it is in reach or not, etc.

7.2 If the articles in a lot are thoroughly mixed as, for example, barrel electroplated articles, a sample drawn anywhere from the lot will meet the requirement of randomness. If the articles are not mixed, and if it is impractical to mix them, bias will result if the sample is drawn from a single or a few layers. Other bias in sampling, such as taking articles from the same place on a plating rack, taking articles from the output of one electroplating tank and not others, and taking articles that appear to be conforming or to be nonconforming, must be avoided. Bias can be avoided by numbering the articles, randomly selecting a group of numbers equal to the sample size, and inspecting the articles with the selected numbers. Ways of doing this are described in the following.

7.3 When random numbers are used to select a sample, each article in the lot is identified by a different number. This may be done by placing the articles in racks or trays where the positions in the racks or trays are numbered. If the articles have serial numbers, these can be used. The numbers of the articles that are to be inspected are selected randomly from a table of random numbers, such as Table 4. Other tables can be found in books dealing with statistics. Also, there are pocket calculators that can generate random numbers.

7.4 As an example, assume that a sample of 13 articles is to be selected from an inspection lot of 80 articles. The articles are numbered 1 through 80. Table 4 then can be used this way. Write the numbers 1 through 14 on 14 slips of paper, and write the numbers 1 through 61 on 61 other slips. Put each group into a container and blindly draw one slip from each group. The number drawn from the group of 14 determines the column number in Table 4. The number from the group of 61 is the row. Assume column 4 and row 30 are drawn. Going to Table 4, the number is found to be 94 305. To decide whether to read up or down the table, flip a coin, with heads the decision to go up and with tails, down. Assume the coin flip is tails. The numbers are chosen as follows: Consider only the first two digits of each group of five because the inspection lot contains 80 articles and only two-digit numbers are needed. Numbers larger than 80 are rejected and numbers that appear more than once are rejected after the first time. The samples of 13 articles, then, if found to be: 77, 56, 55, 12, 30, 49, 78, 64, 46, 67, 7, 29, 31.

7.5 When product articles are arranged in an order without regard to quality, such as in a tray, a sample can be drawn by using a constant-interval procedure. Here, a constant interval is maintained between the articles drawn for the sample. For example, every 9th, 16th, or 24th unit is selected. The first articles drawn from the lot can be determined from the table of random numbers. All other articles are then drawn at a constant interval following the first one. The constant interval is determined by dividing the inspection lot size by the sample size. Care must be taken to guard against a cyclic condition in the lot. For example, if the articles are received packed individually in a box, the articles in the corners of the box may be of poorer quality because of rough handling. If articles are rack-plated and are removed from the plating rack in a fixed sequence, constant interval sampling may result in selecting articles from the same location on the rack.

7.6 As an example of constant-interval sampling, assume that an inspection lot of 3000 articles is to be visually examined for freedom from blisters, pits, nodules, porosity, and staining. A sample of 126 is to be drawn. The constant interval is 23 (3000 divided by 126 is 23.8; round down to 23). A random number from 1 to 23 is selected either from a table (for example, Table 4) or by another appropriate method. After the first article is taken, the remaining articles in the required sample are drawn by selecting every 23rd article from the inspection lot until 126 are selected.

8. Inspection of the Sample

8.1 Each article in the sample is inspected from conformance to each of the finishing specifications and each article is classified as either conforming or nonconforming with respect to each requirement.

8.2 For each requirement in turn, articles that are nonconforming to the requirement are counted. For each requirement that the number of nonconforming articles is no more than the acceptance number of the sampling plan, the inspection lot is accepted with respect to that requirement. If for any requirement the number of nonconforming articles is more than the acceptance number, the lot is rejected with respect to that requirement.

8.3 Note that the inspection lot is classified as accepted or rejected with respect to each requirement separately. Thus if, for example, a plan is used with a sample size of 55 and acceptance number of 2, and if in inspection one article is found nonconforming with respect to coating thickness, another with respect to coating adhesion, and a third with respect to appearance, the inspection lot is accepted because although three articles are nonconforming, no more than one is nonconforming with respect to any one requirement. If, on the other hand, three articles are nonconforming with respect to a single requirement, say thickness, the lot is rejected.
9. Disposition of Lot

9.1 An inspection lot that is acceptable with respect to all requirements, is accepted.

9.2 An inspection lot that is unacceptable with respect to one or more of the requirements, is rejected. If the sampling plan used is an AQL or an LQL plan, the buyer must decide what to do with the lot (3.4).

9.3 If an inspection lot is rejected with respect to one or more of the requirements and an AQL sampling plan is being used, all of the articles are inspected for the requirement or requirements for which the inspection lot was rejected. All nonconforming articles are removed from the inspection lot, replaced with conforming articles, and the lot is then accepted (Note 5).
REFERENCES

(7) ibid, Vol 5, March 1977 pp. 8–12.

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Standard Specification for Coatings of Cadmium Vacuum-Deposited on Iron and Steel\textsuperscript{1}

This standard is issued under the fixed designation B 699; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (\(\epsilon\)) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers the requirements for a cadmium coating vacuum-deposited on iron and steel basis metals. The coating is especially beneficial to those ferrous metals, heat treated to 46HRC and higher or having an ultimate tensile strength greater than 1500 MPa, wherein protection against corrosion and appearance are important (see Appendix X1).

1.2 Vacuum-deposited production items are normally free of hydrogen embrittlement, a danger when using electroplating processes for deposition. Vacuum deposition can handle large high-strength parts that cannot be conveniently mechanically plated in the rotating barrels.

1.3 The coating is provided in various thicknesses up to and including 12 µm (3.1) either in the as-deposited condition or with a supplementary finish (3.2).

1.4 The following precautionary caveat pertains only to the test methods portion, Section 9, of this specification: This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 117 Practice for Operating Salt Spray (Fog) Apparatus\textsuperscript{2}

B 183 Practice for Preparation of Low-Carbon Steel for Electroplating\textsuperscript{3}

B 201 Practice for Testing Chromate Coatings on Zinc and Cadmium Surfaces\textsuperscript{3}

B 242 Practice for Preparation of High-Carbon Steel for Electroplating\textsuperscript{3}

B 254 Practice for Preparation of and Electroplating on Stainless Steel\textsuperscript{3}

B 320 Practice for Preparation of Iron Castings for Electroplating\textsuperscript{3}

B 322 Practice for Cleaning Metals Prior to Electroplating\textsuperscript{3}

B 440 Specification for Cadmium\textsuperscript{4}

B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section\textsuperscript{2}

B 499 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals\textsuperscript{3}

B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method\textsuperscript{3}

B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method\textsuperscript{3}

B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry\textsuperscript{3}

B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings\textsuperscript{3}

B 580 Test Method for Attribute Sampling of Metallic and Inorganic Coatings\textsuperscript{3}

B 602 Test Method for Qualitative Adhesion Testing of Metallic and Inorganic Coatings\textsuperscript{3}

B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings\textsuperscript{3}

E 396 Test Methods for Chemical Analysis of Cadmium\textsuperscript{5}

F 519 Method for Mechanical Hydrogen Embrittlement of Plating Processes and Service Environments\textsuperscript{6}

3. Classification

3.1 Classes—Vacuum-deposited cadmium coatings shall be classified on the basis of thickness, as follows:

<table>
<thead>
<tr>
<th>Class</th>
<th>Minimum Thickness, µm</th>
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<tbody>
<tr>
<td>12</td>
<td>12</td>
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<tr>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>5</td>
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3.2 Types—Vacuum-deposited cadmium coatings shall be identified by types on the basis of supplementary treatment required, as follows:

3.2.1 Type I—As-vacuum-deposited without supplementary chromate treatment (see Appendix X1).

3.2.2 Type II—with supplementary chromate treatment (see Appendix X2).

Note 1: It is strongly recommended that Type II should be used rather

\textsuperscript{1} This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.08.04 on Soft Metals.

\textsuperscript{2} Annual Book of ASTM Standards, Vol 02.05.

\textsuperscript{3} Annual Book of ASTM Standards, Vol 02.05.

\textsuperscript{4} Annual Book of ASTM Standards, Vol 02.04.

\textsuperscript{5} Annual Book of ASTM Standards, Vol 03.05.

\textsuperscript{6} Annual Book of ASTM Standards, Vol 15.03.
than Type I on production items.

4. Ordering Information

4.1 To make the application of this specification complete, the purchaser needs to supply the following information to the supplier in the purchase order or other governing document:

4.1.1 The designation number and year of issue of this document.
4.1.2 Class and type (3.1 and 3.2),
4.1.3 Hardness or tensile strength of the steel parts (5.2),
4.1.4 Heat treatment for stress relief, whether it has been performed or is required,
4.1.5 Undercoating, if required (5.5),
4.1.6 Test of coating composition, if required (6.1),
4.1.7 Significant surfaces (6.2.2),
4.1.8 Luster (6.6),
4.1.9 Sampling, if other than specified (Section 7),
4.1.10 Certification (11.1), and
4.1.11 Supplementary requirements, if applicable.

5. Materials and Manufacture

5.1 Nature of Finish—The cadmium metal for the production of the coating and the resultant coating shall conform to the composition requirements of Specification B 440 and shall contain not less than 99.95 mass % cadmium.

5.2 Stress Relief—Steel parts that have an ultimate tensile strength of 1000 MPa and above and that have been machined, ground, cold-formed, or cold-straightened shall be heat-treated at a minimum of 190°C for 3 h or more for stress relief before cleaning and coating.

5.3 Cleaning of Basis Metal—Proper preparatory procedures and thorough cleaning of the basis metal are essential to ensure satisfactory adhesion and corrosion resistance performance of the coating. The basis metal shall not be exposed to treatments such as cathodic cleaning, and acid treatments involving release of hydrogen on the surface of parts; nor shall materials be used for cleaning that will have damaging effects on the metal, such as pits, intergranular attack, and hydrogen embrittlement. If necessary, cleaning materials for surface preparation should be evaluated in accordance with Method F 519. The basis metal shall be abrasive dry-blasted, using new materials or materials that have not been used on other metals. All loose particles shall be removed by air blasting the parts thoroughly, using clean dry oil-free compressed air. Following air blasting, the basis metal may be outgassed by subjecting it to an electron bombardment with high-energy positive ions in a glow discharge unit. The following practices, where appropriate for precleaning to remove oil, grease, and other foreign materials should be used: B 183, B 242, B 254, B 320, and B 322.

Note 2—Parts may be cleaned by blasting with 120-mesh aluminum oxide or 100-mesh garnet grit with a nozzle pressure of 410 to 620 kPa to remove scale, oxides, or other contamination. Blasted surfaces should not be rougher than the specified finish for the part. When necessary to achieve the proper surface roughness, finer grit may be used, but aluminum oxide coarser than 120-mesh or garnet grit coarser than 100-mesh should not be used. Following air blasting, stainless steel parts should be passivated.

5.4 Deposition Process—Cadmium vacuum deposition shall be conducted in a vacuum of 1.33 × 10⁻⁴ to 3.3 Pa with an evaporation rate of 0.8 to 1.7 mg/s.

5.5 Substrate—Unless otherwise specified, cadmium shall be deposited directly on the basis metal without a preliminary undercoating of another metal. A preliminary undercoating of nickel is permissible with parts made of stainless steel.

5.6 Chromate Treatment (Type II):

5.6.1 Chromate treatment for Type II shall be done in or with special aqueous acid solutions composed of hexavalent chromium along with certain anions that act as a catalyst or film forming compound to produce a continuous, smooth, protective film.

5.6.2 The Type II film shall range in color from an iridescent yellow or a thicker more protective iridescent bronze or brown to the heavier olive drab. It may be dyed to a desired color. When necessary, a desired color shall be indicated by the purchaser and specified by the provision of a suitable colored sample or indicated on the drawing for the part.

5.6.3 Waxes, lacquers, or other organic coatings shall not be permitted as a substitute, nor shall they be used in conjunction with Type II coatings, in order to ensure the conformance to the salt spray or abrasion resistance test requirements. Waxes, etc., may be used to improve lubricity.

6. Coating Requirements

6.1 Coating Composition—When specified, the chemical composition of the coating shall be determined (see X1.3).

6.2 Thickness:

6.2.1 The thickness of the coating everywhere on the significant surfaces shall be at least that of the specified class as defined in 3.1.

6.2.2 Significant surfaces defined as those normally visible (directly or by reflection) are essential to the appearance or serviceability of the article when assembled in normal position; or can be the source of corrosion products that deface visible surfaces on the assembled article. When necessary, the significant surfaces shall be indicated by the purchaser on the applicable drawing of the article, or by the provision of suitably marked samples.

6.2.3 On nonsignificant visible surfaces, the minimum allowable thickness for Class 12 shall be Class 8 (8 µm), for Class 8 it shall be Class 5 (5 µm), and for Class 5 it shall be 4 µm.

6.3 Adhesion—The cadmium coating shall be sufficiently adherent to the basis metal to pass the tests in 9.3. When examined at a magnification of approximately 4 diameters, the coating shall not show separation from the basis metal. The formation of cracks in the coating caused by rupture of the basis metal that does not result in flaking, peeling, or blistering of the cadmium coating shall not be considered as nonconformance to this requirement.

6.4 Abrasion Resistance—The supplementary chromate film (Type II) on the cadmium coating shall be adherent, non-powdery, and shall resist abrasion when tested as detailed in 9.4.

6.5 Corrosion Resistance—Cadmium coatings with Type II treatment shall show neither white corrosion products of cadmium nor basis metal corrosion products at the end of 96 h
test period, when tested by continuous exposure to salt spray in accordance with 9.5. The appearance of corrosion products visible to the unaided eye at normal reading distance shall be cause for rejection, except that white corrosion products at the edges of specimens shall not constitute failure.

NOTE 3—The hours given are the minimum required to guarantee satisfactory performance. Longer periods before the appearance of the white corrosion products, even the as-coated Type I, and rust are possible. Salt spray resistance does not vary in exact proportion with increased plating thickness of Type II coatings. Although hours to failure (red rust) for Type I coatings are not specified, the hours given for Type II reflect the added protection of the chromate treatments without requiring impractical testing periods.

6.6 Luster—Unless otherwise specified by the purchaser, a semi-satin or a light matte luster shall be acceptable.

6.7 Workmanship and Finish:

6.7.1 The coating shall be smooth, adherent, uniform in appearance, and substantially free of blistering, pits, nodules, flaking, and other defects that may affect the function of the coating. The coating shall cover all surfaces as stated in 6.2, including thread roots, thread peaks, corners, recesses, and edges. The coating shall show no indication of contamination or improper operation of equipment used to produce the deposit, such as excessively powdery or darkened coatings. Superficial staining, which results from rinsing or drying, and variations in color or luster shall not be cause for rejection.

6.7.2 Defects and variations in appearance in the coating that arise from surface conditions of the substrate (scratches, pores, roll marks, inclusions, etc.) and that persist in the finish despite the observance of good application techniques shall not be cause for rejection.

NOTE 4—Applied finishes generally perform better in service when the substrate over which they are applied is smooth and free of torn metal, inclusions, pores, and other defects. It is recommended that the specifications covering the unfinished product provide limits for those defects. A metal finisher can often remove defects through special treatments, such as grinding, polishing, electropolishing, and chemical treatments. However, these are not normal in the treatment steps preceding the application of the finish. When desired, they shall become the subject of a special agreement between the purchaser and the supplier.

7. Sampling

7.1 Inspection Lot—An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specifications, that have been coated by a single supplier at one time, or at approximately the same time, under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

7.2 Selection—A random sample of the size required by Test Method B 602 shall be selected from the inspection lot. The articles in the sample shall be inspected for conformance to the requirements of this specification, and the lot shall be classified as conforming or nonconforming to each requirement in accordance with the criteria of the sampling plans in Test Method B 602.

NOTE 5—Test Method B 602 contains three sampling plans that are to be used with nondestructive test methods and a fourth to be used with destructive. The three methods for nondestructive tests differ in the quality level they require of the product. Test Method B 602 requires the use of the plan with the intermediate quality level unless the purchaser specifies otherwise. The purchaser should compare the plans with his needs and state which plan is to be used. If the plans in Method B 602 do not serve the needs, additional ones are given in Guide B 697.

7.3 Specimens—If separate test specimens are to be used to represent the coated articles in a test, the specimens shall be of the nature, size, and number, and shall be processed as required in 8.1, 8.2, and 8.3.

NOTE 6—When both destructive and nondestructive tests exist for the measurement of a characteristic, the purchaser needs to state which is to be used so that the proper sampling plan is selected. Also a test may destroy the coating, but in a noncritical area; or if it destroys the coating, it may be the tested article can be reclaimed by stripping and recoating. The purchaser needs to state whether the test is to be considered destructive or nondestructive.

8. Specimen Preparation

8.1 Vacuum-Coated Parts or Separate Specimens—When the vacuum-coated parts are of such form, shape, size, and value as to prohibit their use, or are not readily adaptable to a test, or when destructive tests of small lot sizes are required, the test shall be made by the use of separate specimens coated concurrently with the articles represented. The separate specimens shall be of a basis metal equivalent to that of the article represented. These separate specimens shall be introduced into a lot at regular intervals before the cleaning operations, preliminary to vacuum coating, and shall not be separated therefrom until after completion of the coating. Conditions affecting the vacuum coating of specimens, including the spacing, coating media, temperature, pressure, etc., in respect to other objects being coated, shall correspond as nearly as possible to those affecting the significant surfaces of the articles represented. Unless a need can be demonstrated, separately prepared specimens shall not be used in place of production items for nondestructive and visual examinations.

8.2 Thickness, Adhesion, and Abrasion Resistance Specimens—If separate specimens for thickness, adhesion, and abrasion resistance tests are required, they shall be strips approximately 25 mm wide, 100 mm long, and 1 mm thick, or cylindrical pieces with a cross-sectional area approximately equal to that of the production item.

8.3 Corrosion Resistance Specimens—If separate specimens for the corrosion resistance test are required, they shall be panels not less than 150 mm long, 100 mm wide, and approximately 1 mm thick.

9. Test Methods

9.1 Composition—Chemical composition of the cadmium coating shall be determined when required on the purchase order by procedures given in Test Methods E 396 or by other methods (chemical, electrochemical, spectrochemical, or X-ray fluorescence) specified on the purchase order as long as they can determine composition to within the desired limits.

9.2 Thickness:

9.2.1 The thickness of the coating shall be determined by Test Methods B 487, B 499, B 504, B 567, or B 568, as applicable. Other methods may be used if it can be demonstrated that the uncertainty of the measurements within these methods is less than 10%.
9.2.2 Thickness measurements of Type II deposits shall be made after application of the supplementary treatment. Whenever Test Methods B 504, B 567, or B 568 is used, the Type II coating shall be removed from the test area before the thickness is measured. Removal shall be done by using a mild abrasive (a paste of levigated alumina or magnesium oxide) rubbed on gently with the finger.

9.3 Adhesion—Adhesion of the vacuum-deposited cadmium coating to the basis metal shall be tested in a manner that is consistent with the service requirements of the coated article. The ability to separate the coating from the substrate by peeling as distinct from flaking caused by rupture of the deposit or of the basis metal, shall be evidence of failure. One of the following methods for determining adhesion shall be used:

9.3.1 The surface of the coated article shall be scraped or sheared with a sharp edge, knife, or razor blade through the coating down to the basis metal and examine under 4X magnification for evidence of non-adhesion.

9.3.2 The part shall be plastically deformed if possible, by clamping it in a vise and bending the projecting portion back and forth until rupture occurs.

9.3.3 Any suitable procedure, such as the burnishing test, the draw test, or the scribe test in accordance with Test Methods B 571 can be used.

NOTE 7—There is no single satisfactory test for evaluating the adhesion of vacuum-deposited coatings. Those given in 9.3 are widely used; however, other tests may prove more applicable in specific cases.

9.4 Abrasion Resistance—The abrasion resistance of the supplementary chromate film (Type II coating) shall be determined in accordance with Practice B 201.

9.5 Salt Spray Corrosion Resistance:

9.5.1 The 5% neutral salt spray (fog) test as defined in Test Method B 117 shall be used.

9.5.2 Parts with Type II supplementary chromate film shall be aged at room temperature for 24 h before subjection to the salt spray test.

NOTE 8—In many instances, there is no direct relation between the results of an accelerated corrosion test and the resistance to corrosion in other media, because several factors, such as the formation of protective films and temperature, which influence the progress of corrosion, vary greatly with the conditions encountered. The results obtained in the test should, therefore, not be regarded as a direct guide to the corrosion resistance of the tested materials in all environments where these materials may be used. Also, performance of different materials in the test cannot always be taken as a direct guide to the relative corrosion resistance of these materials in service.

9.6 Visual Examination—Material for compliance with the requirements of luster (see 6.6) and workmanship (see 6.7) after vacuum deposition.

10. Rejection and Rehearing

10.1 Coatings that fail to conform to the requirements of this specification or to authorized modifications shall be rejected. They may be reconsidered for acceptance by rectifying inspection in accordance with Test Method B 602 and Guide B 697 when allowed by the purchaser. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of test, the producer or supplier may make claim for rehearing. Finishes that show imperfections during subsequent manufacturing operations may be rejected.

11. Certification

11.1 When specified in the purchase order or contract, the producer’s or supplier’s certification shall be furnished to the purchaser stating that samples representing each lot have been manufactured, tested, and inspected in accordance with the specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

12. Packaging and Package Marking

12.1 Preservation, packaging, and packing methods for vacuum-deposited cadmium parts or articles employed by a supplier shall be such as to preclude damaging during shipment and handling (see Appendix X3).

13. Keywords

13.1 cadmium coatings, vacuum deposited; vacuum deposited coatings, cadmium

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirement shall apply only when specified by the purchaser as part of the purchaser’s order or contract and for all agencies of the United States Government.

S1. Responsibility for Inspection

S1.1 The producer or supplier shall be responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein unless disapproved by the purchaser. The purchaser retains the right to perform any of the inspections and tests set forth in this specification where such inspections and tests are deemed necessary to ensure that the supplies and services conform to the prescribed requirements.
X1. VACUUM-DEPOSITED CADMIUM COATINGS

X1.1 Application and Use—Hardened, heat-treated ferrous alloys with the vacuum-deposited cadmium are widely used by the aerospace industry to minimize the problem of hydrogen embrittlement and provide the corrosion protection of cadmium. The Type I (as-deposited) coating is useful for lowest cost protection where early formation of white corrosion products would not be detrimental.

X1.2 Limitations—The Type I vacuum-deposited cadmium coatings should not be used on production items that in service will reach a temperature of 225°C or higher, or come in contact with other parts that reach these temperatures. Cadmium-coated parts that may be subjected to heat from soldering, brazing, or welding operations should be so labeled or tagged to indicate being so coated because of the danger from poisonous cadmium oxide vapors evolved during those operations.

X1.3 Composition—The composition of the deposited cadmium coating has been specified in order to indicate the purity of the material that is to be used for processing and to control the advantages of the vacuum procedure, that is, no contaminations are present in the chambers and no possibility of local galvanic corrosion occurring on the coating surface between the residuals and cadmium. With other methods of cadmium deposition, there is always a possibility of coating contamination because of the residuals present in the plating bath or the media for the peen plating. No simple method exists for determining the extremely low residual contents of the cadmium deposit; however the X-ray fluorescence techniques is presently considered the most satisfactory.

X2. SUPPLEMENTARY TREATMENT (TYPE II)

X2.1 Purpose—The prime purpose of the supplementary finish (Type II) with unleached chromates on cadmium coatings is to retard or prevent the formation of white corrosion products on surfaces exposed to stagnant water, salt water, marine atmospheres, high humidity, or cyclic condensation and drying. This treatment should also delay the appearance of red corrosion on the cadmium coated articles from the basis metals, indicating destruction of the coating. The iridescent yellow to olive drab chromate films are satisfactory as undercoats if production items are to receive an organic paint system application, generally required by the aerospace industry. Supplementary finishes can prevent finger markings and corrosion of parts which may occur at room temperature during assembly and storage. Leached chromate films are not recommended for use as a supplementary finish with vacuum-deposited cadmium coatings.

X2.2 Limitations—Supplementary chromate treatments should not be applied to production items that will not be painted and will be continuously exposed to temperatures in excess of 65°C or intermittently exposed for short periods to a temperature of approximately 150°C.

X2.3 Handling Precautions—Chromate-treated articles, which involve only dipping in chemical solutions, normally require 24 h at 20 to 30°C to render them suitable for handling without damage to the finish coating while the latter is in the gelatinous form. It is important with such coatings that the supplementary film is not damaged while wet in order to comply with workmanship requirements.

X3. PACKAGING LIMITATIONS

X3.1 Cadmium-coated articles without supplementary protective films should not be packaged or packed in nonventilated containers, either together or in contact with electrical and electronic equipment, because of the danger of deleterious effects on cadmium coating from unstable electrical insulation. In addition to organic electrical insulation, phenolic resinous substances and others containing unsaturated carbon-to-carbon linkages, such as oil paints and impregnated papers, cause an abnormal attack on the cadmium by setting free, in the presence of moisture, formic acid, butyric acid, etc. Corrosion of cadmium-coated items has been noted when they have been packaged in direct contact with container materials such as wood or cardboard. Corrosion has been especially severe if the container materials have become wet or have been stored under conditions of high humidity.
Standard Specification for Electrodeposited Coatings of Silver for Engineering Use

This standard is issued under the fixed designation B 700; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers requirements for electrodeposited coatings of silver used for engineering purposes that may be mat, bright, or semibright and are not less than 98 % silver purity.

1.2 Coatings of silver covered by this specification are usually employed for solderable surfaces, electrical contact characteristics, high electrical and thermal conductivity, thermocompression bonding, wear resistance of load-bearing surfaces, and spectral reflectivity.

1.3 In the Appendixes important characteristics of electrodeposited silver coatings are briefly described which must be considered when used in engineering applications, namely electrical conductivity (see Appendix X1), silver migration (see Appendix X2), thickness (see Appendix X3), hardness (see Appendix X4), and atmospheric tarnish (see Appendix X5).

1.4 The following hazards caveat pertains only to the test methods section of this specification: This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 252 Guide for Preparation of Zinc Alloy Die Castings for Electroplating Conversion Coatings
B 253 Guide for Preparation of Aluminum Alloys for Electroplating
B 254 Practice for Preparation of and Electroplating on Stainless Steel
B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings
B 322 Practice for Cleaning Metals Prior to Electroplating
B 343 Practice for Preparation of Nickel for Electroplating with Nickel
B 374 Terminology Relating to Electroplating
B 481 Practice for Preparation of Titanium and Titanium Alloys for Electroplating
B 482 Practice for Preparation of Tungsten and Tungsten Alloys for Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
B 507 Practice for Design of Articles to be Electroplated on Racks
B 542 Terminology Relating to Electrical Contacts and Their Use
B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
B 571 Test Methods for Adhesion of Metallic Coatings
B 578 Test Method for Microhardness of Electroplated Coatings
B 579 Specification for Electrodeposited Coatings of Tin-Lead Alloy (Solder Plate)
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 678 Test Method for Solderability of Metallic-Coated Products
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 762 Method of Variables Sampling of Metallic and Inorganic Coatings
B 849 Specification for Pre-treatments of Iron or Steel for Reducing the Risk of Hydrogen Embrittlement
B 850 Guide for Post-Coating Treatments of Steel for Reducing the Risk of Hydrogen Embrittlement
D 3951 Practice for Commercial Packaging

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1 This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.08.02 on Precious Metal Coatings. Current edition approved May 10, 1997. Published February 1998. Originally published as B 700 – 81. Last previous edition B 700 – 90.

2 Annual Book of ASTM Standards, Vol 02.05.


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3. Terminology

3.1 Definitions—Many of the terms used in this specification are defined in Terminologies B 374 or B 542.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 rack-plating—an electrodeposition process in which articles to be coated are mounted on racks or other fixtures during the process.

3.2.2 significant surfaces—surfaces that are normally visible, directly or by reflection, or that are essential to the serviceability or function of the article or which can be the source of corrosion products or tarnish films that interfere with the function or desirable appearances of the article. When necessary, the significant surfaces shall be indicated on the drawings of the parts, or by the provisions of suitably marked samples.

3.2.2.1 Discussion—Variation in the coating thickness from point-to-point on a coated article is an inherent characteristic of electroplating processes. Therefore, the coating thickness will have to exceed the specified value at some points on the significant surfaces to ensure that the thickness equals or exceeds the specified value at all points. The average coating thickness on the article usually will be greater than that specified; how much greater is largely determined by the shape of the article (see Practice B 507) and the characteristics of the electroplating process. Additionally, the average coating thickness on an article will vary from article to article within a production lot. If all the articles in a production lot are to meet the thickness requirement, the average coating thickness of a production lot as a whole will be greater than the average necessary to ensure that a single article meets requirements.

3.2.3 strike or flash—a thin, typically less than 0.25-µm (10 µ-in.) metallic coating layer between metallic coatings to improve adhesion.

3.2.4 underplating—an application of a metallic coating layer between the basis metal or substrate and the topmost metallic coating or coatings (see 6.3.4).

4. Classification

4.1 Electrodeposited coatings of silver shall be classified for Type based on minimum purity, Grade whether bright, semibright, or mat, Class if supplementary surface treatment is applied, and thickness in micrometers.

4.2 Purity—Specify by Type as follows:

Type 1—99.9 % min
Type 2—99.0 % min
Type 3—98.0 % min

NOTE 1—Metallic or organic brighteners used for grain refinement may be present in the electrodeposit so long as they do not interfere with the stated function of the coating and are acceptable to the purchaser (see Appendix X1).

4.3 Surface Appearance—Specify by Grade in letter code as follows:

Grade A, Mat—Electrodeposits without luster, obtained from electroplating solutions operated without the use of brighteners.

Grade B, Bright—Electrodeposits obtained by the use of brighteners in the electroplating bath.

Grade C, Bright—Electrodeposits obtained by mechanical or chemical polishing of Grade A silver coatings.

Grade D, Semibright—Electrodeposits obtained by the use of brighteners in the electroplating bath.

4.4 Supplementary Surface Treatment—Specify by Class in letter code as follows:

Class N—A finish that has had no supplementary tarnish resistant (that is, chromate) treatment (see Appendix X5).

Class S—A finish that has had a supplementary tarnish resistant (that is, chromate) treatment.

Note 2—Class S is not suitable for food service applications.

5. Ordering Information

5.1 To make application of this standard complete, the purchaser needs to supply the following information to the seller in the purchase order or other governing document:

5.1.1 Name, designation, and year of issue of this standard.

5.1.2 Type (see 4.2), Grade (see 4.3), Class (see 4.4) and Thickness (see 6.6 and Appendix X3).

5.1.3 Nature of Substrate—If, for example, it is high strength steel, the need for stress relief (see 6.3.2.1) and embrittlement relief (see 6.3.5.1). If it is copper, an undercoat is needed (see S1.3) for some applications.

5.1.4 Significant Surfaces (see section 3.2.2).

5.1.5 Appearance (see 6.7).

5.1.6 Underplates (see 6.3.4 and S1.3).

5.1.7 Requirements and methods of testing for one or more of the following: need for and type of test specimens (see 8.1), thickness (see 6.6, 8.2, and Appendix X3), adhesion (see 6.8 and 8.3), hardness (see 6.10.1 and 8.7), absence of embrittlement (see 8.4), solderability (see 6.9 and 8.5), spectral reflectance (see 6.10.2 and 8.8), or electrical conductivity (see 6.10.3 and 8.9).

5.1.8 Sampling Plans and Quality Assurance (see Section 7 and S1.2).

6. Coating Requirements

6.1 Nature of Coating—The coating essentially shall be silver, considering the type specified, produced by electrodeposition from aqueous electrolytes.

6.2 Purity of Coating—The coating shall meet the chemical composition requirements of the specified type as defined in 4.2 and measured as described in 8.6.

6.3 Process:

6.3.1 The basis metal shall be subjected to such cleaning procedures as are necessary to ensure a surface satisfactory for subsequent electroplating. Materials used for cleaning shall
have no damaging effects on the basis metal resulting in pits, intergranular attack, stress corrosion cracking, or hydrogen embrittlement.

NOTE 3—For basis metal preparations, the following appropriate ASTM standards are recommended: Practices B 183, B 242, B 252, B 254, B 281, B 322, B 343, B 481, and B 482, and Guide B 253.

6.3.2 Preplating Operations—Electroplating shall be applied after all basis metal heat treatments and mechanical operations such as forming, machining, and joining of the article have been completed.

NOTE 4—Silver deposits may be used to facilitate mechanical operations such as forming and drawing. In these applications, silver is applied before such process steps.

6.3.2.1 Stress Relief Treatment—Parts that are made of steels with ultimate tensile strength of 1000 MPa or over (hardness of 31 HRC or greater) that have been machined, ground, cold-formed or cold-straightened subsequent to heat treatment, may require stress relief heat treatment when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specification B 849 may be consulted for a list of pretreatments that are used widely.

6.3.3 Strike—The final silver coating shall be preceded by a silver or gold strike for optimum adhesion.

6.3.4 Underplating—A nickel or nickel-alloy intermediate layer, at least 1 µm thick, shall be applied before the silver electroplate when the product being plated is made from copper or copper alloy. Nickel underplatings are also applied for other reasons.

6.3.5 Post-Plating Procedures:

6.3.5.1 Embrittlement Relief—Parts that are made of steels with ultimate tensile strength of 100 MPa or over (hardness of 31 HRC or greater), as well as surface hardened parts, may require post-coating hydrogen embrittlement relief baking when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specification B 850 may be consulted for a list of post-treatments that are used widely.

6.4 Surface Appearance—The coating’s surface finish shall meet the requirements of the specified grade defined in 4.3.

6.5 Supplementary Post Treatment—The coating shall meet the requirements of the specified class defined in 4.4.

6.6 Thickness—The silver coating thickness on significant surfaces shall be at least that specified (see Appendix X3) when measured as described in 8.2.

6.7 Appearance—Silver electroplated coated articles shall be covered completely on all surfaces as specified in the manufacturing document and shall have a uniform appearance with no visible defects to the extent that the nature of the basis metal and good commercial practice permit. The requirement for uniform color or appearance need not apply for subsequent passivation or other treatments of the silver.

6.7.1 Defects—Defects in the surface of the basis metal such as scratches, pits, non-conducting inclusions, and roll and die marks may adversely affect the appearance and performance of applied coatings. Such defects that persist in the finish despite the observance of good metal finishing practices shall not be cause for rejection.

NOTE 5—Coated finishes generally perform better in service when the substrate over which they are applied is smooth and free from torn metal, inclusions, pores, and other defects. It is recommended that the specifications covering the unfinished product provides limits for those defects. A metal finisher often can remove defects by means of special treatments such as grinding, polishing, abrasive blasting, chemical treatments, and electropolishing. However, these are not normal for the treatment steps preceding application of the finish. When they are desired, they are the subject of special agreement between the purchaser and the supplier.

6.8 Adhesion—The silver coatings shall be free of blisters and peeled areas indicating poor adhesion when tested in accordance with 8.3.

NOTE 6—Some applications may require no separation by any mechanical means such as machining or milling through the interface.

6.9 Solderability—The silver plated surfaces shall produce solder coatings which shall be bright, smooth, and uniform. At least 95 % of the sample surface shall show good wetting when tested as described in 8.5.

6.10 Supplementary Requirements:

6.10.1 Hardness—If a hardness requirement is specified, the hardness of the silver coatings shall conform to that specified as measured as described in 8.7.

6.10.2 Spectral Reflectance—The spectral reflectance of the silver coatings, if required, shall conform to that specified when measured as described in 8.8.

6.10.3 Electrical Conductivity—The electrical conductivity of the silver coatings, if required, shall conform to that specified when measured as described in 8.9.

7. Sampling

7.1 A random sample of the size required by Test Method B 602 or Method B 762 shall be selected from the inspection lot (see 7.2). The articles in the lot shall be classified as conforming or nonconforming to each requirement according to the criteria of the sampling plans in the chosen method.

NOTE 7—Test Method B 602 contains four sampling plans, three for use with nondestructive test methods; the fourth is for use with destructive test methods. The three methods for nondestructive tests differ in the quality level they require of the product. Test Method B 602 requires use of the plan with the intermediate quality level unless the purchaser specifies otherwise. It is recommended that the purchaser compare the plans with his needs and state which plan is to be used. If the plans in Test Method B 602 do not serve the needs, additional ones are given in Guide B 697 which provides a large number of plans and also gives guidance in the choice of a plan. When Guide B 697 is specified, the buyer and seller need to agree on the plan to be used.

NOTE 8—Method B 762 is a variables sampling plan. Such plans can only be used when a test yields a measured quantity, such as thickness, and when the requirements are stated as a numerical unit also such as thickness. Method B 762 contains several plans for special needs. Buyer and seller may agree on the plan or plans to be used; if not, Method B 762 identifies the plan to be used.

NOTE 9—When both destructive and nondestructive tests exist for the measurement of a characteristic, the purchaser needs to state which is to be used so the proper sampling plan is selected. Whether or not a test is destructive may not always be clear. A test may destroy the coating but in a noncritical area. The purchaser needs to state whether the test is to be considered destructive or nondestructive. The decision is important because the plans for destructive tests are significantly less able to discriminate between acceptable and unacceptable lots. This is because fewer parts are tested.
7.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specifications, that have been coated by a single supplier at one time or approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

7.3 If special test specimens are used to represent the coated articles in a test, the specimens shall be of the nature, size, and number and shall be processed as required in 8.1.

8. Test Methods

8.1 Special Test Specimens:

8.1.1 Special test specimens are used to represent the coated articles in a test if the articles are of a shape, size or material that is not suitable for the test, or if it is preferred not to submit samples to a destructive test because, for example, they are expensive or few in number. The permission or requirement to use special test specimens, their number, the material they are to be made of, and their size and shape shall be stated by the purchaser.

8.1.2 The special test specimen shall duplicate those characteristics of the article that influence the property being tested; and it shall be processed with the article through those process steps that influence the property.

8.1.3 The special test specimens used to represent an article in an adhesion, solderability, or appearance test shall be made of the same material, shall be in the same metallurgical condition, and shall have the same surface condition as the articles they represent. They shall be placed in the production lot of and be processed along with the articles they represent.

8.1.4 Special test specimens used to represent an article in a coating thickness test shall be introduced into the process at the point where the coatings are applied and shall be carried through all the steps that have a bearing on the coating thickness.

NOTE 10—When special test specimens are used to represent a coated article in a thickness test, the specimens will not necessarily have the same thickness and thickness distribution as the article unless the specimens and the articles are of the same general size and shape. Therefore, before coated articles may be accepted on the basis of a thickness test performed on test specimens, the relationship between the thickness on special test specimens and the thickness on the part needs to be established. The criterion of acceptance is that the thickness on the specimen corresponds to the required thickness on the articles.

8.2 Thickness—The coating thickness shall be measured at locations on significant surfaces where it would be expected to be a minimum either nondestructively or destructively:

8.2.1 Nondestructive—Measure thickness of silver coatings by beta backscatter (Test Method B 567), by X-ray spectrometry (Test Method B 568), or by the magnetic method (Test Method B 499).

8.2.2 Destructive—Measure thickness of silver coatings by the coulometrical technique (Test Method B 504) or by microscopic cross-sectioning procedures (Test Method B 487).

8.3 Adhesion—Determine adhesion qualitatively with one of the procedures given in Test Methods B 571. Other procedures, such as machining milling, or shot peening may prove more appropriate in special cases.

8.4 Hydrogen Embrittlement Relief—Steel products that are required as described in 6.3.5.1 to be heat treated to remove hydrogen embrittlement shall be evaluated as directed by Method F 519 as a means of evaluating the process, using special test specimens (see 8.1).

8.5 Solderability—Determine solderability qualitatively by Test Method B 678.

NOTE 11—Other useful tests may be found in Specification B 579.

8.6 Purity of Coating—Obtain the composition of the silver coating by determining the impurities (that is silver by difference) by emission spectroscopy, x-ray fluorescence, atomic absorption, or other methods capable of determining quantities of 0.01 mass % or less.

8.7 Hardness—Measure low load microhardness in accordance with Test Method B 578 (see Appendix X4).

8.8 Specular Reflectance—Measure reflectance in the spectral region of 0.60 ± 0.05 µm on a coated surface using a device suitable for determinations at an angle of 45° employing a narrow acceptance angle (suited for mirror finishes).

8.9 Electrical Conductivity—Measure electrical conductivity by Test Method E 1004 using separate test specimens.

9. Packaging

9.1 Some packaging materials may emit fumes that can be deleterious to the appearance or performance, or both, of electroplated silver surfaces. Useful guidelines for commercial packaging can be found in Specification D 3951.

10. Special Government Requirements

10.1 The following special requirements shall apply when the ultimate purchaser is the U.S. Government or an agent of the U.S. Government.

10.1.1 Sampling—For government acceptance the sampling plan is to be in accordance with ANSI/ASQC Z1.4.

10.1.2 Quality Assurance:

10.1.2.1 Inspection Responsibility—The producer or supplier shall be responsible for the performance of all inspection and test requirements specified herein. Except as otherwise specified in the contract or order, the producer or supplier may use his own or any other suitable facilities for the performance of the inspection and test requirements specified herein, unless disapproved by the purchaser. The purchaser shall have the right to perform any of the inspections and tests set forth in this specification where such inspections are deemed necessary to assure the material conforms to the prescribed requirements.

10.1.3 Undercoats—The silver electroplating shall be applied over an intermediate coating of nickel or nickel over copper on steel, zinc, and zinc base alloys. Copper and copper base alloys require intermediate coatings. Copper alloy basis metal articles on which a nickel undercoat is not used and other basis metal whereon a copper undercoat is employed shall not be used for continuous service in excess of 149°C.

10.1.4 Packaging—Parts plated for the U.S. Government and Military, including as subcontracts, shall be packaged in accordance with Practice D 3951.
X1. ELECTRICAL RESISTIVITY

X1.1 The electrical resistivity of silver deposits will vary with the type of silver electrolyte used. The resistivity will be generally in the range from 0.017 to 0.020 \( \mu \Omega \cdot m \). Resistivity of pure silver is 0.0162 \( \mu \Omega \cdot m \). Sulfur- and selenium-containing electrolytes yield deposits having resistivity values 10 to 15% higher than that of pure silver; those of antimony-brightened deposits can be substantially higher than that of pure silver. Periodic reverse current has been reported to yield deposits with about 1% higher resistivity than the International Annealed Copper Standard versus about 6% higher than IACS for deposits produced by direct current alone.\(^7\)


X2. SILVER MIGRATION

X2.1 When silver coatings are used as electrical conductors they may be susceptible to electromigration effects. Silver migration refers to the electrochemical transport of the metal from one conductor to another under the influence of an applied d-c electrical field. It can occur whenever the insulator separating the polarized (or *biased*) conductors (as on PC boards, flexible circuitry, chip carriers, or IC ceramics) has acquired sufficient moisture to allow electrolytic (ionic) conduction to occur.

X2.1.1 In the simplest case, metallic silver on the conductor with the more positive potential (anode) is oxidized to a more soluble form. The resulting positively-charged ions then move under the influence of the field through moisture paths on or in the insulator toward the more negative conductor (cathode), where they are reduced back to silver metal (Fig. X2.1).

X2.1.2 In practice, electromigration can produce two separate, but not always distinct, phenomena, both of which can lead to impairment of a circuit’s electrical integrity. These are *silver staining* and filamental or dendritic bridging. Colloidal silver deposits appear as brownish stains on insulator areas where migrating silver ions have been chemically or photolytically reduced to metallic silver before they are able to reach the cathode. Silver dendrites, on the other hand, result from the fact that the ions tend to deposit at localized sites on the cathode in the form of needles or spikes. Once these nuclei have formed, the higher current density at their tips will greatly increase the probability of further deposition. This shows up as an accelerated growth outward from the tips in the form of thin black filaments of silver, extending from the cathode back toward the positive conductor. In the course of this growth, branching usually occurs at definite crystallographic angles, resulting in a characteristic *dendritic* structure.

X2.1.3 When a filament finally bridges the gap between polarized conductors, a sudden drop in electrical resistance will occur. Although the magnitude of the initial resistance drop will be small (because of the small cross-sectional area of a single filament), a rapid increase in additional bridges will soon lower the resistance sufficiently to produce circuit failures. Bridging also can be produced by colloidal silver deposits, especially at temperatures above 70°C and on contaminated surfaces.

X2.1.4 The primary operating parameters that promote electromigration problems are:

X2.1.4.1 Moisture (that is, high relative humidity),
X2.1.4.2 Contamination on the insulator surface,
X2.1.4.3 Voltage difference between conductors,
X2.1.4.4 Narrow spacing widths between conductors, and
X2.1.4.5 Elevated temperatures coupled with high relative humidity. The tendency towards electromigration also depends upon the nature of the insulator material and its surface condition as well as on the type of silver plating used. All of these factors are discussed in a 1988 publication.\(^8\)

X3. SILVER COATING APPLICATIONS

<table>
<thead>
<tr>
<th>Application</th>
<th>Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Minimum for short-term shelf life solderability</td>
<td>1</td>
</tr>
<tr>
<td>Minimum for contact connectors having limited wear</td>
<td>2.5</td>
</tr>
<tr>
<td>Nominal thickness for thermocompression bonding and nominal thickness</td>
<td>5</td>
</tr>
<tr>
<td>for domestic hollowware</td>
<td></td>
</tr>
<tr>
<td>Medium-quality hollowware, short-life domestic cutlery and</td>
<td>10</td>
</tr>
<tr>
<td>flatware, and suggested thickness for thermocompression bonding</td>
<td></td>
</tr>
<tr>
<td>and die attachment for semiconductors</td>
<td></td>
</tr>
<tr>
<td>Normal hotel flatware and high-quality domestic flatware and</td>
<td>20</td>
</tr>
<tr>
<td>hollowware</td>
<td></td>
</tr>
<tr>
<td>Applications where very severe wear resistance is required, such as</td>
<td>≥40</td>
</tr>
<tr>
<td>machine bearings and for high-quality flatware</td>
<td></td>
</tr>
</tbody>
</table>

X4. HARDNESS

X4.1 The hardness of unalloyed silver deposits is much higher than that for annealed wrought silver, probably due to a fine grain size. Electrolyte composition, operating parameters, and deposition conditions affect hardness, as well as the presence of small amounts of organic and inorganic brighteners and alloying elements.9

X5. SILVER TARNISH

X5.1 Silver readily forms creeping sulfide tarnish films in sulfur-containing environments. Free sulfur present from certain rubber compounds and cardboard materials cause growth of silver sulfide films at rapid rates. Such films create high electrical contact resistance and decrease solderability of silver surfaces. Anti-tarnish treatments are helpful though not completely effective.

X5.2 Anti-tarnish treatments commercially available include electrolytic processes based on chromates, immersion processes based on various film-forming organic compounds; and immersion tinplates. Process parameters and degree of protection afforded by these various treatments vary widely. There also are available various packaging materials that incorporate volatile organic compounds, which provide so-called vapor barrier protection when in contact or close proximity to silver surfaces.

Standard Test Method for Apparent Density of Powders Using Arnold Meter

This standard is issued under the fixed designation B 703; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure for determining the apparent density of both free- and non-free-flowing powders, premixes, and blended mixes.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 212 Test Method for Apparent Density of Free-Flowing Metal Powders
B 215 Practices for Sampling Finished Lots of Metal Powders
B 243 Terminology of Powder Metallurgy
B 329 Test Method for Apparent Density Powders of Refractory Metals and Compounds by Scott Volumeter
B 417 Test Method for Apparent Density of Non-Free-Flowing Metal Powders

3. Terminology

3.1 Definitions—Useful definitions of terms for metal powders and powder metallurgy are found in Terminology B 243.

3.2 Definitions of Terms Specific to This Standard:
3.2.1 Arnold Density Meter—This instrument enables the user to determine the density of any powder or mix, whether or not the powder has flow characteristics. It duplicates the action of the fill shoe of the press, consequently the values obtained on metal powders are approximately 0.2 g/cm³ higher than those obtained with the Hall, Test Method B 212; Carney, Test Method B 417; or Scott, Test Method B 329, instruments.

4. Summary of Test Method

4.1 This test method consists of slowly sliding a bushing partially filled with powder over a hole in a hardened steel block, collecting and weighing the powder, and calculating its apparent density.

5. Significance and Use

5.1 The apparent density is an important measure of a material characteristic inherent in the powder, which is useful to the powder producers as well as end users in determining lot to lot consistency. Knowledge of the apparent density of the final mix as obtained with this test method is very beneficial to the powder metallurgy (P/M) parts fabricator for setting compression ratios for fixed fill die cavities.

5.2 This test method may be part of a purchase agreement between the powder manufacturer and P/M parts producer, or it may be an internal quality control test for either party.

6. Apparatus

6.1 Test Block—A hardened, tempered, and demagnetized steel block (60 HRC Min.) having a center hole 31.6640 ± 0.0025 mm (1.2466 ± 0.0001 in.) and a height of 25.4000 ± 0.0025 mm (1.0000 ± 0.0001 in.) that corresponds to a volume of 20 cm³ (1.22 in.³) (Fig. 1).

6.2 Bushing, either brass or bronze, approximately 38 mm (1.50 in.) inside diameter (ID) by 45 mm (1.75 in.) outside diameter (OD) by 38 mm long (Fig. 1).

6.3 Weighing Paper—A sheet of cellophane or glazed or waxed paper measuring approximately 150 by 150 mm square (6.0 by 6.0 in.).

6.4 Balance—A balance having a capacity of at least 200 g suitable for weighing to 0.01 g.

7. Sampling

7.1 Obtain a test sample in accordance with Practices B 215.

7.2 The powder sample shall be of sufficient volume to fill the bushing to about three quarters of its height.

8. Preparation of Apparatus

8.1 Clean test block and bushing thoroughly to eliminate

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1 This specification is under the jurisdiction of ASTM Committee B-9 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.02 on Base Metal Powders.

2 Annual Book of ASTM Standards, Vol 02.05.

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1 An editorial change was made in 9.4 in September 1999.

3 The Arnold Density Meter complete with bushing is available from Arnold P/M Consulting Services, 648 Cedar Road, St. Marys, PA 15857. Also available from Alcan Powders & Pigments, 901 Lehigh Avenue, Union NJ 07083-7632.
any loose powder particles.

9. Procedure

9.1 Place the steel block on a sheet of preweighed or tared paper.

9.2 Place the bushing on the block, on either side of the hole.

9.3 Fill the bushing slowly and carefully to three-quarters of its height with powder. (A ring on the ID of the bushing indicates the proper fill height.)

9.4 With downward pressure on the bushing, slowly slide the bushing toward the hole while twisting it. This gives a snowplow action to the powder as it falls slowly into the hole. Continue this motion until the bushing passes the hole. Stop, and again with downward pressure on the bushing, slide it straight back over the hole to its starting position. The sliding action must be slow enough to allow for complete filling of the steel block cavity.

9.5 Remove the steel block from the preweighed paper being careful not to tip the block and spill additional powder on to the paper.

9.6 Transfer the preweighed or tared paper to a balance and weigh. Calculate the density from the following equation:

\[
\text{Apparent Density, g/cm}^3 = \frac{\text{Mass in grams}}{20 \text{ cm}^3}
\]  

10. Report

10.1 Report the apparent density to the nearest 0.01 g/cm³.

To minimize confusion with other test methods, report as Arnold Density, g/cm³.

11. Precision and Bias

11.1 Precision—Precision has been determined from an interlaboratory study performed by Subcommittee B09.02.

11.1.1 Apparent Density Using the Arnold Meter:

\[
\text{Repeatability } r = 0.08 \text{ g/cm}^3
\]  

11.1.2 In 95% of such tests, on the basis of test error alone, duplicate tests in the same laboratory by the same operator, on one homogeneous lot of powder, will differ by no more than the stated amount.

\[
\text{Reproducibility } R = 0.15 \text{ g/cm}^3
\]  

11.1.3 For 95% of comparative trials done in two different laboratories and on the basis of test error alone, a single test on the same homogeneous lot of powder will differ by no more than the stated amount.

11.2 Bias—No bias statement can be made because there is no accepted standard or reference powder for apparent density.

12. Keywords

12.1 apparent density, Arnold Density, density of non-free-flowing powders, metal powders
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Standard Specification for
Sintered Copper Structural Parts for Electrical Conductivity Applications

This standard is issued under the fixed designation B 715; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers sintered Powder Metal (P/M) structural parts of substantially pure copper of two types depending on density. It is anticipated that the parts will be used in applications where high electrical conductivity is required.

1.2 The values stated in inch-pound units are the standard. The SI values in parentheses are for information only and may be approximate.

2. Referenced Documents

2.1 ASTM Standards:
B 243 Terminology of Powder Metallurgy
B 328 Test Method for Density, Oil Content, and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings
E 8 Test Methods of Tension Testing of Metallic Materials

3. Terminology

3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243. Additional descriptive information is available in the Related Materials section of Vol 02.05 of the Annual Book of ASTM Standards.

4. Ordering Information

4.1 Orders for parts under this specification shall include the following information:
4.1.1 Dimensions (see Section 9),
4.1.2 Chemical composition (see 6.1 and Table 1),
4.1.3 Density (see 7.1 and Table 2),
4.1.4 Mechanical properties (see Section 8),
4.1.5 Electrical properties (see 7.2),
4.1.6 Certification (see Section 13), and
4.1.7 Metallography (see Section 14).

5. Materials and Manufacture

5.1 Structural parts shall be made by molding and sintering metal powders followed by repressing and resintering, if necessary, to produce finished parts conforming to the requirements of this specification.

6. Chemical Composition

6.1 The material shall conform to the requirements in Table 1.

6.2 The chemical analysis shall be made in accordance with the methods prescribed in Vol 03.05 of the Annual Book of ASTM Standards, or by any other approved method agreed upon between the manufacturer and the purchaser.

NOTE 1—Iron contamination should be avoided. Iron in solid solution in copper has a deleterious effect on both electrical and thermal conductivity. Iron not in solid solution (admixed) has a much lesser effect on conductivity. An example of the effect of iron on conductivity is shown in Fig. X1.1 appended.

7. Physical Properties

7.1 Density:
7.1.1 The density shall be determined by Test Method B 328. If the density does not vary more than 0.3 g/cm³ from one section of the structural part to any other section, the overall density shall fall within the limits prescribed in Table 2. If the density varies more than 0.3 g/cm³ from one section of the part to another, the manufacturer and the purchaser shall agree upon a critical section of the part where the stresses are highest. The density of this critical section, rather than the average density, shall fall within the limits prescribed in Table 2.

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TABLE 1 Chemical Requirements

<table>
<thead>
<tr>
<th>Element</th>
<th>Composition, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper, min</td>
<td>99.80</td>
</tr>
<tr>
<td>Other, max</td>
<td>0.20</td>
</tr>
</tbody>
</table>

TABLE 2 Density Requirements

<table>
<thead>
<tr>
<th>Type</th>
<th>Dry Density, g/cm³</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>7.8 to 8.3</td>
</tr>
<tr>
<td>II</td>
<td>8.3 min</td>
</tr>
</tbody>
</table>
7.2 Electrical Conductivity:
7.2.1 The manufacturer and the purchaser shall agree on qualification tests to determine the electrical conductivity.\(^4\) The test shall be made on sample parts or specimens molded to a given density using an apparatus based on the eddy-current principle.

7.2.1.1 Conductivity is determined with an instrument that indicates the resistance of a material to the flow of eddy currents. Prior to making the tests, the instrument is allowed to warm up for a period of time recommended by the manufacturer. The instrument is adjusted using three standards of known conductivity supplied by the manufacturer. Test specimens must be at the same temperature as the reference materials used in adjusting the instrument. Several readings at different locations are taken on each test specimen to obtain a representative average value.

7.2.1.2 No specimen preparation is required providing the surface is flat in the probe area.

7.2.1.3 Electrical conductivity values shall be reported in percent IACS (International Annealed Copper Standards).

Note 2—Typical electrical conductivity values which may be expected from special specimens molded to size are given in Table X1.1.

8. Mechanical Properties

8.1 The manufacturer and the purchaser shall agree on both qualification tests and limits for the determination of mechanical properties. These tests shall be made on sample parts or specimens and should be determined after consideration of the function a part is to perform. Subsequent to the approval of the first sample parts on which the qualification tests have been set up, all parts in the shipment shall conform to the limits agreed upon.

Note 3—Typical properties in tension which may be expected from special specimens molded to size are given in Table X1.1.

9. Permissible Variations in Dimension

9.1 Permissible variations in dimensions shall be within the limits specified on the drawings which describe the structural parts that accompany the order, or variations shall be within the limits specified in the order.

10. Sampling

10.1 Lot—Unless otherwise specified, a lot shall consist of parts of the same form and dimensions, made from powder of the same composition, molded and sintered under the same conditions, and submitted for inspection at one time.

10.2 Chemical Analysis—If required by purchase agreement, at least one sample for chemical analysis shall be taken from each lot. A representative sample of chips may be obtained by dry-milling, drilling, or crushing at least two pieces with clean dry tools without lubrication. In order to obtain oil-free chips, the parts selected for test shall have the oil extracted in accordance with Test Method B 328, if necessary.

10.3 Mechanical Tests—The manufacturer and purchaser shall agree upon a representative number of specimens for mechanical tests.

10.4 At least two samples shall be taken for conductivity measurement from each lot.

11. Inspection

11.1 Unless otherwise specified, inspection of parts supplied on contract shall be made by the purchaser.

12. Rejection and Rehearing

12.1 Parts that fail to conform to the requirements of this specification may be rejected. Rejection should be reported to the producer or supplier promptly, and in writing. In case of dissatisfaction with test results, the producer or supplier may make claim for a rehearing.

13. Certification

13.1 When specified in the purchase order or contract, a producer’s certification shall be furnished to the purchaser that the parts were manufactured, sampled, tested, and inspected in accordance with this specification and have been found to meet the requirements. When specified in the purchase order or contract, a report of the test results shall be furnished.

13.2 The purchase order must specify whether or not the certification includes chemistry.

13.3 Upon request of the purchaser in the contract or order, the certification of an independent third party indicating conformance to the requirements of this specification may be considered.

14. Supplementary Requirements

14.1 Metallographic Examination—When specified in the purchase order or contract, either or both of the following supplementary requirements may be applied. Details of these supplementary requirements shall be agreed upon in writing between the producer or supplier and purchaser. Supplementary requirements shall in no way negate any requirement of the specification itself.

14.1.1 Sintering—Requirements for uniformity and quality of sintering may be agreed upon.

14.1.2 Porosity—Requirements excluding excessively large pores may be included when specified and agreed upon in writing.

15. Keywords

15.1 conductive powder metallurgy; copper powder metallurgy; electrical parts; powder metallurgy; structural copper parts; thermally conductive parts

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X1.1 Typical tensile and electrical conductivity properties of molded and sintered copper specimens are shown in Table X1.1. These data do not constitute a part of this specification. They merely indicate to the purchaser the properties that may be expected from special tension specimens conforming to the specified density and chemical composition requirements. It should be thoroughly understood that the values represent specimens molded to size and not specimens cut from commercial parts or specimens machined from sample blanks. The tension tests are run on flat specimens approximately 1/4 by 1/4 in. in cross section with a gage length of 1 in. For specimen size and test details, refer to Test Methods E 8. The electrical conductivity may be measured on the finished part if it is at least 1/2 in. in diameter. See also Fig. X1.1.5

1. Scope

1.1 This test method covers determination of the microhardness of powder metallurgy (P/M) parts and applications of microhardness test results to methods for determination of the case depth. Technique for measurement of case depth of P/M parts by observation is also outlined.

1.2 Part A: Microhardness Measurement—This procedure covers test methods to determine the microhardness of P/M parts with the Knoop (HK) or the Vickers (HV) indenters. Procedures for surface preparation of the P/M material prior to microhardness measurement are included.

1.3 Part B: Case Depth Measurement—Procedures and methods for determination of both effective case depth and observed case depth for P/M parts are included. The principles of Part A on Microhardness Measurement are utilized to measure case depth.

1.4 The values stated in inch-pound units are to be regarded as the standard. The values in parentheses are for information only.

1.5 This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 243 Terminology of Powder Metallurgy
E 384 Test Method for Microhardness of Materials

3. Terminology

3.1 Definitions of powder metallurgy (P/M) terms can be found in Terminology B 243. Additional descriptive information is available in the Related Material section of Vol. 02.05 of the Annual Book of ASTM Standards.

4. Summary of Test Method

4.1 Part A: Microhardness is measured by using a calibrated machine to force a diamond indenter of specific geometry, under a known test load, into the surface of the test material.

4.1.1 Ordinarily, the impression of the indenter is measured optically and correlated with available tables to obtain a value in the desired hardness scale; or, the optical measurement can be used to calculate a hardness number.

NOTE 1—This test method is designed specifically for use on P/M parts. It is intended to be a companion to Test Method E 384. There are specific differences that are intentional; otherwise the details on equipment and procedures in Test Method E 384 shall be adhered to.

4.1.2 A new technique, on direct HRC equivalent, is recommended as part of the test method. It involves constructing a plot of microhardness equivalent (HRC) versus the measured diagonal length of the impression made in the material by the indenter. This master plot can then be used to obtain an HRC equivalent directly from the optical measurement.

4.2 Part B: Case Depth is defined as the distance from the surface of a part to a point below the surface where:

4.2.1 There is a drop in hardness below some prescribed level,

4.2.2 There is a divergence from a linear decrease in hardness as a function of the distance from the surface, or

4.2.3 There is an easily seen transition in metallurgical structure.

5. Significance and Use

5.1 Especially for P/M materials, microhardness is useful for determination of the actual hardness of the metal matrix. Also, in most metallic materials, microhardness can be used to differentiate between metallurgical phases and non-metallic inclusions. Of particular interest, microhardness tests can be used to determine the actual hardness of the case in surface hardened materials and to help define the useful thickness of such cases.

5.2 Cases, hardened layers, are used on P/M parts and metal parts produced by other methods to provide required properties economically. Proper engineering function of the case requires proper hardness and a specified thickness.

6. Test Specimens

6.1 Specimen Mounting:

6.1.1 Mounting is recommended for convenience in surface preparation, edge retention, and testing. The specimen should be adequately supported in the mounting medium.
6.1.2 Edge retention is important to proper depth measurement of the case. The mounting material must be selected to provide good edge retention and sufficient rigidity so that no movement of the specimen can occur during the application of load.

6.1.3 At sample densities below 6.6 g/cm³ it is advisable to vacuum impregnate the specimen with a suitable resin or epoxy to support the structure.

7. Surface Preparation

7.1 Surface preparation is critical to obtaining sound microhardness measurements. With the inherent porosity of most P/M materials it is essential in surface preparation to remove all smeared metal and clearly identify the pores so that they can be avoided during placement of the hardness drops and enhance optical determination that the indent is completely contained within solid structure. Recommended procedures for surface preparation are presented in Appendix X1.

8. Procedure

8.1 Part A—Microhardness Measurement:

8.1.1 A microindentation hardness test is made using a calibrated machine to force a diamond indenter of specific geometry, under a test load of 100 gf (0.9807 N) into the surface of the test material and to measure the diagonal or diagonals optically. Optional loads are acceptable only upon agreement between customer and producer.

8.1.1.1 Test Method E 384 presents requirements of the testing machine and optical system for hardness indent measurement.

8.1.2 A direct HRC equivalent hardness value is obtained by measurement of the 100 gf (0.9807 N) Knoop or Vickers diagonal length and selecting the corresponding HRC value from an appropriate plot of HRC versus filar unit diagonal length. It is assumed that the indentation is an imprint of the undeformed indenter.

8.1.3 The HRC microhardness versus Knoop or Vickers filar unit diagonal graph must be individually constructed. Four HRC standard blocks selected over the range 20 to 65 HRC are metallographically mounted, measured with the selected microhardness indenter at 100 gf (0.9807 N) and the appropriate diagonal measurement graphed in terms of HRC value. A straight line is drawn between these points and future measurements read directly in HRC value from intersection of the indent diagonal value with the constructed line. (Fig. 1 is an example of this graph.)

8.2 Part B—Case Depth Measurement:

8.2.1 The test procedure covers determination of case depth in powder metal parts utilizing the prescribed principles presented in Part A when the measurements are taken on a properly prepared section of the part where that section’s geometric relationship is 90 ± 5° to the surface of the part.

8.2.2 Case depth will be defined as either effective or observed case depth. Effective case depth is determined by measurement of microhardness at a series of known distances from the part surface to a designated hardness level. Observed case depth is determined by measurement of the distance from the surface of the metallographically observed case to core transition zone structure.

8.2.3 Effective Case Depth Procedure:

8.2.3.1 At a determined distance from the part surface, make a minimum of three acceptable measurements. Repeat this procedure at incremental distances from the part surface maintaining a distance between impressions of at least 2.5 times the width of the smallest diagonal. Measurements obviously deformed due to underlying void should be discarded. Indentations should not be placed in soft phases such as copper or the centers of nickel-rich austenite regions. Randomly encountered fine pearlite in the martensite should not be excluded as a measurement location. A lightly etched surface is helpful in defining these regions, such as etching for about 6 to 7 s in 2 % Nital.

8.2.3.2 Effective case depth will be determined by the distance from the surface, beyond which, the hardness is below 50 HRC or an agreed upon value. When the hardness versus depth relationship is graphed, effective case depth will be at the divergence point in the linear microhardness to surface depth relationship indicated in Fig. 2.
8.2.3.3 Effective case depth determined by variance from a customer-producer agreed upon value, which is often 50 HRC, will be the distance from the part surface to the point where the microhardness falls below that specified value on a graph of hardness versus depth relationship. The microhardness will be the average value of three acceptable impressions and the case depth the average distance those impressions lie from the part surface.

8.2.3.4 Effective case depth determined by divergence in the linear microhardness to surface distance relationship will be resolved by plotting the average value of three acceptable impressions taken at incremental distance from the part surface versus the average distance of the three averaged impressions from the surface. The effective case depth determined by this technique will be the second point at which the microhardness diverges from the linear relationship with surface depth as illustrated in Fig. 2. A vertical line from that point of divergence to the x axis of the plot will determine the case depth and a horizontal line from the point of divergence to the y axis will determine the microhardness of the case hardened structure.

8.2.4 Observed Case Depth Procedure:

8.2.4.1 In those materials where a metallurgically determined transition zone between case and core structure can be resolved at magnifications of 50 to 100× the case depth will be determined by measurement of the distance of the part surface to the beginning edge of that zone. The beginning of the transition from case to core is characterized by the appearance of fine pearlite colonies mixed in with the martensite. Using a visual estimate, the distance in from the surface where approximately 5% of the area is fine pearlite shall be defined as the case depth.

9. Report

9.1 The report shall include:
9.1.1 The method of microhardness measurement: HK, HV, HRC/HK, or HRC/HV. In each case, the load used in testing shall be expressed as a subscript, for example, HK\textsubscript{100}.
9.1.2 The method of case depth measurement.
9.1.3 The case depth values.
9.1.3.1 The effective case depth is the distance from the part surface at which the measured value falls below the specified microhardness value or the microhardness value and distance from the part surface that divergence from the linear microhardness to surface distance relationship occurs.
9.1.3.2 For observed case depth the distance from the part surface up to, but not including the case to core transition zone, the microhardness of the case measured at the case edge of that transition zone, and the magnification at which the measurement was taken.

10. Precision and Bias

10.1 Precision — Using individual regression lines based upon six-reading averages for each of five HRC test blocks ranging from 25.4 HRC to 63.2 HRC seven laboratories found values of a circulated unknown to average 56.5 HRC. With this method 95% of any future readings would be expected to repeat in a laboratory within ± 4.0 HRC points at this level; for six-reading averages, within ± 1.6 HRC points. For a laboratory to duplicate any of the other laboratories, 95% of the readings should be within ± 5.3 HRC; for six-reading averages, within ± 2.2 HRC.

10.2 Bias — No bias can be defined since there is no way to define true hardness, and therefore any innate deviation from true hardness.

APPENDIX

(Nonmandatory Information)

XI. SAMPLE PREPARATION

X1.1 The methods described in this Appendix are proven practices for metallographic preparation of a microhardness sample. It is recognized that other procedures or materials used in preparation of a sample may be equally as good and can be used on the basis of availability and preference of individual laboratories.

X1.2 Method 1:

X1.2.1 The porous samples should be free of oil or cut-off fluid, using Soxhlet extraction if needed. The samples are then vacuum impregnated with, and mounted in, epoxy resin, to fill porosity and to prevent pick-up of etchants. Using a sample cup or holder to form the mount, a ¾-in. (19 mm) deep layer of epoxy resin is poured over the sample in the cup. The cup is evacuated to ~26 in. Hg (100 Torr) and held at that pressure for 10 min. Ambient air pressure is restored, forcing the resin into most of the sample. Curing can be done at room temperature or accelerated at 50°C.

X1.2.2 The cured mounts are ground on 240, 400, and 600 mesh wet SiC paper, on a rotating wheel, with the polishing direction changed 90° after each paper. Samples are etched for 1 min. in their normal etchant, for example, 2% Nital, to begin to open the porosity. Rough polishing opens smeared pores: 8 to 12 min total on 1 µm alumina (Al\textsubscript{2}O\textsubscript{3}), long napped cloth (for example, Struers felt cloth), 250 r/min, 300 g load, automatic polisher. This polishing opens and exaggerates the pores. The pores are then returned to their true area fraction of porosity by polishing for 4 min at 125 r/min on shorter nap cloth (for example, Struers MOL cloth), with 1 µm diamond paste. Final polishing is done for 20 to 30 s on 0.05 µm deagglomerated alumina, long napped cloth (for example, Buehler Microcloth), 125 r/min, 75 g load, automatic polisher. Polishing may also be done by hand, for the times indicated. The first two polishings require moderate pressure and the final polish requires light pressure.

X1.2.3 The metallographic structure should be free of...
smeared porosity. Generally, at 800 to 1000×, the edge of a smeared over pore will appear as a thin grey line outlining one side of the pore, and occasionally outlining most of the pore.

X1.3  Method 2:

X1.3.1 The specimen should be carefully selected so that it is representative of the region of interest. After selection, the specimen may require sectioning to provide a workable specimen. Sectioning may be made employing a hacksaw, band saw, abrasive, or diamond wheel. For soft materials a hacksaw is sufficient; however, if harder materials are of interest, then an abrasive or diamond wheel may be required.

X1.3.2 Heat should be avoided to prevent occurrence of possible changes in microstructure. If slow feeds are employed, a coolant may not be necessary to avoid temperature build-ups. If abrasive wheels are used, then often a coolant is necessary to prevent heating of the specimen.

X1.3.3 If a coolant is employed, it may be retained within pores. The lubricant must be removed prior to preparation of the specimen for microexamination. This may be accomplished by using a Soxhlet extractor or an ultrasonic cleaner. The extraction condenser is most efficient and least expensive.

X1.3.4 Generally, specimens to be evaluated for microhardness are mounted to provide edge retention. There are many kinds of mounting compounds available. Most common materials include epoxide or bakelite. Of the two, bakelite is preferred because it is harder and therefore provides improved edge retention. Bakelite requires equipment to apply heat and pressure, whereas the epoxides do not.

X1.3.5 After mounting, the specimen is ground to provide a flat, stress-free surface. A belt grinder is generally used first with care to prevent heating of the specimen. Grit size is dependent upon the preference of the metallographer, although finer grits are preferred.

X1.3.6 The specimen is then hand ground on four emery papers, generally of 240, 320, 400, and 600 grit.

X1.3.7 Hand grinding is followed by wet polishing. Several polishing media are employed including diamond paste, magnesium alumina, etc. Grit size varies between 1 and 0.3 µm and is applied to nap-free cloths, such as nylon. To remove remaining scratches and stress, a soft cloth with finer polishing compound is employed. Generally a short napped cloth is preferred. A fine, 0.5 µm alumina is recommended. For best results and to ensure complete freedom of pores from worked metal, repeat the polishing and etching procedure. Final polishing generally requires 3 to 5 min.

X1.3.8 Automated polishing equipment is also available. Automated polishing is accomplished by moving the specimen across a polishing cloth in an abrasive solution undergoing vibrating action. Cloths and abrasives available are numerous and are generally selected by experience of the metallographer.

4 For more specific details concerning specimen mounting and grinding procedures, consult Kehl, “The Principles of Metallurgical Laboratory Practice,” ASM Handbook, American Society of Metals, or other texts containing instructions on prescribed metallographic practice.
LastB-727—83 (1995) standard and is intended only to provide the user of an ASTM standard an indication of what changes have been made to the previous version. Because it may not be technically possible to adequately depict all changes accurately, ASTM recommends that users consult prior editions as appropriate. In all cases only the current version of the standard as published by ASTM is to be considered the official document.
1. Scope
1.1 This practice is a guide to the surface preparation of plastic materials for decorative and functional electroplating, where the sequence of chemical treatments may include: cleaning, conditioning, etching, neutralizing, catalyzing, accelerating, and autocatalytic metal deposition. Surface preparation also includes electrodeposition of metallic strike coatings immediately after autocatalytic metal deposition. These treatments result in the deposition of thin conductive metal films on the surface of molded-plastic materials, and are described in this practice.

1.2 Once molded-plastics materials have been made conductive, they may be electroplated with a metal or combination of metals in conventional electroplating solutions. The electroplating solutions and their use are beyond the jurisdiction scope of this practice.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. (See Section 4.)

2. Referenced Documents
2.1 ASTM Standards:
B 532 Specification for Appearance of Electroplated Plastic Surfaces

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3. Significance and Use

3.1 A variety of metals can be electrodeposited on plastics for decorative or engineering purposes. The most widely used coating consists of three layers—copper plus nickel plus chromium—for decorative applications. However, brass, silver, tin, lead, cadmium, zinc, gold, other metals, and combinations of these are used for special purposes. The key to producing electroplated plastics of high quality lies in the care taken in preparing plastics for electroplating. The information contained in this practice was withdrawn is useful in January 2004 controlling processes for the preparation of plastics for electroplating.

4. Hazards

4.1 Some chemical solutions are exothermic upon mixing or in use, thereby requiring cooling and proper containment to prevent injury to personnel.

4.2 For details on the proper operation and safety precautions to be followed by vapor degreasing, see ASTM STP 310.4

5. General Considerations

5.1 Nature of Plastics Suitable for Electroplating:

5.1.1 Plastics suitable for electroplating may be a combination of one or more polymers so formulated as to allow selective etching of one or more constituents. The most commonly electroplated material, acrylonitrile-butadiene-styrene (ABS), is a terpolymer. During etching, soft butadiene rubber particles dispersed in the acrylonitrile-styrene matrix are selectively attacked. The microscopic pockets formed by the etching process provide sites for the physical interlocking of the plastic substrate and subsequently applied metallic coats. The resultant mechanical bonding is instrumental in achieving metal to plastic adhesion.

5.2 Plastics Suitable for Electroplating:

5.2.1 The plastics materials commonly used for injection molded articles to be electroplated are:

5.2.1.1 Acrylonitrile-butadiene-styrene (ABS),
5.2.1.2 Polypropylene,
5.2.1.3 Polysulfone,
5.2.1.4 Modified Polyphenylene Oxide,
5.2.1.5 Polycarbonate,
5.2.1.6 Polyester, and
5.2.1.7 Nylon.

5.2.2 The preparation of these materials for electroplating generally involves the basic steps described in this practice, but substantial variations may be necessary to achieve optimum results with section 10.6.3.1 plastics other than ABS.

5.3 Molding Considerations:

5.3.1 The chemical nature of plastics combined with the nature of the injection molding process produces plastic parts that are somewhat heterogeneous in composition and structure. During the molding of ABS, for example, the shape, size, and distribution of butadiene rubber particles may vary considerably within a part and may affect the uniformity of subsequent surface etching treatments. As a result, under- and over-etching of the surface may occur, either of which can interfere with the adhesion of metal coatings. The use of a properly formulated etchant combined with an organic conditioner may overcome problems of nonuniform etching.

5.3.2 Although it may be possible to overcome problems of nonuniform etching by suitable chemical treatments, control of the injection molding process is critical if plastic parts are to be electroplated successfully. It is essential that the resin be thoroughly dried before molding. The temperature of the mold and all heating zones, the pressure, the total cycle time, and the fill time must be controlled and monitored. Devices exist for controlling all molding parameters precisely.

5.3.3 The visible defects that may arise in the molding process are described in Specification B 532. Molded parts that are obviously defective should not be processed without the approval of the purchaser.

5.3.4 Mold release agents interfere with the adhesion of metallic coatings on plastic substrates and should not be used.

5.4 Process Selection:

5.4.1 Due to the complexity and proprietary nature of commercially available processes for preparing plastics for electroplating, a complete process should be selected for a specific type of plastic, and operated according to the specific instructions of the supplier of the process.

5.5 Handling of Molded Plastic Parts:

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2 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard’s Document Summary page on the ASTM website.

3 Withdrawn.

5.5.1 Molded-plastic parts must be kept clean and carefully handled. It is a common practice to use cotton gloves in removing parts from the mold and for all subsequent handling.

5.5.2 The trimming of plastic parts and the removal of flash and runners should be done with care to avoid introducing surface defects. These and other mechanical finishing operations should be completed before beginning the chemical treatment of parts for electroplating. Runners are sometimes left intact to facilitate racking for electroplating at a later stage.

5.6 Racking:

5.6.1 Molded-plastic parts can be prepared for electroplating in barrels, trays, or baskets and then transferred to racks designed specifically for electroplating, or they can be processed on racks that are used in both the preparation and electroplating cycles. Which method of racking to use may be dictated by the size of the parts, by efficiency, and other considerations. The first is the bulk method; the second is called “through-racking.”

5.6.2 Bulk Method—Small parts are often processed in polypropylene baskets or plastic-coated steel baskets. Somewhat larger parts can be processed in layered baskets made of stainless steel (UNS Types S30400 or S31600), titanium, or plastic-coated mild steel. Parts are placed as closely as possible compatible with the need to provide for complete solution wetting and drainage.

5.6.3 Through-Racking:

5.6.3.1 The design of racks to be used in both preparation and electroplating processes is dictated by the requirements of electroplating and the corrosive nature of the solutions.

5.6.3.2 Rack splines and hooks are generally made of copper or copper alloys. Rack cross bars are made of copper or copper alloys if they are to conduct current from the splines to the contacts, but may be made of steel if their function is solely to strengthen and make the rack rigid. Rack contacts are usually stainless steel, although titanium has also been used. If spring action is necessary, phosphor bronze may be used as the contact member with a short stainless steel piece for the tip.

5.6.3.3 The entire rack is sandblasted, primed, and coated with plastisol before use, except for the stainless steel contacts. During the preparation process, the rack coating may become coated with metal, but this does not usually occur because hexavalent chromium is absorbed in the plastisol and prevents autocatalytic metal deposition from occurring.

5.6.3.4 Control of immersion times in neutralizing, catalyzing, and accelerating steps is critical to prevent metal deposition on the rack coating.

5.6.3.5 Parts are positioned on racks to optimize the thickness and appearance of electrodeposited coatings, and to minimize solution dragout.

5.6.3.6 It may be necessary to use current thieves, shields, or auxiliary anodes to obtain uniform metal distribution. The number of contacts is greater for plastic parts than for comparable metal parts. For example, if the total area being electroplated is less than 0.02 m², one contact point is usually sufficient; if the area is 0.25 to 0.60 m², 16 contact points are recommended.

5.6.3.7 Metal deposited autocatalytically or electrolytically must be chemically removed from contacts after each cycle. This is usually accomplished by using nitric acid-containing solutions, or proprietary rack strippers.

6. Preparation of Plastic Substrates

6.1 Alkaline Cleaning:

6.1.1 Cleaning in alkaline solutions is optional. If the parts are carefully handled and kept clean after molding, alkaline cleaning can usually be avoided.

6.1.2 Fingerprints, grease, and other shop soil should be removed by soaking plastic-molded parts in mild alkaline solutions that are commercially available. A suitable solution may contain 25 g/L of sodium carbonate and 25 g/L of trisodium phosphate operated at 55 to 65°C. Parts are immersed in the solution for 2 to 5 min (see Note 1).

NOTE 1—Thorough rinsing after alkaline cleaning and after each of the following processing steps is essential. Multiple water rinses are recommended.

6.2 Conditioning:

6.2.1 Conditioning is an optional step that precedes the etching step. Conditioning can eliminate adhesion problems associated with inadequate etching. The conditioner may be a solution of chromic and sulfuric acids, or it may contain an organic solvent. Proprietary solutions are available and should be operated according to supplier’s directions.

6.2.2 Chromic/Sulfuric Acid Type—This type of conditioner may contain 30 g/L of chromic acid and 300 mL/L of sulfuric acid (93 mass %; density 1.83 g/mL) dissolved in water and is maintained at a temperature of 60°C ± 3°C. Parts are immersed in the solution for 1 to 2 min. Because of the relatively large amount of sulfuric acid in the solution, the acrylonitrile-styrene matrix, as well as the butadiene phase, are attacked.

6.2.3 ROrganic Solvent Type—This type of conditioner is a solution of an organic solvent in deionized water. The organic solvent may be acetonitrile or any ketone; for example, 2,4-pentandione is sometimes used. The solution may contain 100 to 125 mL/L of the appropriate organic solvent and is maintained at a temperature of 40 to 45°C. Treatment is by immersion of the plastic parts for 2.0 to 2.5 min (see Note 2 and Note 3).

NOTE 2—Solutions containing volatile organic solvents require adequate ventilation and must not contact metals. These materials chelate ionic metal...
contaminants. Annealed polypropylene tanks are therefore used to hold this type of solution.

**NOTE 3**—Multiple hot water rinses are required after using the organic solvent-type conditioner. Because organic solvents soften and swell the plastic surface, time of immersion and of transfer to rinse tanks may affect the appearance of the final product, and should be controlled.

**6.3 Etching:**

6.3.1 Etchants are strong oxidizing solutions that microroughen and chemically alter the surface of molded plastic parts. The etching step is the most important step in achieving serviceable adhesion of metals to plastics. Commercially used etchants are either chromic acid types, chromic/sulfuric acid types, or chromic-sulfuric-phosphoric acid types.

6.3.2 **Chromic Acid Type**—Concentrated etchants usually contain greater than 850 g/L of chromic acid and as much as 1200 g/L. The temperature of the solution is maintained at 50 ± 3°C and treatment is by immersion for 8 to 10 min. Concentrated solutions of chromic acid tend to oxidize butadiene rubber particles in the case of ABS, selectively.

6.3.3 **Chromic/Sulfuric Acid Type**—This type of etchant may contain 250 to 350 g/L of chromic acid and 200 to 250 mL/L of sulfuric acid (93 mass %, density 1.83 mL/L) dissolved in water. Immersion times of 5 to 10 min at a solution temperature of 65 ± 5°C are commonly used. Several proprietary baths are available.

6.3.4 **Chromic-Sulfuric-Phosphoric Acid Type**—This type of etchant solution normally consists of 3 % by mass chromic acid, 56 % by mass sulfuric acid (density 1.83 g/mL), 10.5 % by mass phosphoric acid (density 1.87 g/mL), and the balance water. An immersion time of about 3 min at 74 to 77°C is commonly used.

**6.4 Neutralizing (Sensitizing):**

6.4.1 After thorough rinsing, all residual chromic acid must be chemically removed from the surface of the molded-plastic parts. Neutralizers are used and are typically mild acid or alkaline solutions containing complexing or reducing agents. In the case of ABS, it is common to use a solution containing a mixture of an acid salt and a reducing agent, such as sodium bisulfite, to eliminate all traces of chromic acid. Typical processing conditions are 1 to 2 min immersion at 40°C.

6.4.2 Neutralizers may also contain ionic surfactants to increase the adsorption of catalyst. The use of surfactants, however, can lead to activation of the rack coating and subsequent metal deposition on the rack. Surfactants should therefore be used with caution. Ionic surfactants are not normally used in processing ABS (see Note 4).

**NOTE 4**—Some plastics, for example, polyphenylene oxide, may require treatment in dilute solutions of ethylenediamine after neutralization to insure adequate adsorption of the catalyst.

**6.5 Catalyzing (Activating):**

6.5.1 Small amounts of palladium are chemically deposited on the surface at this stage of processing. Palladium functions as a catalyst for autocatalytic deposition of copper or nickel to follow. Palladium is deposited either by the older, two-step procedure or by the more reliable one-step procedure.

6.5.2 **Two-Step Procedure**—The molded-plastic parts are first immersed in a solution of stannous chloride, 6 to 10 g/L, operated at pH 1.8 to 2.4 (pH adjusted with hydrochloric acid), and at 20 to 25°C for 1 to 3 min. The parts are then rinsed thoroughly to remove excess hydrochloric acid and transferred to a solution containing 0.1 to 1.0 g/L of palladium chloride dissolved in water at a pH of 1.6 to 2.0 adjusted with sulfuric acid. The palladium ions adsorbed on the surface react with stannous ions to form palladium metal and stannic chloride in the interstices of the etched plastic components. After thorough rinsing, the parts can be coated with nickel or copper by autocatalytic deposition.

6.5.3 **One-Step Procedure**—The molded-plastic parts are immersed in a solution of colloidal stannous chloride and palladium chloride containing excess hydrochloric acid. The stannous chloride concentration is 120 to 140 g/L, the palladium metal concentration is 0.05 to 0.15 g/L, and the hydrochloric acid concentration is approximately 3.0 N (see Note 5). The solution is maintained at 20 to 30°C and the parts are immersed in the solution for 1 to 3 min. Rinsing in water leads to the formation of metallic palladium nuclei on the surface surrounded by stannic hydroxide. The stannic hydroxide is removed in the acceleration step prior to autocatalytic deposition.

**NOTE 5**—These solutions are proprietary and considerably more difficult to prepare than implied in this section.

**6.6 Acceleration:**

6.6.1 Stannous hydroxide is removed by treatment in a dilute solution of hydrochloric acid, or a solution of an acid salt. Fluoride or fluorinated compounds are often added to increase the effectiveness of the acceleration process. In most cases, a solution containing 1.0 N hydrochloric acid maintained at a temperature of 50°C, agitated with air, in which parts are immersed for 30 to 60 s adequately removes excess stannous chloride and stannic/stannous hydroxide remaining on the surface after the one-step palladium activation procedure.

**6.7 Autocatalytic Deposition:**

6.7.1 Solutions for the autocatalytic deposition of either copper or nickel are used to render plastic parts conductive. The solutions contain a metal salt, a reducing agent, a complexant, a stabilizer, and buffers to control pH. The palladium on the surface...
of the plastic parts acts as a catalyst to initiate deposition after which the autocatalytic reduction of the metal occurs. A uniform metal film about 0.25 to 0.5-µm thick is deposited (see Note 6).

6.7.2 Autocatalytic Nickel—The commercially available processes for autocatalytic nickel deposition on plastics commonly use sodium hypophosphite as the reducing agent. The solutions are operated at 30 to 35°C, at pH 10 to 11, and produce a nickel deposit with 2 to 6 % phosphorous. Parts are kept immersed in the solution for 5 to 10 min to achieve the desired thickness of metal. Although there may be considerable variation in bath formulations, autocatalytic nickel solutions may contain nickel sulfate, sodium citrate, ammonium chloride, ammonium hydroxide, sodium hypophosphate, and sodium hydroxide. Stabilizers are used to prevent spontaneous decomposition of the solution and to control the deposition rate.

6.7.3 Autocatalytic Copper—The commercially available processes for autocatalytic deposition of copper on plastics use formaldehyde as the reducing agent. Room temperature processes are used, as well as high temperature ones. Each produce deposits of pure copper. Parts are immersed in the solution for 5 to 10 min.

**Note 6**—Autocatalytic nickel or copper is used commercially in the preparation of plastics, and either may give acceptable results. The use of autocatalytic copper, however, may improve the performance of electroplated plastics in wet corrosive environments. Parts that are inadequately etched or that are made from grades of plastics that are difficult to etch are less likely to fail in severe corrosive environments when autocatalytic copper is used.12,13,14

6.8 Electrodeposited Strikes:

6.8.1 Additional thicknesses of either copper or nickel are applied by low-current density electrodeposition from suitable strike baths. Metal thickness is increased to 2.5 to 4.0 µm to facilitate electrodeposition of decorative or functional coatings by conventional means.

6.8.2 Typical copper and nickel strike solutions are given in Table 1.

6.8.3 The most commonly applied decorative coating consists of layers of copper, nickel, and chromium, but other metals may be combined to achieve decorative effects.

### 7. Process Control

#### 7.1 General:


<table>
<thead>
<tr>
<th>Table 1 Typical Strike Solution Compositions</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Copper Strike:</strong></td>
</tr>
<tr>
<td>Pyrophosphate Type:</td>
</tr>
<tr>
<td>- Copper (Cu)</td>
</tr>
<tr>
<td>- Pyrophosphate (P₂O₇⁻₄)</td>
</tr>
<tr>
<td>- Nitrate (NO₃⁻)</td>
</tr>
<tr>
<td>- Ammonia (NH₃)</td>
</tr>
<tr>
<td>- pH</td>
</tr>
<tr>
<td>- Temperature</td>
</tr>
<tr>
<td>- Voltage, d-c</td>
</tr>
<tr>
<td>- Current density</td>
</tr>
</tbody>
</table>

| Acid Copper Sulfate Type:                   |
| - Copper sulfate (CuSO₄·3H₂O)               | 210–225 g/L     |
| - Sulfuric acid (H₂SO₄)                     | 18–22°C         |
| - Voltage, d-c                             | 2.5–3.0 V       |
| - Current density                          | 250–400 A/m²    |

| **Nickel Strike:**                         |
| Watts Type:                                |
| - Nickel sulfate (NiSO₄·6H₂O)              | 300–340 g/L     |
| - Nickel chloride (NiCl₂·6H₂O)            | 30–60 g/L       |
| - Boric acid (H₃BO₃)                       | 3–4 g/L         |
| - pH                                       | 45–55°C         |
| - Voltage, d-c                             | 2.5–3.0 V       |
| - Current density                          | 250–600 A/m²    |

| Sulfamate Type:                            |
| - Nickel sulfamate                         | 30–45 g/L       |
| - Boric acid (H₃BO₃)                       | 3.0–4.5 g/L     |
| - pH                                       | 45–55°C         |
| - Current density                          | 250–600 A/m²    |

A Usually available as a liquid concentrate.
7.1.1 The pH, temperature, and composition of the solutions used in preparing plastics for electroplating must be carefully controlled to achieve satisfactory results. Conventional analytical and other measuring techniques may be used. Suppliers can provide instructions for control of proprietary processes.

7.2 Etchants:
7.2.1 The concentration of trivalent chromium increases with continuous use of chromic acid-containing etchants. Trivalent chromium reduces the ability of the etchant to oxidize the surface of the plastic. The maximum tolerable concentration of trivalent chromium is 20 g/L in etchants containing about 350 g/L of chromic acid, and 90 g/L in etchants containing 1200 g/L of chromic acid. The trivalent chromium concentration can be adjusted by removing a portion of the end solution and adding proper amounts of chromic acid and water to compensate for quantities removed. Trivalent chromium can also be converted to hexavalent chromium by electrolytic oxidation.

7.3 Catalysts:
7.3.1 The stability of the eighth year since solution used in the one-step procedure for chemically depositing palladium on plastics is dependent on the chloride concentration. The chloride concentration is controlled by additions of hydrochloric acid or sodium chloride.

7.3.2 Hexavalent chromium ions carried over from etchants and from other sources interfere with the functioning of the catalyst.

7.4 Accelerators:
7.4.1 Accelerators are especially sensitive to contamination by hexavalent chromium that may be carried over from the etchant solutions. The mineral acid types can tolerate a maximum of 10 ppm. Acid salt types can tolerate a maximum of 300 ppm. Reducing agents, such as stannous chloride and sodium bisulfite, are sometimes used to convert hexavalent chromium to trivalent chromium and prolong the life of the accelerator.

7.5 Appearance:
7.5.1 The appearance of plastic parts changes during processing and these changes can be used to control the process to some extent:
7.5.1.1 After alkaline cleaning and rinsing, plastic parts should be uniformly covered with a film of water.
7.5.1.2 After conditioning, plastic parts should appear slightly dull.
7.5.1.3 After etching, plastic parts should have lost their original glossy appearance, and should be uniformly wetted with water after rinsing. Variations in surface finish at this stage are usually attributed to variations in stress within the part.
7.5.1.4 Adsorption of the catalyst causes the parts to appear light tan in color. The absence of surface coloring is evidence of improper processing that may lead to incomplete coverage and poor adhesion at later stages.
7.5.1.5 Accelerators cause the parts to be only slightly lighter in color than in the previous step.
7.5.1.6 After autocatalytic deposition, the plastic parts should be uniformly and completely covered by the metallic deposit.

7.6 Adhesion Tests:
7.6.1 A qualitative adhesion test may be used on production parts to judge the effectiveness of the preparation process. After autocatalytic deposition, rinsing, and drying, apply a strip of pressure-sensitive tape smoothly on the surface pressing the tape firmly to eliminate all air bubbles. Quickly pull the tape off at a 90° angle. Appearance of metal particles on the tape indicates poor surface preparation.

7.6.2 Test Method B 533 provides methods for the measurement of the peel strength of electroplated plastics using standard plaques. The methods can be used to monitor the effectiveness of the preparation process and changes that occur during use. Standard plaques available from various sources are processed and then electroplated with 35 ± 5 µm of copper or nickel. The peel strength can be measured in various ways as discussed in Test Method B 533. Typical values of peel strength are given in Table 2.

7.6.3 The thermal cycle tests described in Test Method B 553 and in Specification B 604 can be used to evaluate the adhesion of metals electrodeposited on plastics and thus, the effectiveness of the entire process, including the chemical preparation treatments covered in this practice.

---

<table>
<thead>
<tr>
<th>Type of Plastic</th>
<th>Peel Strength, a N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper</td>
<td>Nickel</td>
</tr>
<tr>
<td>ABS</td>
<td>40–110</td>
</tr>
<tr>
<td>Modified polyphenylene oxide</td>
<td>10–50</td>
</tr>
<tr>
<td>Polypropylene</td>
<td>20–110</td>
</tr>
<tr>
<td>Impact styrene</td>
<td>10–20</td>
</tr>
<tr>
<td>Polysulfone</td>
<td>20–100</td>
</tr>
<tr>
<td>Polycarbonate</td>
<td>10–50</td>
</tr>
</tbody>
</table>

a Measured on 25-mm wide strips. The thickness of the electrodeposited metal was 35 ± 5 µm, as recommended in Test Method B 533.
8. Keywords

8.1 activation; cleaning; deoxidizing; plastics; preparation; striking
Standard Specification for Autocatalytic (Electroless) Nickel-Phosphorus Coatings on Metal

1. Scope

1.1 This specification covers requirements for autocatalytic (electroless) nickel-phosphorus coatings applied from aqueous solutions to metallic products for engineering (functional) uses.

1.2 The coatings are alloys of nickel and phosphorus produced by autocatalytic chemical reduction with hypophosphite. Because the deposited nickel alloy is a catalyst for the reaction, the process is self-sustaining. The chemical and physical properties of the deposit vary primarily with its phosphorus content and subsequent heat treatment. The chemical makeup of the plating solution and the use of the solution can affect the porosity and corrosion resistance of the deposit. For more details, see ASTM STP 265 (1) and Refs (2) (3) (4) and (5) also refer to Figs. X1.1, Figs. X1.2, and Figs. X1.3 in the Appendix of Guide B 656.

1.3 The coatings are generally deposited from acidic solutions operating at elevated temperatures.

1.4 The process produces coatings of uniform thickness on irregularly shaped parts, provided the plating solution circulates freely over their surfaces.

1.5 The coatings have multifunctional properties, such as hardness, heat Hardenability, abrasion, wear and corrosion resistance, magnetics, electrical conductivity provide diffusion barrier, and solderability. They are also used for the salvage of worn or mismatched parts.

1.6 The low phosphorous (2 to 4 % P) coatings are microcrystalline and possess high as-plated hardness (620 to 750 HK 100). These coatings are used in applications requiring abrasion and wear resistance.

1.7 Lower phosphorus deposits in the range between 1 and 3 % phosphorus are also microcrystalline. These coatings are used in electronic applications providing solderability, bondability, increased electrical conductivity, and resistance to strong alkali solutions.

1.8 The medium phosphorous coatings (5 to 9 % P) are most widely used to meet the general purpose requirements of wear and corrosion resistance.

1.9 The high phosphorous (more than 10 % P) coatings have superior salt-spray and acid resistance in a wide range of applications. They are used on beryllium and titanium parts for low stress properties. Coatings with phosphorus contents greater than 11.2 % P are not considered to be ferromagnetic.

1.10 The values stated in SI units are to be regarded as standard.

1.11 The following precautionary statement pertains only to the test method portion, Section 9, of this specification. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 368 Test Method for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Testing) 3
B 374 Terminology Relating to Electroplating 3
B 380 Test Method of Corrosion by the Corrodkote Procedure 3
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section 3
B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method 3
B 537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure 3
B 567 Method for Measurement of Coating Thickness by the Beta Backscatter Method 3
B 568 Method for Measurement of Coating Thickness by X-Ray Spectrometry 3

3 Annual Book of ASTM Standards, Vol 02.05.
4. Coating Classification

4.1 The coating classification system provides for a scheme to select an electroless nickel coating to meet specific performance requirements based on alloy composition, thickness and hardness.

4.1.1 TYPE describes the general composition of the deposit with respect to the phosphorus content and is divided into five categories which establish deposit properties (see Table 1).

Note 1—Due to the precision of some phosphorus analysis methods a deviation of 0.5 % has been designed into this classification scheme. Rounding of the test results due to the precision of the limits provides for an effective limit of 4.5 and 9.5 % respectively. For example, coating with a test result for phosphorus of 9.7 % would have a classification of TYPE I.

3.2 Other Definitions—Terminology B 374 defines most of the technical terms used in this specification.

3. Terminology

3.1 Definition:

3.1.1 significant surfaces—those substrate surfaces which the coating must protect from corrosion or wear, or both, and that are essential to the performance.

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4.2 Service Condition Based on Thickness:

4.2.1 Service condition numbers are based on the severity of exposure in which the coating is intended to perform and minimum coating thickness to provide satisfactory performance (see Table 2).

4.2.1 SC0 Minimum Service, 0.1 µm—This is defined by a minimum coating thickness to provide specific material properties and extend the life of a part or its function. Applications
include requirements for diffusion barrier, undercoat, electrical conductivity and wear and corrosion protection in specialized environments.

4.2.3 **SC1 Light Service**, 5 µm—This is defined by a minimum coating thickness of 5 µm for extending the life of the part. Typical environments include light-load lubricated wear, indoor corrosion protection to prevent rusting, and for soldering and mild abrasive wear.

4.2.4 **SC2 Mild Service**, 13 µm—This is defined by mild corrosion and wear environments. It is characterized by industrial atmosphere exposure on steel substrates in dry or oiled environments.

4.2.5 **SC3 Moderate Service**, 25 µm—This is defined by moderate environments such as non marine outdoor exposure, alkali salts at elevated temperature, and moderate wear.

4.2.6 **SC4 Severe Service**, 75 µm—This is defined by a very aggressive environment. Typical environments would include acid solutions, elevated temperature and pressure, hydrogen sulfide and carbon dioxide oil service, high-temperature chloride systems, very severe wear, and marine immersion.

**Note 2**—The performance of the autocatalytic nickel coating depends to a large extent on the surface finish of the article to be plated and how it was pretreated. Rough, non uniform surfaces require thicker coatings than smooth surfaces to achieve maximum corrosion resistance and minimum porosity.

4.3 **Post Heat Treatment Class**—The nickel-phosphorus coatings shall be classified by heat treatment after plating to improve coating adhesion and or hardness (see Table 3).

4.3.1 **Class 1**—As-deposited, no heat treatment.

4.3.2 **Class 2**—Heat treatment at 260 to 400°C to produce a minimum hardness of 850 HK100.

4.3.3 **Class 3**—Heat treatment at 180 to 200°C for 2 to 4 h to improve coating adhesion on steel and to provide for hydrogen embrittlement relief (see section 6.6).

4.3.4 **Class 4**—Heat treatment at 120 to 130°C for at least 1 h to increase adhesion of heat-treatable (age-hardened) aluminum alloys and carburized steel (see Note 3).

4.3.5 **Class 5**—Heat treatment at 140 to 150°C for at least 1 h to improve coating adhesion for aluminum, non age-hardened aluminum alloys, copper, copper alloys and beryllium.

**TABLE 3 Classification of Post Heat Treatment**

<table>
<thead>
<tr>
<th>CLASS</th>
<th>Description</th>
<th>Temperature (°C)</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>No Heat Treatment, As Plated</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Heat Treatment for Maximum Hardness</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>TYPE I</td>
<td>260</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td></td>
<td>285</td>
<td>16</td>
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<tr>
<td></td>
<td></td>
<td>320</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>400</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>TYPE II</td>
<td>350 to 380</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>TYPE III</td>
<td>360 to 390</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>TYPE IV</td>
<td>365 to 400</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>TYPE V</td>
<td>375 to 400</td>
<td>1</td>
</tr>
<tr>
<td>3</td>
<td>Hydrogen Embrittlement and Adhesion on Steel</td>
<td>180 to 200</td>
<td>2 to 4</td>
</tr>
<tr>
<td></td>
<td>Adhesion on Steel</td>
<td>120 to 130</td>
<td>1 to 6</td>
</tr>
<tr>
<td></td>
<td>Age Hardened Aluminum</td>
<td>140 to 150</td>
<td>1 to 2</td>
</tr>
<tr>
<td></td>
<td>Adhesion on Beryllium and Aluminum</td>
<td>300–320</td>
<td>1–4</td>
</tr>
</tbody>
</table>

4.3.6 **Class 6**—Heat treatment at 300 to 320°C for at least 1 h to improve coating adhesion for titanium alloys.

**Note 3**—Heat-treatable aluminum alloys such as Type 7075 can undergo microstructural changes and lose strength when heated to over 130°C.

5. **Ordering Information**

5.1 The following information shall be supplied by the purchaser in either the purchase order or on the engineering drawing of the part to be plated:

5.1.1 **Title**, ASTM designation number, and year of issue of this specification.

5.1.2 **Classification of the deposit by type, service condition, class**, (see 4.1, 4.2 and 4.3).

5.1.3 **Specify maximum dimension and tolerance requirements, if any**.

5.1.4 **Peening, if required** (see 6.5).

5.1.5 **Stress relief heat treatment before plating**, (see 6.3).

5.1.6 **Hydrogen Embrittlement Relief after plating**, (see 6.6).

5.1.7 Significant surfaces and surfaces not to be plated must be indicated on drawings or sample.

5.1.8 **Supplemental or Special Government Requirements** such as, specific phosphorus content, abrasion wear or corrosion resistance of the coating, solderability, contact resistance and packaging selected from Supplemental Requirements.

5.1.9 **Requirement for a vacuum, inert or reducing atmosphere for heat treatment above 260°C to prevent surface oxidation of the coating** (see S3).

5.1.10 **Test methods for coating adhesion, composition, thickness, porosity, wear and corrosion resistance**, if required, selected from those found in Section 9 and Supplemental Requirements.

5.1.11 **Requirements for sampling** (see Section 8).

**Note 4**—The purchaser shall furnish separate test specimens or coupons of the basis metal for test purposes to be plated concurrently with the articles to be plated (see 8.4).
6.3 Stress Relief:

6.3.1 Pretreatment of Iron and Steel for Reducing the Risk of Hydrogen Embrittlement—Parts that are made of steel with ultimate tensile strength of greater than 1000 Mpa (hardness of 31 HRC or greater), that have been machined, ground, cold formed, or cold straightened subsequent to heat treatment, shall require stress relief heat treatment when specified by the purchaser. The basis metal to pass the specified adhesion test (see 9.4 and Test Methods B 571).

6.6.1 Heat treatment shall be performed preferably within 1 h but not more than 3 h of plating on plated after plating of steel parts to reduce the risk of hydrogen embrittlement. In all cases, the duration of the heat treatment shall commence from the time at which the whole of each part attains the specified temperature.

6.6.2 High-strength steel parts with actual tensile strengths greater than 1000 MPa (corresponding hardness values 300 HV10, 303 HB or 31 HRC) and surface hardened parts shall be processed after coating in accordance with Specification B 850.

6.7 Heat Treatment After Plating to Improve Adhesion—To improve the adhesion of the coating to various substrates, the heat treatments in Table 3 should be performed as soon as practical after plating (see 4.3).

6.8 Heat Treatment After Plating to Increase Hardness:

6.8.1 To increase the hardness of the coating a heat treatment of over 260°C is required. Table 3 describes the heat treatment for maximum hardness.

6.8.2 See Appendixes 3 and 4 and Guide B 656; Figs. X1.2 and Figs. X1.3.

6.8.3 A heat treatment at 260°C for greater than 20 h should be used to reduce the loss of surface hardness and strength of some ferrous basis metals. Avoid rapid heating and cooling of plated parts. Sufficient time must be allowed for large parts to reach oven temperature.

Note 7—The length of time to reach maximum hardness varies with the phosphorus content of the deposit. High phosphorus deposits may require longer time or a higher temperature, or both. Individual alloys should be tested for maximum hardness attainable, especially for conditions of lower temperatures and longer times.

7. Requirements

7.1 Process—The coating shall be produced from an aqueous solution through chemical reduction reaction.

7.2 Acceptance Requirements—These requirements are placed on each lot or batch and can be evaluated by testing the plated part.

7.2.1 Appearance:

7.2.1.1 The coating surface shall have a uniform, metallic appearance without visible defects such as blisters, pits, pimples, and cracks (see 9.2).

7.2.1.2 Imperfections that arise from surface conditions of the substrate which the producer is unable to remove using conventional pretreatment techniques and that persist in the coating shall not be cause for rejection (see 6.1). Also, discoloration due to heat treatment shall not be cause for rejection unless special heat treatment atmosphere is specified (see section 5.1.9).

7.2.2 Thickness—The thickness of the coating shall exceed the minimum requirements in Table 2 as specified by the service condition agreed to prior to plating (see 9.3). After coating and if specified, the part shall not exceed maximum dimension on significant surface (see section 5.1.3).

Note 9—The thickness of the coating cannot be controlled in blind or small diameter deep holes or where solution circulation is restricted.

7.2.3 Adhesion—The coating shall have sufficient adhesion to the basis metal to pass the specified adhesion test (see 9.4 and Test Methods B 571).

7.2.4 Porosity—The coatings shall be essentially pore free when tested according to one of the methods of 9.6. The test method, the duration of the test, and number of allowable spots per unit area shall be specified (see section 5.1.10 and 9.6).
7.3 Qualification Requirements—These requirements are placed on the deposit and process and are performed on specimens to qualify the deposit and plating process. The tests for these qualification requirements shall be performed monthly or more frequently.

7.3.1 Composition—Type II, III, IV, V deposits shall be analyzed for alloy composition by testing for phosphorus (see 9.1). The weight percent of phosphorus shall be in the range designated by type classification (see 4.1).

7.3.2 Microhardness—The microhardness of Class 2 deposits shall be determined by Test Method B 578 (Knoop). For Class 2 coatings, the microhardness shall equal or exceed a minimum of 850 (HK100 (or equivalent Vickers) (see 4.3 and 9.5). The conversion of Vickers to Knoop using Tables E 140 is not recommended.

7.3.3 Hydrogen Embrittlement—The process used to deposit a coating onto high strength steels shall be evaluated for hydrogen embrittlement by Test Method F 519.

8. Sampling

8.1 The purchaser and producer are urged to employ statistical process control in the coating process. Properly performed this will ensure coated products of satisfactory quality and will reduce the amount of acceptance inspection.

8.1.1 Sampling plans can only screen out unsatisfactory products without assurance that none of them will be accepted.

8.2 The sampling plan used for the inspection of a quantity of coated parts (lot) shall be Test Method B 602 unless otherwise specified by purchaser in the purchase order or contract (see section 5.1.11 and S.11.1).

Note 10—Usually, when a collection of coated parts (the inspection lot) is examined for compliance with the requirements placed on the parts a relatively small number of samples, the part, is selected at random and inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection sample. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Three standards Test Method B 602, Guide B 697, and Test Method B 762 contain sampling plans that are designed for the sampling inspection of coatings.

Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one for use with tests that are destructive. The purchaser and producer may agree on the plan(s) to be used. If they do not, Test Method B 602 identifies the plan to be used.

Guide B 697 provides a large number of plans and also gives guidance on the selection of a plan. When Guide B 697 is specified, the purchaser and producer need to agree on the plan to be used.

Test Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The last must yield a numerical value and certain statistical requirements must be met. Test Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The purchaser and producer may agree on the plan(s) to be used. If they do not, Test Method B 762 identifies the plan to be used.

An inspection lot shall be defined as a collection of coated parts which are of the same kind, that have been produced to the same specification, that have been coated by a single producer at one time or approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

8.3 All specimens used in the sampling plan for acceptance tests shall be made of the same basis material and in the same metallurgical condition as articles being plated to this specification.

8.4 All specimens shall be provided by the purchaser unless otherwise agreed to by the producer.

Note 11—The autocatalytic nickel process is dynamic and a daily sampling is recommended. For Coatings requiring alloy analysis and corrosion testing weekly sampling should be considered as an option.

9. Test Methods

9.1 Deposit Analysis for Phosphorus:

9.1.1 Phosphorus Determination—Determine mass % phosphorus content according to Practice E 60, Test Methods E 352, or Test Method E 156 on known weight of deposit dissolved in warm concentrated nitric acid.

9.1.2 Composition can be determined by atomic absorption, emission or X-ray fluorescence spectrometry.

Note 12—Inductively coupled plasma techniques can determine the alloy to within 0.5 %. The following analysis wavelength lines have been used with minimum interference to determine the alloy.

Ni 216.10 nm Cd 214.44 nm Fe 238.20 nm
P 215.40 nm Co 238.34 nm Pb 283.30 nm
P 213.62 nm Cr 284.32 nm Sn 198.94 nm
Al 202.55 nm Cu 324.75 nm Zn 206.20 nm

9.2 Appearance—Examine the coating visually for compliance with the requirements of 7.2.1.

9.3 Thickness:

Note 13—Eddy-current type instruments give erratic measurements due to variations in conductivity of the coatings with changes in phosphorus content.

9.3.1 Microscopical Method—Measure the coating thickness of a cross section according to Test Method B 487.

Note 14—To protect the edges, electroplate the specimens with a minimum of 5 µm of nickel or copper prior to cross sectioning.

9.3.2 Magnetic Induction Instrument Method—Test Method B 499 is applicable to magnetic substrates plated with autocatalytic nickel deposits, that contain more than 11 mass % phosphorus (not ferromagnetic) and that have not been heat treated. The instrument shall be calibrated with deposits plated in the same solution under the same conditions on magnetic steel.

9.3.3 Beta Backscatter Method—Test Method B 567 is only applicable to coatings on aluminum, beryllium, magnesium, and titanium. The instrument must be calibrated with standards having the same composition as the coating.

Note 15—The density of the coating varies with its mass % phosphorus content (See Appendix X2).

9.3.4 Micrometer Method—Measure the part, test coupon, or pin in a specific spot before and after plating using a suitable micrometer. Make sure that the surfaces measured are smooth, clean, and dry.

9.3.5 Weigh, Plate, Weigh Method—Using a similar substrate material of known surface area, weigh to the nearest milligram before and after plating making sure that the part or coupon is dry and at room temperature for each measurement.
Calculate the thickness from the increase in weight, specific gravity, and area as follows:

\[
coating\ thickness,\ \mu m = 10 \frac{W}{(A \times D)}
\]

(1)

where:

- \( W \) = weight gain in milligrams,
- \( A \) = total surface area in square centimetres, and
- \( D \) = grams per cubic centimetres (see Appendix X2).

9.3.6 Coulometric Method—Measure the coating thickness in accordance with Test Method B 504. The solution to be used shall be in accordance with manufacturer’s recommendations. The surface of the coating shall be cleaned prior to testing (see Note 14).

9.3.6.1 Calibrate standard thickness specimens with deposits plated in the same solution under the same conditions.

9.3.7 X-Ray Spectrometry—Measure the coating thickness in accordance with Test Method B 568. The instrument must be calibrated with standards having the same composition as the coating.

Note 16—This method is only recommended for deposits in the as-plated condition. The phosphorus content of the coating must be known to calculate the thickness of the deposit. Matrix effect due to the distribution of phosphorus in layers of the coating also effect the measurement accuracy and require that calibration standards be made under the same conditions as the production process.

9.4 Adhesion:

9.4.1 Bend Test (Test Methods B 571)—A sample specimen is bent 180° over a mandrel diameter 4× the thickness (10 mm minimum) of the specimen and examined at 4× power magnification for flaking or separation at the interface. Fine cracks in the coating on the tension side of the bend are not an indication of poor adhesion. Insertion of a sharp probe at the interface of the coating and basis metal to determine the adhesion is suggested.

Note 17—Appropriate test specimens are strips approximately 25 to 50 mm wide, 200 to 300 mm long and 3 to 6 mm thick.

9.4.2 Impact Test—A spring-loaded center punch with a point having 2 to 3 mm radius is used to test adhesion of the coating on nonsignificant surfaces of the plated part. Make three closely spaced indentations and examine under 10× magnification for flaking or blistering of the coating, which is cause for rejection.

9.4.3 Thermal Shock—The coated part is heated to 200°C in an oven and then quenched in room temperature water. The coating is examined for blistering or other evidence of poor adhesion at 4× magnification.

9.5 Microhardness—The microhardness of the coating can be measured by Test Method B 578 using Knoop indenter and is reported in Knoop Hardness Number (HK). It will vary depending on loads, type of indenter, and operator. A100 g load is recommended. The rhombic Knoop indenter gives higher hardness readings than the square-base pyramidal Vickers diamond indenter for 100 to 300 g loads, see Ref (6). For maximum accuracy, a minimum coating thickness of 75 µm is recommended. Conversions of Vickers or Knoop hardness number to Rockwell C is not recommended.

Note 18—On thick (75 µm+) coatings on steel a surface microhardness determination is permissible.

9.6 Porosity—There is no universally accepted test for porosity. When required, one of the following tests can be used on the plated part or specimen.

9.6.1 Ferroxyl Test for Iron Base Substrates—Prepare the test solution by dissolving 25 g of potassium ferricyanide and 15 g of sodium chloride in 1 L of distilled water. After cleaning, immerse the part for 30 s in the test solution at 25°C. After rinsing and air drying, examine the part for blue spots, which form at pore sites.

9.6.2 Boiling Water Test for Iron-Base Substrates—Completely immerse the part to be treated in a vessel filled with aerated water at room temperature. Apply heat to the beaker at such a rate that the water begins to boil in not less than 15 min, nor more than 20 min after the initial application of heat. Continue to boil the water for 30 min. Then remove the part, air dry, and examine for rust spots, which indicate pores.

Note 19—Aerated water is prepared by bubbling clean compressed air through distilled water by means of a glass diffusion disk at room temperature for 12 h. The pH of the aerated water should be 6.7 + 0.5.

9.6.3 Aerated Water Test for Iron-Base Substrates—Immerse the part for 4 h in vigorously aerated Type IV or better water (see Specification D 1193) at 25 ± 2°C temperature and then examine the part for rust spots.

9.6.4 Alizarin Test for Aluminum Alloys—Wipe the plated part or specimen with 10 mass % sodium hydroxide solution. After 3 min contact, rinse, and apply a solution of alizarin sulfonate prepared by dissolving 1.5 g of methyl cellulose in 90 mL of boiling water to which, after cooling, 0.1 g sodium alizarin sulfonate, dissolved in 5 mL of ethanol is added. After 4 min contact, apply glacial acetic acid until the violet color disappears. Any red spots remaining indicate pores.

9.6.5 Porosity Test for Copper Substrates—Wipe the plated part or specimen with glacial acetic acid. After 3 min, apply a solution of potassium ferrocyanide prepared by dissolving 1 g of potassium ferrocyanide and 1.5 g methyl cellulose in 90 mL of boiling distilled water. The appearance of brown spots after 2 min indicate pores.

9.7 Other Test Methods—Test methods which have been developed that are equal to or better than these may be substituted. The precision and bias requirements will vary for each type of test. If an alternate test is specified it shall be agreed upon between the producer and the purchaser.

10. Rejection and Rehearing

10.1 Part(s) that fail to conform to the requirements of this standard may be rejected. Rejection shall be reported to the producer promptly in writing. In the case of dissatisfaction occurs with the results of a test, the producer may make a claim for a hearing. Coatings that show imperfections may be rejected.

11. Certification

11.1 When specified in the purchase order or contract, the purchaser shall be furnished certification that the samples representing each lot have been processed, tested and inspected as directed in this specification and the requirements have been
met. When specified in the purchase order or contract, a report of the test results shall be furnished.

12. Keywords
12.1 autocatalytic; chemical nickel; coatings; conductive; corrosion resistance; electroless; functional; nickel; nickel phosphorus; wear resistance

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirements shall apply only when specified by the purchaser in the contract or order.

S1.1 Shot Peening—When specified by the purchaser in the ordering information, the part(s) shall be shot peened prior to plating in accordance with Specification B 851 or MIL-S-13165.

S1.2 Composition—When specified by the purchaser in the ordering information the phosphorus content shall be maintained in the deposit to within 1%. Use the test methods described in 9.1.

S1.3 Inert Atmosphere—When specified by the purchaser in the ordering information, the coating shall be heat treated in a vacuum, inert, or reducing atmosphere to prevent surface oxidation of the coating.

S1.4 Hydrogen Embrittlement—When specified by the purchaser in the ordering information the plating process shall be evaluated at the time of processing parts for hydrogen embrittlement using Test Method F 519.

S1.5 Abrasive Wear—When specified by the purchaser in the ordering information, the coating shall be tested for abrasion wear resistance using the method in Appendix X1 of this specification. The coating shall meet a maximum wear rate which is specified by the purchaser and agreed to by the producer.

S1.6 Adhesive Wear—When specified by the purchaser in the ordering information, the coating shall be tested for adhesive wear resistance using Test Method D 2714 or Test Method D 2670. The wear rate shall be specified by the purchaser and agreed to by the producer.

S1.7 Contact Resistance—When specified by the purchaser in the ordering information, the coating shall be tested for contact resistance using Test Method B 667.

S1.8 Solderability—When specified by the purchaser in the ordering information, the unaged coating shall pass Test Method B 678 on solderability.

S1.9 Corrosion Resistance—When specified by the purchaser in the ordering information the coating shall pass any special corrosion tests agreed to by the producer. The corrosion resistance of the coating to a specific liquid medium can be determined by means of immersion tests (see Practice G 31) or electrochemical test (see Practices G 5 and G 59).

S1.10 Pitting Corrosion Resistance—Use Method G 85 (acetic acid-salt spray test), Method B 368 (copper-accelerated acetic acid-salt spray, CASS), or Method B 380 (Corrodkote) to evaluate the corrosion resistance of the coating to pitting.

S1.11 Special Government Requirements:
S1.11.1 Sampling—Part(s) plated for the US Government and Military use shall use MIL-STD-105 as the sampling plan.

S1.11.2 Shot Peening—High strength steel part(s) processed for US Government and Military use shall be shot peened in accordance with MIL-S-13165 or rotary flap peened in accordance with MIL-R-81841. (see Note 6).

S1.11.3 Packaging—Parts shall be packaged in accordance with Practice D 3951.

APPENDIXES
(Nonmandatory Information)

X1. TABER ABRASER WEAR TEST METHOD

X1.1 Scope

X1.1.1 This test method will evaluate the resistance of the coating to abrasive wear. The test is performed by rotating a plated panel under rotating rubber wheels and weighing the panel after each 1000 cycles for weight loss. Duration of the test is 6000 cycles and it can be extended to 25 000 cycles for more complete results.

Note X1.1—Variation in results have been attributed to the humidity in the laboratory and the storage conditions of the CS-10 wheels. Care should be taken to control the humidity between tests.

X1.1.2 The results are variable between tests and therefore three plated test specimens should be tested to 6000 cycles each. The results should be averaged without the first 1000 cycles and the abrasion wear resistance is reported as the weight loss in mg/1000 cycles (Taber Wear Index).
X1.2 Apparatus

X1.2.1 Taber Abraser Wear Testing Unit—The unit must be capable of loading with 1000 g load and operating with a vacuum.

X1.2.2 Abrasion Wheels—Use CS-10 (resilient rubber) Taber wheels. To reface the wheels use CS-11 discs from Taber. The hardness of CS-10 wheels can change with time and can effect the reproducibility of results (see Test Method D 4060).

X1.2.3 Test Specimens—Test specimens shall be made from 20 gage CR steel 4 by 4 in. (100 by 100 by 1.3 mm) with a 0.250 (6.35 mm) hole in the center. Test specimens are available from Taber.

X1.2.4 Analytical Balance—Scale which is capable of measuring to 150 g ± 0.1 mg.

X1.3 Procedure

X1.3.1 Plate three specimens with 0.001 in. (25 µm) of nickel phosphorus coating.

X1.3.2 Wear test the specimens. For each of the three specimens complete the following steps:

X1.3.2.1 Run the CS-10 wheels on the coating for 1000 cycles to remove any surface roughness. The wheels shall be loaded with 1000 g with a vacuum on high for the entire test.

X1.3.2.2 Cool and weigh the specimen to the nearest 0.1 mg.

X1.3.2.3 Dress the CS-10 wheels with a CS-11 disc for 50 cycles.

X1.3.2.4 Abrasion test the coating with 1000 g load for 1000 cycles.

X1.3.2.5 Repeat X1.3.2.2, X1.3.2.3 and X1.3.2.4 until a total of 6000 cycles have been accomplished for each specimen.

X1.4 Reporting

X1.4.1 Determine the average weight loss in milligrams for each specimen per 1000 cycles Taber Wear Index and the mean weight loss per 1000 cycles for all specimens. Report the mean and standard deviation for the coating.
Density of Electroless Nickel Deposits

![Graph showing the density of electroless nickel deposits vs. phosphorus content.]

- ISO 4527, Annex C
- Vendor, Elmic, unpublished
- Kanigen, ASTM STP No. 265, Electroless Nickel Plating 1959
- Oak Ridge, D. D. Smith, Thermal Conductivity of Electroless Nickel-Phosphorus Alloy Plating, National Science Foundation, 1963
- Gorbunova, K. M. Gorbunova and A. A. Nkisforova, Physicochemical Principles of Nickel Plating, National Science Foundation, 1963
- Vendor, Schering, unpublished analytical results, Schering KG, Berlin 1982

FIG. X2.1 Density of Autocatalytic Nickel Phosphorus Alloy Summary of Reported Values
X3. HARDNESS VERSUS HEAT TREATMENT

HARDNESS OF ELECTROLESS NICKEL VS TEMPERATURE VS TYPE

FIG. X3.1 Hardness of Autocatalytic Nickel Phosphorus Versus Heat Treatment Versus Phosphorus

X4. ALLOY TYPES

NOTE X4.1—These different alloy TYPES are produced from processes which are specifically formulated and controlled. Additional requirements for internal stress, purity, and elongation may be necessary for some applications.

X4.1 The physical differences of these TYPES provide for a wide variation in performance in wire bonding, contact resistance, machineability, lubricity, reactivation and melting point. These differences are produced by micro structural differences between Ni + Ni3P and Ni3P. These differences also effect non-destructive thickness testing by X-Ray and magnetic means. Careful attention to the selection of TYPE will insure optimum performance of the deposit in the intended application.
X5. SUMMARY PROGRAM 14

X5.1 Results of 110 Month Exposure of Autocatalytic Nickel Deposits at Kure Beach, North Carolina:

X5.1.1 Program 14 is part of an ongoing marine exposure testing process at the 75 m site at LaQue Center for Corrosion Technology, Wrightsville, NC (Kure Beach). The program called for the plating of both Type IV and Type V deposits on standard smooth and ground steel Q Panels.

X5.1.2 There were nine different sources of deposits, each providing five lots of five panels. The program involved plating 12.5, 25, and 75μm thicknesses on smooth and ground surface and one smooth lot heat treated for hardness at 550°C for 2 h. The heat treatment temperature was considerably higher than typical processing and was chosen to evaluate the formation of diffusion products of iron, nickel, and phosphorus.

X5.1.3 The following matrix of test panels were prepared and exposed with subsequent analysis for alloy and thickness. Panels were rated at Kure Beach each year by a team from ASTM Committee B8 using Practice B 537.

X5.1.4 The interpretation of the results of these exposure test should be made on the basis of general performance of the coating on panels. Base metal condition, undercoats, surface preparation, and post processing all have a significant effect on the performance and should be given careful consideration when designing the part and pretreatment processing sequence.
TABLE X5.1 Results of 110 Month Exposure of Autocatalytic (Electroless) Nickel Deposits at Kure Beach, North Carolina

<table>
<thead>
<tr>
<th>Deposit Type</th>
<th>Source</th>
<th>Lot</th>
<th>Surface</th>
<th>Heat Treatment</th>
<th>Thickness, µm Target</th>
<th>Thickness, µm Actual</th>
<th>Phosphorus Alloy %wt</th>
<th>Practice B 537 Ratings 110 Months</th>
</tr>
</thead>
<tbody>
<tr>
<td>V G</td>
<td>3</td>
<td>Smooth No</td>
<td>75</td>
<td>80.5</td>
<td>10.5</td>
<td>10.9</td>
<td>3.6 ± 3.0</td>
<td></td>
</tr>
<tr>
<td>V E</td>
<td>3</td>
<td>Smooth No</td>
<td>75</td>
<td>77</td>
<td>10.0</td>
<td>10.2</td>
<td>3.0 ± 0.3</td>
<td></td>
</tr>
<tr>
<td>V C</td>
<td>3</td>
<td>Smooth No</td>
<td>75</td>
<td>46</td>
<td>9.9</td>
<td>10.4</td>
<td>6.2 ± 0.4</td>
<td></td>
</tr>
<tr>
<td>IV J</td>
<td>3</td>
<td>Smooth No</td>
<td>75</td>
<td>9.0</td>
<td>9.6 ± 1.1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>IV H</td>
<td>3</td>
<td>Smooth No</td>
<td>75</td>
<td>8.9</td>
<td>10.0 ± 0.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>IV B</td>
<td>3</td>
<td>Smooth No</td>
<td>75</td>
<td>79</td>
<td>8.3</td>
<td>7.3</td>
<td>6.8 ± 1.3</td>
<td></td>
</tr>
<tr>
<td>IV D</td>
<td>3</td>
<td>Smooth No</td>
<td>75</td>
<td>77</td>
<td>8.2</td>
<td>9.5</td>
<td>0.0 ± 0.0</td>
<td></td>
</tr>
<tr>
<td>IV K</td>
<td>3</td>
<td>Smooth No</td>
<td>75</td>
<td>7.5</td>
<td>6.0 ± 0.8</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>IV I</td>
<td>3</td>
<td>Smooth No</td>
<td>75</td>
<td>80</td>
<td>7.1</td>
<td>6.0 ± 0.8</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

A The conclusions from this marine exposure program have been summarized by Dr. George DiBari of INCO in his paper Marine Corrosion Performance of EN Coating on Steel, Final Report on ASTM Program 14 which was presented at EN91 Conference, Product Finishing.

REFERENCES

Standard Specification for Electrodeposited Copper for Engineering Uses

This standard is issued under the fixed designation B 734; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

**e1** NOTE—Section 6.8.1 was editorially updated in May 2003.

1. Scope

1.1 This specification covers requirements for electrodeposited coatings of copper used for engineering purposes. Examples include surface hardening, heat treatment stop-off, as an underplate for other engineering coatings, for electromagnetic interferences (EMI) shielding in electronic circuitry, and in certain joining operations.

1.2 This specification is not intended for electrodeposited copper when used as a decorative finish, or as an undercoat for other decorative finishes.

1.3 This specification is not intended for electrodeposited copper when used for electroforming.

2. Referenced Documents

2.1 ASTM Standards:

- B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
- B 242 Practice for Preparation of High-Carbon Steel for Electroplating
- B 254 Practice for Preparation of and Electroplating on Stainless Steel
- B 320 Practice for Preparation of Iron Castings for Electroplating
- B 322 Practice for Cleaning Metals Prior to Electroplating
- B 374 Terminology Relating to Electroplating
- B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section
- B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
- B 507 Practice for Design of Articles to Be Electroplated on Racks
- B 555 Guide for Measurement of Electrodeposited Metallic Coating Thicknesses by the Dropping Test
- B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
- B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
- B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings
- B 588 Test Method for Measurement of Thickness of Transparent or Opaque Coatings by Double-Beam Interference Microscope Technique
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
- B 659 Guide for Measuring Thickness of Metallic and Inorganic Coatings
- B 678 Test Method for Solderability of Metallic-Coated and Related Metallic Coatings
- B 832 Guide for Electroforming with Nickel and Copper
- B 849 Specification for Pre-Treatments of Iron or Steel for Reducing Risk of Hydrogen Embrittlement
- B 850 Guide for Post-Coating Treatments of Steel for Reducing the Risk of Hydrogen Embrittlement
- B 851 Specification for Automated Controlled Shot Peening of Metallic Articles Prior to Nickel, Auto Catalytic Nickel, or Chromium Plating, or as Final Finish
- B 3951 Practice for Commercial Packaging
- F 519 Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments

2.2 Military Standard:

- MIL-R-81841 Rotary Flap Peening of Metal Parts
- MIL-S-13165 Shot Peening of Metal Parts
- MIL-W-81840 Rotary Flap Peening Wheels

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1 This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.08.01 on Engineering Coatings.


2 Annual Book of ASTM Standards, Vol 02.05.
3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 significant surfaces—those surfaces normally visible (directly or by reflection) that are essential to the appearance or serviceability of the article when assembled in a normal position; or which can be the source of corrosion products that deface visible surfaces on the assembled article. When necessary, the significant surface shall be indicated on the drawing of the article, or by the provision of suitably marked samples.

NOTE 1—When significant surfaces are involved on which the specified thickness of coating cannot readily be controlled, such as threads, holes, deep recesses, and bases of angles, it will be necessary to apply thicker coatings on the more accessible surfaces, or to use special racking or both.

3.1.2 inspection lot—a collection of coated articles that; are of the same type; have been produced to the same specifications; have been coated by a single supplier at one time, or at approximately the same time, under essentially identical conditions; and are submitted for acceptance or rejection as a group.

3.2 Definitions—For definitions of the technical terms used in this specification see Terminology B 374.

4. Classification

4.1 The electrodeposited copper is classified according to thickness of the electrodeposit in the following table:

<table>
<thead>
<tr>
<th>Class</th>
<th>Minimum Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>12</td>
<td>12</td>
</tr>
<tr>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>x</td>
<td>Thickness specified</td>
</tr>
</tbody>
</table>

4.2 Where required, dimensional tolerances allowed for the specified electroplated copper thickness shall be specified.

5. Ordering Information

5.1 The buyer shall supply to the producer in the purchase order or engineering drawings; marked samples or other governing documents the following information:

5.1.1 Title, ASTM designation number (Specification B 734), and date of issue.

5.1.2 Classification or thickness of electrodeposited copper (see 4.1).

5.1.3 Significant surfaces if other than defined in 3.1.1.

5.1.4 Sampling plan (Section 7).

5.1.5 Number of test specimens for destructive testing (Section 8), and

5.1.6 Thickness, adhesion, solderability, porosity and number of pores acceptable, or hydrogen embrittlement tests and methods required (Section 8).

5.2 Where required, dimensional tolerances allowed for the specified electroplated copper thickness shall be specified.

5.3 In addition to the requirements of 5.1 and when the parts to be electroplated are supplied to the electroplater by the buyer, the buyer shall also supply the following information as required.

5.3.1 Identity of the base material by alloy identification such as ASTM, AISI, or SAE numbers, or equivalent composition information.

5.3.2 Hardness of the parts, and

5.3.3 Heat treatment for stress relief, whether it has been performed or is required.

5.4 If required by either party, the manufacturer of the parts to be electroplated shall provide the electroplating facility with separate test specimens (see section 8.1).

6. Coating Requirements

6.1 Appearance—The coating on the significant surfaces of the product shall be smooth and free of visual defects such as blisters, pits, roughness, cracks, flaking, burned deposits, and uncoated areas. The boundaries of electroplating that cover only a portion of the surface shall, after finishing as indicated in the drawing, be free of beads, nodules, jagged edges and other detrimental irregularities. Imperfections and variations in appearance in the coating that arise from surface conditions of the basis metal (scratches, pores, roll marks, inclusions, etc.) and that persist in the finish despite the observance of good metal finishing practices shall not be cause for rejection.

NOTE 3—Electroplated finishes generally perform better when the substrate over which they are applied is smooth and free of deep scratches, torn metal, pores, inclusions, and other defects. It is recommended that the specifications covering the unfinished product provide limits for these defects. A metal finisher can often remove defects through special treatments such as grinding, polishing, abrasive blasting, and special chemical treatments. However, these are not normal treatment steps. When they are desired, they must be agreed upon between the buyer and the producer.

6.2 Thickness—The thickness of the copper coating on the significant surfaces shall conform to the requirements of the specified class as defined in Section 4.

NOTE 4—Variation in the coating thickness from point-to-point on a coated article is an inherent characteristic of electroplating processes. Therefore, the coating thickness will have to exceed the specified value at some points on the significant surfaces to ensure that the thickness equals or exceeds the specified value at all points. As a result, the average coating thickness on an article will usually be greater than the specified value; how much greater is largely determined by the shape of the article (see Practice B 307) and the characteristics of the electroplating process. Additionally, the average coating thickness on an article will vary from article to article within a production lot. Therefore, if all of the articles in a production lot are to meet the thickness requirement, the average coating thickness of the production lot as a whole will be greater than the average necessary to ensure that a single article meets the requirements.

NOTE 5—When electroplating threaded parts such as machine screws, care is required to avoid too much plate buildup on the crest of the thread. In such applications a maximum plate thickness allowable on the crests may require that thicknesses in other areas be thinner.

6.3 Porosity—When specified, the coating shall be sufficiently free of pores to pass the porosity test specified in 8.4.

6.4 Solderability—When specified, the coating shall meet the requirements of Test Method B 678.

6.5 Pretreatment of Iron and Steel for Reducing the Risk of Hydrogen Embrittlement—Parts for critical applications that are made of steels with ultimate tensile strengths of 1000 MPa, hardness of 31 HRC or greater, that have been machined, ground, cold formed, or cold straightened subsequent to heat treatment, shall require stress relief heat treatment when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specification B 849 may be consulted for a list of pretreatments that are used widely.
6.6 Post Coating Treatment of Iron and Steel for Reducing the Risk of Hydrogen Embrittlement—Parts for critical applications that are made of steels with ultimate tensile strengths of 1000 MPa, hardness of 31 HRC or greater, as well as surface hardened parts, shall require post coating hydrogen embrittlement relief baking when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specification B 850 may be consulted for a list of post treatments that are used widely.

6.7 Peening of Metal Parts—If peening is required before electroplating to induce residual compressive stress to increase fatigue strength and resistance to stress corrosion cracking of the metal parts, refer to MIL-S-13165, MIL-R-81841, MIL-W-81840, and Specification B 851.

6.8 Supplementary Requirements:

6.8.1 Packaging—If packaging requirements are to be met under this specification, they shall be in accordance with Practice D 3951, or as specified in the contract or order. (Warning—Some contemporary packaging materials may emit fumes that are deleterious to the surface of the coating.)

7. Sampling

7.1 The sampling plan used for the inspection of a quantity of the coated articles shall be as agreed upon between the purchaser and the seller.

Note 6—Usually, when a collection of coated articles, the inspection lot (7.2), is examined for compliance with the requirements placed on the articles, a relatively small number of the articles, the sample, is selected at random and is inspected. The inspection lot then is classified as complying or not complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Three standards, Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for the sampling inspection of coatings. Test Method B 602 contains four sampling plans, three for use with tests that are non-destructive and one when they are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 identifies the plan to be used. Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and seller need to agree on the plan to be used. Methods B 762 can be used only for coating requirements that have numerical limit, such as coating thickness. The test must yield a numerical value and certain statistical requirements must be met. Methods B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Methods B 762 identifies the plan to be used.

Note 7—When both destructive and non-destructive tests exist for the measurement of a characteristic, the purchaser needs to state which is to be used so that the proper sampling plan is selected. A test may destroy the coating in a non-critical area; or, although it may destroy the coating, a tested part can be reclaimed by stripping and recoating. The purchaser needs to state whether the test is to be considered destructive or non-destructive.

7.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specifications, that have been coated by a single supplier at one time, or at approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

7.3 If special test specimens are used to represent the coated articles in a test, the number used shall be that required in 8.1.1.

8. Test Methods

8.1 The permission or the requirement to use special test specimens, the number to be used, the material from which they are to be made, and their shape and size shall be stated by the purchaser.

Note 8—Test specimens often are used to represent the coated articles in a test if the articles are of a size, shape, or material that is not suitable for the test, or if it is preferred not to submit articles to a destructive test because, for example, the articles are expensive or few in number. The specimen should duplicate the characteristics of the article that influence the property being tested.

8.1.1 Special test specimens used to represent articles in an adhesion, porosity, corrosion resistance, or appearance test shall be made of the same material, in the same metallurgical condition, and have the same surface condition as the articles they represent, and be placed in the production lot of and be processed along with the articles they represent.

8.1.2 Special test specimens used to represent articles in a coating thickness test may be made of a material that is suitable for the test method even if the represented article is not of the same material. For example, a low-carbon steel specimen may represent a brass article when the magnetic thickness test is used (Test Method B 499). The thickness specimen need not be carried through the complete process with the represented article. If not, introduce it into the process at the point where the coating is applied and carry it through all steps that have a bearing on the coating thickness. In rack plating, rack the specimen in the same way with the same distance from and orientation with the anodes and other items in the process as the article it represents.

Note 9—When special test specimens are used to represent coated articles in a thickness test, the specimens will not necessarily have the same thickness and thickness distribution as the articles unless the specimens and the articles are of the same general size and shape. Therefore, before finished articles can be accepted on the basis of a thickness test performed on special test specimens, the relationship between the thickness on the specimen and the thickness on the part needs to be established. The criterion of acceptance is that thickness on the specimen that corresponds to the required thickness on the article.

8.2 Thickness—Measure the coating thickness at locations on the significant surface designated by the purchaser, and make the measurement with an accuracy of 10 % or better by use of one of the following test methods: Test Methods B 487, B 499, B 504, B 568, and B 588.

Note 10—Other thickness measurement methods may be used where it can be demonstrated that the uncertainty is less than 10 %.

8.3 Adhesion—Use one or more of the methods described in Test Methods B 571 as agreed and as specified in the purchase order or other specifying document.

8.4 Porosity—Conduct the ferroxyl test as described in Appendix X1. Observe the results after 5 min. The part fails if more than the number of pores per unit area specified by the purchaser is found.

8.5 Solderability—Conduct the test in accordance with Test Method B 678. The coating shall be deemed solderable if the solder coating is adherent, bright, smooth, and uniform over at least 95 % of the test surface.

Note 11—Electroplated copper surfaces become more difficult to
solder as they age. It may be necessary to clean the copper surface just prior to the soldering test or soldering operation.

8.6 Embrittlement Relief—Parts shall be examined visually for cracks indicating embrittlement failure, or the effectiveness of the relief treatment shall be determined by a procedure specified by the purchaser.

Note 12—Test Method F 519 describes hydrogen embrittlement testing that utilizes specially machined test specimens.

9. Rejection and Rehearing

9.1 Materials that fail to conform to the requirements of this specification shall be rejected. Rejection shall be reported to the producer or the supplier promptly, and in writing. In case of dissatisfaction with the results of a test, the producer or supplier may make a claim for a rehearing. Finishes that show imperfections during subsequent manufacturing operations may be rejected.

10. Certification

10.1 The purchaser may require in the purchase order or contract that the producer or supplier give to the purchaser certification that the finish was produced and tested in accordance with this specification and found to meet the requirements. The purchaser may similarly require that a report of the test results be furnished.

APPENDIX

(Nonmandatory Information)

X1. MODIFIED FERROXYL TEST (ALSO SEE GUIDE B 765)

X1.1 General

X1.1.1 This method reveals discontinuities, such as pores, in electroplated copper on iron or steel.

Note X1.1—This test is slightly corrosive to copper, particularly if the test period is extended appreciably (3 min or more) beyond the 5-min period. The test is very sensitive to the superficial presence of iron, that is, blue spots can occur on an electrodeposited copper surface that has been in sufficient contact with a piece of iron to leave a trace of the iron on the copper surface.

X1.2 Materials

X1.2.1 Three solutions and strips of “wet strength” filter paper are required.

X1.2.1.1 Solution A, is prepared by dissolving 50 g of white gelatine and 50 g of sodium chloride in 1 L of warm (45°C) distilled water.

X1.2.1.2 Solution B, is prepared by dissolving 50 g of sodium chloride and 0.1 g of a non-ionic wetting agent in 1 L of distilled water.

X1.2.1.3 Solution C, is prepared by dissolving 10 g of potassium ferricyanide in 1 L of distilled water.

X1.3 Procedure

X1.3.1 Immerse filter paper strips in Solution A (which is kept sufficiently warm to keep the gelatine dissolved), then remove and allow to dry. Just before use, immerse the dry filter paper strips in Solution B just long enough to thoroughly wet all of the filter paper. Firmly press the filter paper against the thoroughly cleaned and degreased electroplated copper surface to be tested. Allow 5-min contact time for the test period (see Note X1.1). If the filter paper should become dry during the test, moisten again with Solution B. Remove the papers at the end of the contact period and place at once into Solution C. Sharply defined blue markings will appear on the papers indicating basis metal corrosion or porosity.

X1.4 Report

X1.4.1 The report shall include the following information:

X1.4.1.1 The area of surface tested.
X1.4.1.2 The total number and diameter of all spots on the filter paper oriented to surface area tested.
X1.4.1.3 The highest number of spots visible within a square area as defined and specified by the purchaser.
Standard Test Method for
Measurement of Thickness of Metallic Coatings by Measurement of Cross Section with a Scanning Electron Microscope

This standard is issued under the fixed designation B 748; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of metallic coating thicknesses by examination of a cross section with a scanning electron microscope (SEM).

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   E 3 Guide for Preparation of Metallographic Specimens
   E 766 Practice for Calibrating the Magnification of a Scanning Electron Microscope

3. Summary of Test Method

3.1 A test specimen is cut, ground, and polished for metallographic examination by an SEM of a cross section of the coating. The measurement is made on a conventional micrograph or on a photograph of the video waveform signal for a single scan across the coating.

4. Significance and Use

4.1 This test method is useful for the direct measurement of the thicknesses of metallic coatings and of individual layers of composite coatings, particularly for layers thinner than normally measured with the light microscope.

4.2 This test method is suitable for acceptance testing.

4.3 This test method is for the measurement of the thickness of the coating over a very small area and not of the average or minimum thickness per se.

4.4 Accurate measurements by this test method generally require very careful sample preparation, especially at the greater magnifications.

4.5 The coating thickness is an important factor in the performance of a coating in service.

5. Equipment

5.1 The scanning electron microscope shall have a resolution of at least 50 nm. Suitable instruments are available commercially.

6. Factors Affecting the Measurement Reliability

6.1 Surface Roughness—If the coating or its substrate is rough relative to the coating thickness, one or both of the interfaces bounding the coating cross section may be too irregular to permit accurate measurement of the average thickness in the field of view.

6.2 Taper of Cross Section—If the plane of the cross section is not perpendicular to the plane of the coating, the measured thickness will be greater than the true thickness. For example, an inclination of 10° to the perpendicular will contribute a 1.5% error. True thickness, \( t \), equals measured thickness, \( t_m \), multiplied by the cosine of the angle of inclination (\( \theta \)):

\[
  t = t_m \times \cos(\theta).
\]

(See X1.3.2.)

6.3 Specimen Tilt—Any tilt of the specimen (plane of the cross section) with respect to the SEM beam, may result in an erroneous measurement. The instrument should always be set for zero tilt.

6.4 Oblique Measurement—If the thickness measurement is not perpendicular to the plane of the coating, even when there is no taper (6.2) or tilt (6.3), the measured value will be greater than the true thickness. This consideration applies to the conventional micrograph (9.3.1) and to the direction of the single video waveform scans (9.3.2).

6.5 Deformation of Coating—Detrimental deformation of the coating can be caused by excessive temperature or pressure during the mounting and preparation of cross sections of soft coatings.

6.6 Rounding of Edge of Coating—If the edge of the coating cross section is rounded, that is, if the coating cross section is not completely flat up to its edges, the observed thickness may differ from the true thickness. Edge rounding can be caused by improper mounting, grinding, polishing, or etching.

6.7 Overplating of Specimen—Overplating of the test specimen serves to protect the coating edges during preparation of cross sections and thus to prevent an erroneous measurement. Removal of coating material during surface preparation for overplating can cause a low thickness measurement.

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1 This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on Test Methods.


2 Annual Book of ASTM Standards, Vol 03.01.
6.8 Etching—Optimum etching will produce a clearly defined and narrow dark line at the interface of two metals. A wide or poorly defined line can result in an inaccurate measurement.

6.9 Smearing—Polishing may leave smeared metal that obscures the true boundary between the two metals and results in an inaccurate measurement. This may occur with soft metals like lead, indium, and gold. To help identify whether or not there is smearing, repeat the polishing, etching, and measurement several times. Any significant variations in readings indicates possible smearing.

6.10 Poor Contrast—The visual contrast between metals in the SEM is poor when their atomic numbers are close together. For example, bright and semibright nickel layers may not be discriminable unless their common boundary can be brought out sufficiently by appropriate etching and SEM techniques. For some metal combinations, energy dispersive X-ray techniques (see X1.4.5) or backscatter image techniques (see X1.4.6) may be helpful.

6.11 Magnification:

6.11.1 For any given coating thickness, measurement errors tend to increase with decreasing magnification. If practical, the magnification should be chosen so that the field of view is between 1.5 and 3× the coating thickness.

6.11.2 The magnification readout of an SEM is often poorer than the 5% accuracy often quoted and the magnification has been found for some instruments to vary by 25% across the field. Magnification errors are minimized by appropriate use of an SEM stage micrometer and appropriate experimental procedure. (See Practice E 766.)

6.12 Uniformity of Magnification—Because the magnification may not be uniform over the entire field, errors can occur if both the calibration and the measurement are not made over the same portion of the field. This can be very important.

6.13 Stability of Magnification:

6.13.1 The magnification of an SEM often changes or drifts with time. This effect is minimized by mounting the stage micrometer and test specimen side by side on the SEM stage so as to keep the transfer time short.

6.13.2 A change in magnification can occur when adjustments are made with the focusing and other electronic SEM controls. Such a change is prevented by not using the electronic focus controls or other electronic SEM controls after photographing the stage micrometer scale except to focus with the mechanical X, Y, and Z controls. Appropriate manipulation of the X, Y, and Z controls will bring the specimen surface to the focal point of the SEM beam.

6.14 Stability of Micrographs—Dimensional changes of micrographs can take place with time and with temperature and humidity changes. If the calibration micrograph of the stage micrometer scale and the micrograph of the test specimen are kept together and time is allowed for stabilization of the photographic paper, errors from this source will be minimized.

7. Preparations of Cross Sections

7.1 Prepare, mount, grind, polish, and etch the test specimen so that the following occurs:

7.1.1 The cross section is perpendicular to the plane of the coating.

7.1.2 The surface is flat and the entire width of the coating image is simultaneously in focus at the magnification to be used for the measurement.

7.1.3 All material deformed by cutting or cross sectioning is removed.

7.1.4 The boundaries of the coating cross section are sharply defined by contrasting appearance, or by a narrow, well-defined line, and

7.1.5 If the video waveform signal is to be measured, the signal trace is flat except across the two boundaries of the coating.

7.2 For further guidance see Appendix X1.

8. Calibration of Magnification

8.1 Calibrate the SEM with an SEM stage micrometer and determine the magnification factor, M, in accordance with Practice E 766 (see X1.4.2). Other calibration methods may be used if it can be demonstrated that they are sufficiently accurate for meeting the requirement of Section 12.

8.2 If practical, the stage micrometer and the test specimen shall be mounted side by side on the SEM stage.

9. Procedure

9.1 Operate the SEM in accordance with the manufacturer’s instructions.

9.2 Take into account the factors listed in Sections 6 and 12.

9.3 Make a micrograph of the test specimen under the same conditions and instrument settings as used for the calibration and make an appropriate measurement of the micrograph image. Carry out this step in accordance with 9.3.1 or 9.3.2.

9.3.1 Conventional Micrograph:

9.3.1.1 With the boundaries of the coating clearly and sharply defined, make conventional micrographs of the SEM stage micrometer scale and of the test specimen.

9.3.1.2 Measure the micrographs to at least the nearest 0.1 mm using a diffraction plate reader or equivalent device. If this is not practical, it may be because poor sample preparation is causing the boundaries of the coating to be poorly defined.

9.3.2 Video Waveform Signal:

9.3.2.1 Photograph the video waveform signal for a single scan across the coating cross section and across the SEM stage micrometer scale.

9.3.2.2 To measure the coating, measure the horizontal distance between the inflection points of the vertical portions of the scan at the boundaries of the coating. Make the measurements to the nearest 0.1 mm using a diffraction plate reader or equivalent device.

9.3.3 For further guidance see Appendix X1.

10. Calculation and Expression of Results

10.1 Calculate the thickness according to the expression:

\[ T = 1000 \times \frac{d}{M} \]  

where:

- \( T \) = coating thickness, in µm,
- \( d \) = linear distance on micrograph, in mm, and
- \( M \) = magnification factor as defined in Practice E 766.
11. Report

11.1 The report of the measurements shall give the following information:

11.1.1 Date measurements were made,
11.1.2 The title, number, and year of issue of this test method,
11.1.3 Identification of the test specimen(s),
11.1.4 Location of measurement on test specimen(s),
11.1.5 The measured values and their arithmetic mean,
11.1.6 The calibrated magnification as measured with an SEM micrometer scale immediately before the test specimen measurements,
11.1.7 Type of measurement: conventional micrograph or video waveform signal,
11.1.8 Any unusual feature of the measurement that might affect the results, and
11.1.9 Name of individual responsible for the measurements.

12. Precision and Bias

12.1 The instrument, its operation, and its calibration shall be such that the uncertainty of the measurements shall be less than 0.1 µm or 10 %, whichever is larger.

12.2 For a thin gold coating, one laboratory reported measurement uncertainty of 0.039 µm for the SEM stage micrometer scale, 0.02 µm for the measurement of the calibration micrographs, and 0.02 µm for measurement of the video waveform signal scan. Based on practical experience, a repeatability of 0.1 µm or better may be assumed.

APPENDIX

(Nonmandatory Information)

X1. TECHNIQUES OF SPECIMEN PREPARATION AND USE OF THE SEM

X1.1 Introduction

X1.1.1 The preparation of specimens and measurement of coating thickness are greatly dependent on individual techniques and there is a variety of suitable techniques available. (See Guide E 3.) It is not reasonable to specify only one set of techniques, and it is impractical to include all suitable techniques. The techniques described in this appendix are intended as guidance.

X1.2 Mounting

X1.2.1 To prevent rounding of the edge of the coating cross section, the free surface of the coating should be supported so that there is no space between the coating and its support. This is usually achieved by overplating the coating with a coating at least 10 µm thick of a metal of similar hardness to the coating. The overplate should also give an electron signal intensity different from that of the coating. The mounting material or sample surface must be electrically conducting and grounded to prevent a surface charge buildup in the SEM.

X1.3 Grinding and Polishing

X1.3.1 It is essential to keep the cross section surface of the mount perpendicular to the coating. This is facilitated by incorporating additional pieces of a similar metal in the plastic mounting, near the outer edges, by periodically changing the direction of grinding (rotating through 90° and by keeping the grind time and pressure to a minimum). If, before grinding, reference marks are inscribed on the sides of the mount, any inclination from horizontal is easily measurable. Grind the mounted specimens on suitable abrasive paper, using an acceptable lubricant, such as water, and apply minimum pressure to avoid bevelling the surface. Initial grinding should employ 100 or 180 grade abrasive to reveal the true specimen profile and to remove any deformed metal. Subsequently, use Grades 240, 320, 500, and 600 without exceeding grinding times of 30 to 40 s on each paper; alter the direction of scratches by 90° for each change of paper. Then polish successively with 6 to 9, 1, and 0.5-µm diamond on microcloth. Some metallographers prefer the use of 0.3- and 0.05-µm alumina.

X1.3.2 A convenient way to check for tapering of the cross section is to mount a small diameter rod or wire with the specimen so that the perpendicular cross section of the rod is parallel to that of the coating. If a taper is present, the cross section of the rod will be elliptical.

X1.3.3 If the video waveform signal scan technique is used, it is important that scratches be completely removed and that overpolishing does not selectively remove one of the metals more than the other so that the signal scan is distorted. With careful polishing, it is often unnecessary to use chemical etches.

X1.4 Use of SEM

X1.4.1 If the image of the cross section, as revealed in a conventional micrograph, is measured; and if the boundaries of the coating cross section are revealed solely by the photographed contrast between the two materials; the apparent width of the coating cross section can vary, depending on the contrast and brightness settings. The variation can be as great as 10 % without any change in instrument magnification. To minimize the resulting uncertainty, adjust the contrast and brightness so that the image contains surface detail of the materials on either side of each boundary.

X1.4.2 Because the magnification of an SEM can change spontaneously with time and can change as a result of changing other instrument settings, it is advisable to calibrate the instrument immediately before or after measurement of the test
specimen. For critical measurements, the average of measure-
ments made before and after measurement of the test specimen
should be used. This assures that no change in the magnifica-
tion occurred and it provides information about the precision of
the calibration.

X1.4.3 If the video waveform trace is measured, the mea-
surement is made of the horizontal distance between the
inflection points at the boundaries. The inflection point is half
way between the horizontal traces of the two materials.

X1.4.4 For a video-waveform trace, select a portion of the
polished specimen that yields a flat, smooth signal.

X1.4.5 Many SEMs are equipped with energy dispersive
X-ray spectroscopy (EDS) which can be helpful in identi-fying
the metal-coating layers. At best the resolution of EDS is about
1 µm and often it is poorer.

X1.4.6 The use of backscatter images instead of secondary
electron images can also be helpful in distinguishing metal
layers with atomic numbers as close together as 1.0 and with a
resolution of 0.1 µm.
Standard Test Method for
Particle Size Distribution of Metal Powders and Related
Compounds by X-Ray Monitoring of Gravity Sedimentation

1. Scope *

1.1 This test method covers the determination of particle size distributions of metal powders. Experience has shown that this test method is satisfactory for the analysis of elemental tungsten, tungsten carbide, molybdenum, and tantalum powders, all with an as-supplied Fisher number of 6 µm or less, as determined by Test Method B 330. Other metal powders (for example, elemental metals, carbides, and nitrides) may be analyzed using this test method with caution as to significance until actual satisfactory experience is developed (see 7.2). The procedure covers the determination of particle size distribution of the powder in the following two conditions:

1.1.1 As the powder is supplied (as-supplied), and

1.1.2 After the powder has been deagglomerated by rod milling as described in Practice B 859.

1.2 This test method is applicable to particles of uniform density and composition having a particle size distribution range of 0.1 up to 100 µm.

1.2.1 However, the relationship between size and sedimentation velocity used in this test method assumes that particles sediment within the laminar flow regime. This requires that the particles sediment with a Reynolds number of 0.3 or less. Particle size distribution analysis for particles settling with a larger Reynolds number may be incorrect due to turbulent flow. Some materials covered by this test method may settle with Reynolds number greater than 0.3 if particles greater than 25 µm are present. The user of this test method should calculate the Reynolds number of the largest particle expected to be present in order to judge the quality of obtained results. Reynolds number (Re) can be calculated using the following equation

\[ Re = \frac{D^3(\rho - \rho_0)g}{18\eta^2} \]  

where

\( D \) = the diameter of the largest particle expected to be present,
\( \rho \) = the particle density,
\( \rho_0 \) = the suspending liquid density,
\( g \) = the acceleration due to gravity, and
\( \eta \) = is the suspending liquid viscosity.

A table of the largest particles that can be analyzed with Reynolds number of 0.3 or less in water at 35°C is given for a number of metals in Table 1. A column of the Reynolds number calculated for a 30-µm particle sedimenting in the same liquid system is given for each material also.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard information is given in Section 7.

2. Referenced Documents

2.1 ASTM Standards:

- B 330 Test Method for Average Particle Size of Powders of Refractory Metals and Their Compounds by the Fisher Sub-Sieve Sizer
- B 821 Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis
- B 859 Practice for De-agglomeration of Refractory Metal Powders and Their Compounds Prior to Particle Size Analysis
- E 456 Terminology Related to Quality and Statistics
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Method

3.1 A carefully dispersed homogeneous suspension of the powder is permitted to settle in a cell scanned by a collimated X-ray beam of constant intensity. The net X-ray signal is inversely proportional to the sample concentration in the

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1 This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.03 on Refractory Metal Powders.

2 Annual Book of ASTM Standards, Vol 02.05.

dispersing medium, and the particle diameter is related to the position of the X-ray beam relative to the top of the cell. Cumulative mass percent versus equivalent spherical diameter are recorded to yield a particle size distribution curve.

4. Significance and Use

4.1 This test method is useful to both suppliers and users of powders, as outlined in 1.1 and 1.2, in determining particle size distribution for product specifications, manufacturing control, development, and research.

4.2 Users should be aware that sample concentrations used in this test method may not be what is considered ideal by some authorities, and that the range of this test method extends into the region where Brownian movement could be a factor in conventional sedimentation. Within the range of this test method, neither the sample concentration nor Brownian movement are believed to be significant.

4.3 Reported particle size measurement is a function of both the actual particle dimension and shape factor as well as the particular physical or chemical properties being measured. Caution is required when comparing data from instruments operating on different physical or chemical parameters or with different particle size measurement ranges. Sample acquisition, handling, and preparation can also affect reported particle size results.

5. Apparatus

5.1 Gravitational sedimentation particle size analyzer utilizing X-ray extinction to determine particle concentration.

6. Reagents and Materials

6.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 Dispersing Medium—Dissolve 0.10 g of sodium hexametaphosphate \((\text{NaPO}_3)_6\) in 1000 mL of distilled or deionized water.

6.3 Cleaning Solution—Dissolve 0.5 g of laboratory detergent in 1000 mL of distilled or deionized water, or prepare a 0.1 % solution by volume of Triton X-100 using distilled or deionized water.

7. Hazards

7.1 Precautions applying to the use of low intensity X-ray units should be observed.

7.2 Most carbides and nitrides are brittle materials and may be partially deagglomerated or fractured, or both, during the manufacturing process. Different manufacturing processes or changes in the process may affect the apparent particle size distribution as determined by this test method. Thus, caution should be used in evaluating the results, especially for brittle materials.

8. Sample Preparation

8.1 For the as-supplied particle size distribution determinations, this step is not needed.

8.2 For laboratory-milled particle size distribution determinations, use the rod milling technique as outlined in Practice B 859.

9. Procedure

9.1 See the manufacturer’s manual for general operating instructions.

9.2 Set up the instrument in the “percent finer than” mode if necessary. Ensure proper operating conditions by periodically performing base line scan and beam split test if necessary.

9.3 Add appropriate sample weight to the amount of dispersing medium suggested in analyzer instruction manual.

Note 1—Suggested approximate starting weights for tungsten and tungsten carbide are listed in Table 2.

---

TABLE 1 Maximum Diameter of Metal Powders and Related Compounds That Can Be Analyzed with Reynolds Number of 0.3 or Less in Water at 35°C

<table>
<thead>
<tr>
<th>Particle Composition</th>
<th>Particle Density</th>
<th>Maximum Particle Diameter</th>
<th>Reynolds Number for 30 µm&lt;sup&gt;a&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cobalt</td>
<td>8.90</td>
<td>33.19</td>
<td>0.22</td>
</tr>
<tr>
<td>Copper</td>
<td>8.92</td>
<td>33.16</td>
<td>0.22</td>
</tr>
<tr>
<td>Iron</td>
<td>7.86</td>
<td>34.79</td>
<td>0.19</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>10.20</td>
<td>31.55</td>
<td>0.26</td>
</tr>
<tr>
<td>Nickel</td>
<td>8.90</td>
<td>33.19</td>
<td>0.22</td>
</tr>
<tr>
<td>Tantalum</td>
<td>16.60</td>
<td>26.46</td>
<td>0.44</td>
</tr>
<tr>
<td>Tantalum carbide</td>
<td>13.90</td>
<td>28.19</td>
<td>0.36</td>
</tr>
<tr>
<td>Titanium carbide</td>
<td>4.93</td>
<td>41.88</td>
<td>0.11</td>
</tr>
<tr>
<td>Titanium</td>
<td>19.35</td>
<td>25.06</td>
<td>0.51</td>
</tr>
<tr>
<td>Tungsten carbide</td>
<td>15.63</td>
<td>27.03</td>
<td>0.41</td>
</tr>
<tr>
<td>Vanadium</td>
<td>6.11</td>
<td>38.37</td>
<td>0.014</td>
</tr>
<tr>
<td>Vanadium carbide</td>
<td>5.77</td>
<td>39.26</td>
<td>0.13</td>
</tr>
</tbody>
</table>

<sup>a</sup> Reynolds number calculated for 30 µm particle sedimenting in water at 35°C, with a density of 0.9941 g/cm³ and viscosity of 0.7225 cp.
TABLE 2  Suggested Approximate Starting Weights for Tungsten or Tungsten Carbide

<table>
<thead>
<tr>
<th>Nominal Fisher Number According to B 330 of As-Supplied Powder, µm</th>
<th>Weight⁴, g per 25 mL of Dispersing Medium</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.21</td>
</tr>
<tr>
<td>2</td>
<td>0.23</td>
</tr>
<tr>
<td>3</td>
<td>0.26</td>
</tr>
<tr>
<td>4</td>
<td>0.28</td>
</tr>
<tr>
<td>5</td>
<td>0.30</td>
</tr>
<tr>
<td>6</td>
<td>0.33</td>
</tr>
</tbody>
</table>

⁴ The amount of sample required will vary. Increase or decrease the sample weight as needed to provide the level of X-ray attenuation recommended in analyzer instruction manual.

9.4 Sample Dispersion—Follow procedure recommended in Guide B 821.

9.5 Temperature Adjustment:

9.5.1 If the temperature of the solution is above that of the cell chamber after ultrasonic dispersion, cool the solution to within 1°C of the cell chamber prior to the introduction into the cell chamber by stirring and pumping outside the cell chamber (see Note 2). This cooling must be accomplished as soon as possible.

NOTE 2—It may be convenient to use a separate magnetic stirrer and stirring rod.

9.5.2 If the temperature is below the cell chamber temperature, load the sample into the cell or sample chamber of the analyzer and allow sample to circulate until sample temperature is within 1°C of the cell chamber.

9.6 Load the prepared sample into analyzer according to analyzer instruction manual.

9.7 Follow analyzer instruction manual to begin analysis of sample. Be sure to include the use of any necessary sedimentation parameters where necessary.

NOTE 3—Be aware that bubbles may need to be removed from the analysis cell prior to analysis. Some instrumentation perform an automatic scan for bubbles and, if detected, follow with a bubble elimination routine.

NOTE 4—It is recommended that the stirrer be turned off simultaneously with activation of the instrument.

9.8 Rinse the sedimentation cell and sample chamber thoroughly three times with fresh dispersing medium, according to analyzer instruction manual. If it is necessary to clear the cell further at this time, rinse three times with the dilute cleaning solution, followed by an additional cycle of rinses with fresh dispersing medium.

9.9 It is advised that a repeat analysis be performed on a separately weighed portion of the sample, thus providing two distributions on the same powder.

10. Report

10.1 A copy of all the data, either in graphical or tabular form, shall be supplied.

11. Precision and Bias

11.1 Precision—The results of an interlaboratory study to determine the precision of this test method are available in Research Report No. B09–1011,⁶ which is a report on a study done in nine laboratories on two tungsten carbide powders in the rod-milled condition. Although this is not in conformance with the requirements of Practice E 691 (three materials are required; six or more recommended), the user of this test method may infer its precision from this interlaboratory study. The pertinent conclusions are presented below:

11.1.1 The within-laboratory repeatability limit, r, for the median particle size (µm as defined by Terminology E 456), was found to be estimated by the following equation:

\[ r = 0.133M - 0.009 \]  

(2)

where

\[ M = \text{the measured median particle size (µm), in the range of 1.4 to 4.2 µm (r = 0.15 to 0.55 µm in this range).} \]

Duplicate median particle size results from the same laboratory should not be considered suspect unless they differ by more than r.

11.1.2 The between-laboratory reproducibility limit, R, for the median particle size (R as defined by Terminology E 456) was found to be estimated by the following equation:

\[ R = 0.482M - 0.489 \]  

(3)

where

\[ M = \text{the measured median particle size (µm) in the range of 1.4 to 4.2 µm (R = 0.19 to 1.54 µm in this range).} \]

Median particle size results from two different laboratories should not be considered suspect unless they differ by more than R.

11.2 Bias—No absolute method of determining powder particle size exists, nor are there any universally recognized standard or reference powders for this measurement. Therefore, it is not possible to discuss the bias results by this test method.

12. Keywords

12.1 metal powders; particle size; particle size distribution; powdered metals; refractory metal powders; sedimentation particle size distribution

⁶ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: B09–1011.
SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue (B 761 – 98) that may impact the use of this standard.

(1) A precaution concerning Reynolds number of sedimenting particles was added to the Scope in paragraph 1.2.1. Rationale: Since the quality of the results of this test method depend on the use of conditions that provide sedimentation within a laminar flow regime, it is important that the user of the test method be able to determine when such conditions exist. Paragraph 1.2.1 facilitates this determination.

(2) Section 11, Precision and Bias, was revised as a result of a completed interlaboratory study. Rationale: Results of the completed interlaboratory study allowed calculation of expected precision of this technique.

(3) Documents referenced in the revised section 11 were added to the list of referenced documents in Section 2.

(4) Reynolds numbers for 30 µm particles and maximum diameter sedimenting with Reynolds number of 0.3, are given for a number of materials in Table 1. Rationale: Since the quality of the results of this test method depend on the use of conditions that provide sedimentation within a laminar flow regime, it is important that the user of the test method be able to determine when such conditions exist. Table 1 facilitates this determination.
1. Scope

1.1 This test method provides sampling plans that are intended for use in the inspection of metallic and inorganic coatings on products for the purpose of deciding whether submitted lots of coated products comply with the specifications applicable to the coating.

1.2 The sampling plans are variables plans. In plans of this type, several articles of product are drawn from a production lot. A characteristic of the coating on the drawn articles is measured. The values obtained are used to estimate the number of articles in the lot that do not conform to a numerical limit, for example a minimum thickness. The number is compared to a maximum allowable.

1.3 Variables plans can only be used when the characteristic of interest is measurable, the test method gives a numerical measure of the characteristic, and the specification places a numerical limit on the measured value. It is also necessary that the variation of the characteristic from article to article in a production lot be normally distributed (see Appendix X2). Each article must be tested in the same way (for example, coating thickness must be measured at the same location, see X2.7) so that the values from article to article are comparable. If one or more of these conditions are not met, a variables plan cannot be used. Instead, an attributes plan must be used. These are given in Test Method B 602 and Guide B 697.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
- B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings

2.2 Military Standards:
- MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes
- MIL-STD-414 Sampling Procedures and Tables for Inspection by Variables for Percent Defective

2.3 Other:
- ANSI/ASQC Z1.4-1981 Sampling Procedures and Tables for Inspection by Attributes
- ANSI/ASQC Z1.9-1979 Sampling Procedures and Tables for Inspection by Variables for Percent Non-Conformance
- MIL-STD-414 Sampling Procedures and Tables for Inspection by Variables for Percent Defective

3. Terminology Definitions

3.1 destructive test—a test that destroys the tested article or makes it nonconforming to a requirement.

3.2 nondestructive test—a test that neither destroys the tested article nor makes it nonconforming to a requirement.

3.3 inspection lot—a collection of articles of the same kind that is submitted to inspection for acceptance or rejection as a group.

3.4 sample—articles randomly selected from an inspection lot whose quality is used to decide whether or not the inspection lot is of acceptable quality.

3.5 standard deviation—a measure of dispersion equal to the square root of the mean of the squares of the deviations from the arithmetic mean of the distribution (see 9.2.6).

4. Summary of Test Method

4.1 The plans in this test method provide the same protection as the attributes plans in Table 1, Table 2, and Table 3 of Test Method B 602 and are interchangeable with them when the conditions necessary for variables sampling exist. This method has no plan comparable to Table 4 of Test Method B 602, because variables plans are subject to an excessive probability of error when the number of nonconforming articles in a lot is expected to be approximately 1% or less as it is for the Table 4 plan. Also for this reason, comparable variables plans are not given for the smallest lot sizes of Table 1 and Table 2 of Test Method B 602. The plans of Table 4, Table 1, and Table 2 in Test Method B 602 are described as Level I, Level II, and Level III respectively. For consistency, Table 1 and Table 2 of this method are described as Level II

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1 This method is under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on General Test Methods.


2 Annual Book of ASTM Standards, Vol 02.05.

3 Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

4 Available from the American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.
since they are comparable to Table 1 of Test Method B 602, and Table 3 and Table 4 are described as Level III.

4.2 The main advantage of a variables sampling plan over an attributes plan is that fewer articles need to be inspected to obtain the same protection. For example, a sample of 12 using variables can give the same protection as a sample of 50 using attributes. On the other hand, more expensive test methods may be required to yield the measurements required by variables sampling.

4.3 Generally, thickness is the only characteristic of a coating that meets the conditions of a variables plan given in 1.3. For that reason, the plans in this method are designed to be used when the specification for the characteristic in question is a minimum value, which is the usual case for coating thickness. Variables plans can be used when the limit is a maximum and when there are both a minimum and a maximum. Plans for these cases are given in the references.

4.4 The sampling plans in Table 1 and Table 2 of this method are considered to be standard for nondestructive testing and will be used unless the buyer specifies otherwise. Table 5 and Table 6 will be used for destructive testing; these plans use smaller samples to reduce the cost of inspection with a resultant reduction of the ability to distinguish between conforming and nonconforming lots.

4.5 Additional variables plans are given in Appendix X3. Also found there are instructions for the calculation of plans for needs that are not covered.

5. Significance and Use

5.1 Sampling inspection permits the estimation of the overall quality of a group of product articles through the inspection of a relatively small number of product articles drawn from the group.

5.2 The specification of a sampling plan provides purchasers and sellers a means of identifying the minimum quality level that is considered to be satisfactory.

5.3 Because sampling plans yield estimates of the quality of a product, the results of the inspection are subject to error. Through the selection of a sampling plan, the potential error is known and controlled.

5.4 Sampling inspection is used when a decision must be made about what to do with a quantity of articles. This quantity

**TABLE 1 Level II—Sampling Plans for Nondestructive Tests, Standard Deviation Known**

<table>
<thead>
<tr>
<th>Inspection Lot Size</th>
<th>n</th>
<th>k</th>
<th>AQL</th>
<th>LQL</th>
<th>50/50 Point</th>
<th>AOQL</th>
</tr>
</thead>
<tbody>
<tr>
<td>91 through 280</td>
<td>7</td>
<td>1.664</td>
<td>1.1</td>
<td>12</td>
<td>4.8</td>
<td>2.4</td>
</tr>
<tr>
<td>281 through 500</td>
<td>12</td>
<td>1.649</td>
<td>1.7</td>
<td>10</td>
<td>5.0</td>
<td>2.6</td>
</tr>
<tr>
<td>501 through 1 200</td>
<td>16</td>
<td>1.712</td>
<td>1.7</td>
<td>8.2</td>
<td>4.4</td>
<td>2.3</td>
</tr>
<tr>
<td>1 201 through 3 200</td>
<td>25</td>
<td>1.704</td>
<td>2.1</td>
<td>7.4</td>
<td>4.4</td>
<td>2.5</td>
</tr>
<tr>
<td>3 201 through 10 000</td>
<td>36</td>
<td>1.778</td>
<td>2.0</td>
<td>5.9</td>
<td>3.8</td>
<td>2.2</td>
</tr>
<tr>
<td>10 001 through 35 000</td>
<td>52</td>
<td>1.829</td>
<td>2.0</td>
<td>4.9</td>
<td>3.4</td>
<td>2.1</td>
</tr>
<tr>
<td>Over 35 000</td>
<td>82</td>
<td>1.893</td>
<td>1.9</td>
<td>4.0</td>
<td>2.9</td>
<td>1.9</td>
</tr>
</tbody>
</table>

*The AQL, LQL, 50/50 Point, and AOQL are in percent.*

**TABLE 2 Level II—Sampling Plans for Nondestructive Tests, Standard Deviation Unknown**

<table>
<thead>
<tr>
<th>Inspection Lot Size</th>
<th>n</th>
<th>k</th>
<th>AQL</th>
<th>LQL</th>
<th>50/50 Point</th>
<th>AOQL</th>
</tr>
</thead>
<tbody>
<tr>
<td>91 through 280</td>
<td>16</td>
<td>1.663</td>
<td>1.0</td>
<td>12</td>
<td>4.8</td>
<td>2.4</td>
</tr>
<tr>
<td>281 through 500</td>
<td>29</td>
<td>1.649</td>
<td>1.7</td>
<td>10</td>
<td>5.0</td>
<td>2.6</td>
</tr>
<tr>
<td>501 through 1 200</td>
<td>40</td>
<td>1.713</td>
<td>1.7</td>
<td>8.2</td>
<td>4.3</td>
<td>2.2</td>
</tr>
<tr>
<td>1 201 through 3 200</td>
<td>61</td>
<td>1.704</td>
<td>2.1</td>
<td>7.4</td>
<td>4.4</td>
<td>2.5</td>
</tr>
<tr>
<td>3 201 through 10 000</td>
<td>92</td>
<td>1.778</td>
<td>2.0</td>
<td>5.9</td>
<td>3.8</td>
<td>2.2</td>
</tr>
<tr>
<td>10 001 through 35 000</td>
<td>137</td>
<td>1.829</td>
<td>2.0</td>
<td>4.9</td>
<td>3.4</td>
<td>2.1</td>
</tr>
<tr>
<td>Over 35 000</td>
<td>223</td>
<td>1.893</td>
<td>1.9</td>
<td>4.0</td>
<td>3.0</td>
<td>1.9</td>
</tr>
</tbody>
</table>

*The AQL, LQL, 50/50 Point, and AOQL are in percent.*

**TABLE 3 Level III—Sampling Plans for Nondestructive Tests, Standard Deviation Known**

<table>
<thead>
<tr>
<th>Inspection Lot Size</th>
<th>n</th>
<th>k</th>
<th>AQL</th>
<th>LQL</th>
<th>50/50 Point</th>
<th>AOQL</th>
</tr>
</thead>
<tbody>
<tr>
<td>51 through 150</td>
<td>6</td>
<td>1.432</td>
<td>1.8</td>
<td>18</td>
<td>7.6</td>
<td>3.8</td>
</tr>
<tr>
<td>151 through 280</td>
<td>10</td>
<td>1.411</td>
<td>2.7</td>
<td>16</td>
<td>7.9</td>
<td>4.1</td>
</tr>
<tr>
<td>281 through 500</td>
<td>14</td>
<td>1.470</td>
<td>2.8</td>
<td>13</td>
<td>7.1</td>
<td>3.5</td>
</tr>
<tr>
<td>501 through 1 200</td>
<td>23</td>
<td>1.492</td>
<td>3.3</td>
<td>11</td>
<td>6.8</td>
<td>3.8</td>
</tr>
<tr>
<td>1 201 through 3 200</td>
<td>30</td>
<td>1.551</td>
<td>3.2</td>
<td>9.4</td>
<td>6.0</td>
<td>3.5</td>
</tr>
<tr>
<td>3 201 through 10 000</td>
<td>44</td>
<td>1.618</td>
<td>3.1</td>
<td>7.7</td>
<td>5.3</td>
<td>3.2</td>
</tr>
<tr>
<td>10 001 through 35 000</td>
<td>66</td>
<td>1.680</td>
<td>3.0</td>
<td>6.4</td>
<td>4.6</td>
<td>3.0</td>
</tr>
<tr>
<td>Over 35 000</td>
<td>103</td>
<td>1.719</td>
<td>3.0</td>
<td>5.6</td>
<td>4.4</td>
<td>2.9</td>
</tr>
</tbody>
</table>

*The AQL, LQL, 50/50 Point, and AOQL are in percent.*

**TABLE 4 Level III—Sampling Plans for Nondestructive Tests, Standard Deviation Unknown**

<table>
<thead>
<tr>
<th>Inspection Lot Size</th>
<th>n</th>
<th>k</th>
<th>AQL</th>
<th>LQL</th>
<th>50/50 Point</th>
<th>AOQL</th>
</tr>
</thead>
<tbody>
<tr>
<td>51 through 150</td>
<td>12</td>
<td>1.433</td>
<td>1.7</td>
<td>19</td>
<td>7.6</td>
<td>3.8</td>
</tr>
<tr>
<td>151 through 280</td>
<td>19</td>
<td>1.410</td>
<td>2.6</td>
<td>16</td>
<td>7.9</td>
<td>3.7</td>
</tr>
<tr>
<td>281 through 500</td>
<td>29</td>
<td>1.470</td>
<td>2.8</td>
<td>13</td>
<td>7.1</td>
<td>3.8</td>
</tr>
<tr>
<td>501 through 1 200</td>
<td>48</td>
<td>1.494</td>
<td>3.3</td>
<td>11</td>
<td>6.7</td>
<td>3.8</td>
</tr>
<tr>
<td>1 201 through 3 200</td>
<td>66</td>
<td>1.551</td>
<td>3.2</td>
<td>9.4</td>
<td>6.0</td>
<td>3.5</td>
</tr>
<tr>
<td>3 201 through 16 000</td>
<td>102</td>
<td>1.618</td>
<td>3.1</td>
<td>7.7</td>
<td>5.3</td>
<td>3.2</td>
</tr>
<tr>
<td>16 001 through 35 000</td>
<td>159</td>
<td>1.680</td>
<td>3.0</td>
<td>6.4</td>
<td>4.6</td>
<td>3.0</td>
</tr>
<tr>
<td>Over 35 000</td>
<td>248</td>
<td>1.717</td>
<td>3.0</td>
<td>5.6</td>
<td>4.3</td>
<td>2.9</td>
</tr>
</tbody>
</table>

*The AQL, LQL, 50/50 Point, and AOQL are in percent.*

**TABLE 5 Sampling Plans for Destructive Tests, Standard Deviation Known**

<table>
<thead>
<tr>
<th>Inspection Lot Size</th>
<th>n</th>
<th>k</th>
<th>AQL</th>
<th>LQL</th>
<th>50/50 Point</th>
<th>AOQL</th>
</tr>
</thead>
<tbody>
<tr>
<td>26 through 1 200</td>
<td>5</td>
<td>1.262</td>
<td>2.3</td>
<td>25</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>1 201 through 35 000</td>
<td>10</td>
<td>1.411</td>
<td>2.7</td>
<td>16</td>
<td>7.9</td>
<td></td>
</tr>
<tr>
<td>Over 35 000</td>
<td>14</td>
<td>1.519</td>
<td>2.5</td>
<td>12</td>
<td>6.5</td>
<td></td>
</tr>
</tbody>
</table>

*The AQL, LQL, and 50/50 Point are in percent.*
may be a shipment from a supplier, articles that are ready for a subsequent manufacturing operation, or articles ready for shipment to a customer.

5.5 In sampling inspection, a relatively small number of articles (the sample) is selected randomly from a larger number of articles (the inspection lot); the sample is inspected for conformance to the requirements placed on the articles. Based on the results, a decision is made whether or not the lot conforms to the requirements.

5.6 Since only a portion of a production lot is inspected, the quality of the uninspected articles is not known. The possibility exists that some of the uninspected articles are nonconforming. Therefore, basic to any sampling inspection plan is the willingness of the buyer to accept lots that contain some nonconforming articles. The number of nonconforming articles in accepted lots is controlled by the size of the sample and the criteria of acceptance that are placed on the sample.

5.7 Acceptance sampling plans are used for the following reasons:

5.7.1 When the cost of inspection is high and the consequences of accepting a nonconforming article are not serious.

5.7.2 When 100 % inspection is fatiguing and boring and, therefore, likely to result in errors.

5.7.3 When inspection requires a destructive test, sampling inspection must be used.

5.8 In acceptance sampling by variables, the coating characteristic of each article in the sample is measured. Using the arithmetic mean of these values, the standard deviation of the process, and the factor \( k \) that is found in the Tables, a number is calculated (see 9.3). If this number equals or exceeds the specified minimum, the inspection lot conforms to the requirements. If it is less, the lot does not conform. If the standard deviation of the process is not known, the standard deviation of the sample is calculated and used.

5.9 The use of a sampling plan involves the balancing of the costs of inspection against the consequences of accepting an undesirable number of nonconforming articles. There is always a risk that a random sample will not describe correctly the characteristics of the lot from which it is drawn, and that an unacceptable lot will be accepted or an acceptable lot will be rejected. The larger the sample, the smaller this risk but the larger the cost of inspection.

5.10 To understand the risks, consider that if every article in an inspection lot conforms to its requirements, every article in the sample will conform also. Such lots will be accepted (Note 1). If only a few articles in an inspection lot are nonconforming, the sample probably will indicate that the lot is acceptable; but there is a small probability that the sample will indicate that the lot is unacceptable. The larger the proportion of nonconforming articles in an inspection lot, the more likely it will be that the sample will indicate that the lot is unacceptable. If every article in an inspection lot is nonconforming, a sample will always indicate that the lot is unacceptable.

**Note 1**—Throughout this method, it is assumed that no mistakes are made in sampling, measurement, and calculation.

5.11 The probability of accepting an inspection lot that contains nonconforming items is often described in terms of the Acceptable Quality Level (AQL) and the Limiting Quality Level (LQL). The AQL is the quality level that is considered to be acceptable. The LQL is a quality level that is considered to be barely tolerable. A sampling plan is selected that has a high probability of accepting lots of AQL quality and of rejecting lots of LQL quality. In this method, the AQL given for a sampling plan is the quality level of lots that have a 95 % probability of being accepted. The LQL is the quality level of lots that have a 10 % probability of being accepted or, in other words, a 90 % probability of being rejected. The tables in this method give the AQL and LQL of each plan. They also give the 50/50 point, the quality level of a lot that is just as likely to be accepted as rejected.

5.12 The disposition of nonconforming inspection lots is beyond the scope of this method because, depending on the circumstances, lots may be returned to the supplier, kept and used, put to a different use, scrapped, reworked, or dealt with in some other way. An alternative is rectifying inspection in which rejected lots are screened and used.

5.13 In rectifying inspection, when an inspection lot is rejected, all of the articles in the lot are inspected and nonconforming ones are removed. They may be replaced with conforming articles. The now 100 % conforming lot is accepted. With this practice, the average quality level for a series of lots taken as a whole will be better because of the addition of the 100 % conforming lots. When the incoming lots are of a good quality level, the average quality level of a series of lots will be even better when the rejected lots are rescreened and resubmitted. When incoming lots are of a poor quality level, the average quality of a series of accepted lots will again be good because many of the incoming lots will be rejected and upgraded. At intermediate quality levels of incoming lots, the average quality level of a series of accepted lots will again be improved, but it will not be improved as much as in either of the above cases; and there will be an intermediate quality level where the degree of improvement is the least. This improved quality level is called the Average Outgoing Quality Limit (AOQL). It is the worst condition that can occur under rectifying inspection. The tables give the AOQL for each plan. There is no AOQL for the plans used with destructive tests because destructive tests cannot be used to screen rejected lots.

**Note 2**—The AOQLs given in the tables are strictly correct only when the sample is small with respect to the lot. If this is not the case, the correct AOQL will be smaller than the tabulated value. The correct values are obtained by multiplying the tabulated values by the following equation:

\[
1 - \frac{\text{sample size}}{\text{lot size}}
\]  

(1)

5.14 Rectifying inspection will substantially increase the cost of inspection if the incoming lots are much worse than AQL quality.

### TABLE 6 Sampling Plans for Destructive Tests, Standard Deviation Unknown*

<table>
<thead>
<tr>
<th>Inspection Lot Size</th>
<th>n</th>
<th>k</th>
<th>AQL</th>
<th>LQL</th>
<th>50/50 Point</th>
</tr>
</thead>
<tbody>
<tr>
<td>26 through 1 200</td>
<td>9</td>
<td>1.181</td>
<td>2.8</td>
<td>12</td>
<td></td>
</tr>
<tr>
<td>1 201 through 35 000</td>
<td>19</td>
<td>1.412</td>
<td>2.5</td>
<td>16</td>
<td></td>
</tr>
<tr>
<td>Over 35 000</td>
<td>34</td>
<td>1.497</td>
<td>2.8</td>
<td>12</td>
<td></td>
</tr>
</tbody>
</table>

*The AQL, LQL, and 50/50 Point are in percent.
5.15 Rectifying inspection is used only when required by the purchaser.

6. Ordering Information

6.1 Unless otherwise specified by the purchaser, the sampling plans given in Table 1 and Table 2 will be used for nondestructive testing, and the plans given in Table 5 and Table 6 for destructive testing.

6.2 When either a nondestructive or a destructive test can be used to inspect an article for conformance to a particular requirement, the purchaser should specify which test is to be used. When a test is neither clearly nondestructive nor destructive, the purchaser should specify which it is considered to be.

Note 3—The nature of a destructive test can be such that the tested article can be reclaimed, for example by stripping and reapplying the coating. Other tests can destroy the coating in nonessential locations, in which case the article can still be functional. In these instances, the purchaser needs to decide and state whether the tests are to be considered destructive or nondestructive.

6.3 Rectifying inspection will be used only when specified by the purchaser. When rectifying inspection is used, nonconforming articles will be replaced with conforming ones only when specified by the purchaser.

7. Formation of Inspection Lot

7.1 An inspection lot shall be formed from articles that are of the same kind, that have been produced to the same specification, and that have been coated by a single supplier at identical conditions.

Note 4—These requirements are intended to ensure that the lot is homogeneous and that variations between articles in the lot are the result only of the inherent variation of the production process (see Appendix X1).

8. Sampling

8.1 General—A sample shall be selected randomly from the inspection lot. If the test method to be used is nondestructive, the sample size shall be that directed in 8.2. If the test method is destructive, the sample size shall be that directed in 8.3.

8.2 Nondestructive Tests—For nondestructive testing, the size of the sample shall be that specified for the sampling plan level that is required by the purchaser. The sampling plans are given for Level II in Table 1 and Table 2 and for Level III in Table 3 and Table 4. If the purchaser does not specify the level, Level II shall be used. The plans in Table 1 and Table 3 shall be used when the standard deviation of the coating process is known. Table 2 and Table 4 plans shall be used when the standard deviation is not known and must be estimated from the sample values.

8.3 Destructive Tests—For destructive testing, the size of the sample shall be that specified in Table 5 when the standard deviation of the process is known and Table 6 when it is not known.

8.4 The sample shall be drawn randomly from the inspection lot, that is, in a manner that ensures each article an equal chance of being selected regardless of other considerations such as location in the inspection lot, appearance, quality, location on a fixture during coating, and chronological relationship to the other articles. Random sampling procedures are given in the Appendixes.

9. Calculations

9.1 Calculate the arithmetic mean of the measured characteristic by adding the values obtained for the articles and dividing the number of articles that were tested using the following equation:

\[
\bar{X} = \frac{\sum_{i=1}^{n} X_i}{n}
\]  

where:

- \( \bar{X} \) = arithmetic mean of the measured values,
- \( X_i \) = measured value,
- \( \sum_{i=1}^{n} X_i \) = sum of the measured values, and
- \( n \) = number of articles tested.

9.2 If the standard deviation of the coating process is known, continue the calculations as directed in 9.3. The symbol for the standard deviation for the process is \( \sigma \). If the standard deviation for the process is not known, calculate an estimated value from the measurements obtained from the sample as directed in 9.2.1 through 9.2.6. The symbol for this estimated standard deviation is \( s \).

9.2.1 Subtract the arithmetic mean from the first measured value using the following equation:

\[
X_1 - \bar{X}
\]  

9.2.2 Calculate the square of the difference obtained in 9.2.1 using the following equation:

\[
(X_1 - \bar{X})^2
\]  

9.2.3 Repeat 9.2.1 and 9.2.2 for each measured value.

9.2.4 Add all of the squares obtained in 9.2.2 and 9.2.3 using the following equation:

\[
(X_1 - \bar{X})^2 + (X_2 - \bar{X})^2 + \ldots + (X_n - \bar{X})^2 = \sum_{i=1}^{n} (X_i - \bar{X})^2
\]  

9.2.5 Divide the sum obtained in 9.2.4 by one less than the number of articles that were tested using the following equation:

\[
\frac{\sum_{i=1}^{n} (X_i - \bar{X})^2}{n - 1}
\]  

9.2.6 Calculate the square root of the value obtained in 9.2.5 using Eq 6. This is standard deviation, \( s \).

\[
s = \sqrt{\frac{\sum_{i=1}^{n} (X_i - \bar{X})^2}{n - 1}}
\]  

Note 5—The following equation can also be used:

\[
s = \sqrt{\frac{\sum X_i^2 - (\sum X_i)^2}{n - 1}}
\]  

9.3 Using the \( k \) that is in the table and the standard deviation
from 9.2, calculate the following number when the standard deviation is known:

$$\bar{X} - k\sigma$$  \hspace{1cm} (9)

or, calculate the following when the standard deviation is not known:

$$\bar{X} - ks$$  \hspace{1cm} (10)

10. Inspection and Lot Classification

10.1 Inspection—Each article in the sample shall be inspected as directed in the applicable coating standard.

10.2 Lot Classification:

10.2.1 The number calculated in 9.3 shall be compared to the minimum number stated in the coating specification. If the number in 9.3 equals or exceeds the specified minimum, the lot conforms to the requirements. If it is less than the specified minimum, the lot does not conform.

10.2.2 When specified by the purchaser, nonconforming lots shall be 100% inspected, and nonconforming articles shall be removed. When required by the purchaser, the nonconforming articles shall be replaced with conforming articles.

APPENDIXES

(Nonmandatory Information)

X1. DRAWING OF SAMPLES

X1.1 The success of acceptance sampling is totally dependent on the sample being drawn from the lot at random. Random sampling means that the selection of an article for the sample is totally by chance and that every article in the lot is equally likely to be selected. If the articles in the inspection lot are thoroughly mixed, such as barrel-plated articles, a sample drawn from anywhere in the lot will be random (see X2.5). Rack-plated articles cannot be sampled this way unless thorough mixing is done before sampling, otherwise a random sampling procedure must be used. Methods of random sampling are described in the following paragraphs.

X1.2 When random numbers are used to select a sample, each article in the lot is identified by a different number. If the units have serial numbers, the serial numbers can be used. The numbers of the articles that are to be inspected are selected from a table of random numbers such as Table X1.1. Other tables of random numbers can be obtained from books on statistics. Some pocket calculators are designed to generate random numbers.

X1.3 As an example, assume that a sample of 12 articles is to be selected from an inspection lot of 80 articles. The articles are numbered 1 through 80. A pencil is allowed to fall blindly at some number in Table X1.1. Starting at this point, a coin is tossed to decide whether to go up or down the column; heads, up; tails, down. If the pencil falls on column 10, line 11, and the coin is tails; the decision is to read down the column until 12 numbers are chosen. Take the first two digits in each group of five digits. The selection of random numbers is made as follows: the 85’s are rejected because they are over 80, and the second 06 is rejected because it has already appeared. The sample then consists of articles numbered 31, 20, 8, 26, 53, 65, 64, 46, 22, 6, 41, and 67.

X1.4 When product items are arranged in an order without regard to quality, such as articles in a tray, a sample can be drawn by using the constant interval procedure. Here, a constant interval is maintained between the items drawn for the sample. For example, every 9th, 19th, or 24th unit is selected. The first item drawn from the lot can be determined from the table of random numbers. All other items are then drawn at a constant interval following the first item. The constant interval is determined by dividing the lot size by the sample size.

X1.5 As an example, assume that a lot of 3000 items is to be inspected. In accordance with Table 3, a sample of 30 items is to be drawn. The constant interval is 100 (3000 divided by 30). A random number from 1 to 100 is selected either from a table or by another appropriate method. After the first item is taken, the remaining items in the required sample are drawn by selecting every 100th item from the lot until 30 are selected.

X1.6 References (1 through 10) give additional information and procedures on random sampling.

X1.7 The numbers of a random sample can be generated by the following Microsoft BASIC computer program:

---

5 The boldface numbers in parentheses refer to the list of references appended to this method.
10 REM—Program to select random samples for testing
20 PRINT "MATRIX LOADED"
30 PRINT "FOR A LOT SIZE OF:";LPRINT "FOR A LOT SIZE OF:";L
40 PRINT "AND A SAMPLE SIZE OF:";S:LPRINT "AND A SAMPLE SIZE OF:";S
50 PRINT "THE SAMPLE NUMBERS ARE:";
60 FOR R = 1 TO S
70 PRINT A(M);",";:LPRINT A(M);",";
80 IF A(M) <> 0 THEN 220
90 NEXT R
100 PRINT "THE END OF SAMPLE LIST";LPRINT "THE END OF SAMPLE LIST"
110 NEXT S
120 NEXT R

TABLE X1.1 Table of Random Numbers

<table>
<thead>
<tr>
<th>Line</th>
<th>Column</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10480</td>
</tr>
<tr>
<td>2</td>
<td>22368</td>
</tr>
<tr>
<td>3</td>
<td>24130</td>
</tr>
<tr>
<td>4</td>
<td>42167</td>
</tr>
<tr>
<td>5</td>
<td>57670</td>
</tr>
<tr>
<td>6</td>
<td>77921</td>
</tr>
<tr>
<td>7</td>
<td>99562</td>
</tr>
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<td>8</td>
<td>96301</td>
</tr>
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<td>9</td>
<td>89579</td>
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<td>10</td>
<td>85475</td>
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<td>11</td>
<td>28918</td>
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<td>12</td>
<td>63553</td>
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<td>16</td>
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<td>00498</td>
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<td>29676</td>
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<td>28</td>
<td>00742</td>
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<td>05366</td>
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<td>00582</td>
</tr>
<tr>
<td>32</td>
<td>00275</td>
</tr>
<tr>
<td>33</td>
<td>69011</td>
</tr>
<tr>
<td>34</td>
<td>57695</td>
</tr>
<tr>
<td>35</td>
<td>25976</td>
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<td>91567</td>
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<td>40</td>
<td>92157</td>
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<td>41</td>
<td>14577</td>
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<td>98427</td>
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<td>43</td>
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<td>44</td>
<td>70060</td>
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<td>45</td>
<td>53976</td>
</tr>
<tr>
<td>46</td>
<td>76072</td>
</tr>
<tr>
<td>47</td>
<td>90725</td>
</tr>
<tr>
<td>48</td>
<td>64364</td>
</tr>
<tr>
<td>49</td>
<td>09862</td>
</tr>
<tr>
<td>50</td>
<td>95012</td>
</tr>
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<td>51</td>
<td>15664</td>
</tr>
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<td>52</td>
<td>16408</td>
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<tr>
<td>53</td>
<td>18629</td>
</tr>
<tr>
<td>54</td>
<td>73115</td>
</tr>
<tr>
<td>55</td>
<td>57491</td>
</tr>
</tbody>
</table>
X.2 NORMAL DISTRIBUTION

X.2.1 Articles produced by a manufacturing process are never identical. Minor variations in the process occur that affect the characteristics of the articles. Such variations often occur at random and tend to cancel each other out. Under these conditions, the articles are quite similar to each other. Less often, the chance variations do not cancel out and some articles will differ from the typical. As a result, it is quite common for the characteristics of the articles to vary from the average, and the greater the deviation the fewer articles there are. Frequently, this distribution of the articles can be closely described by a mathematical equation, which when plotted, gives the bell-shaped curve shown in Fig. X.2.1. This is called a normal Gaussian distribution.

Note X.2.1—There is also a random variation introduced by the measurement method. This normally is small relative to the product variation and, thus, of little consequence.

X.2.2 Along the horizontal, X axis in Fig. X.2.1 is plotted the numerical value of the characteristic that is being considered, for example, the thickness of the coating. The area beneath the curve and above the X axis represents the total of all the articles of a given thickness in a production lot. The arithmetic mean thickness is X, which is at the middle of the curve. The vertical line at X divides the curve in half so that half of the area is to the left, thicknesses less than the mean, and half is to the right, thicknesses greater than the mean. It can be seen that if a plating thickness specification is given as a minimum value, and if the mean thickness of the lot equals the specification value, the thickness of the plating on half of the parts will be below the specification limit; that is, half of the article will be nonconforming. Usually, it is required that most of the articles be conforming, which means that the mean thickness has to exceed the specified minimum. The standard deviation can be used to determine by how much.
X2.3 If the mean thickness is one standard deviation higher than the specified minimum thickness, the thickness will be less than the specified minimum on 16% of the articles. If the mean is two standard deviations above the specification value, there will be about 2.3% nonconforming articles in the lot. Hence, if the standard deviation of the process is known, a mean thickness can be calculated that will ensure that no more than a given percentage (for example, the AQL) of a lot will be nonconforming. Once a lot of articles is produced, a random sample of the articles can be inspected and their mean calculated. If the mean is equal to or larger than the required mean, it is known that the percentage of nonconforming articles in the lot is no more than the AQL. Actually, the mean of the sample can be different from that of the lot. In variables plans, an additional factor is placed in the calculation to allow for this. In processes where the standard deviation is not known and the sample standard deviation is used in the calculation, there is another potential error. This is guarded against by using larger samples.

X2.4 It is important to remember that variables sampling plans are based on the normal curve. If the product of a manufacturing process is not distributed normally, the use of a variables plan based on the normal curve will give invalid results. Therefore, statistical tests should be made of a process to confirm that the product characteristic is approximately normally distributed before a variables plan is used in the sampling inspection of the product. Tests for normality are described in Ref. (9).

X2.5 The distribution of plating thickness on barrel-plated articles tends to be normal, provided good barrel plating practices are observed. A production lot that consists of several barrel loads will also be normal if the loads are produced under essentially the same conditions. But if two or more loads that have different average thicknesses are mixed, the mixed lot may not conform to the normal distribution.

X2.6 The distribution of coating thickness on products that are processed on racks may or may not be normal. Tests of normality are required to determine if a variables plan can be used in these cases.

X2.7 The thickness of the coating must be measured at the same location on every article in the sample. If this is not done, the variation in thickness that occurs naturally over the surface of a product will result in invalid values for the average and the standard deviation.

### TABLE X3.1 Sampling Plans, Standard Deviation Known

<table>
<thead>
<tr>
<th>LQL</th>
<th>1</th>
<th>2</th>
<th>5</th>
<th>10</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>n</td>
<td>k</td>
<td>50/50</td>
<td>AOQL</td>
</tr>
<tr>
<td>5</td>
<td>18</td>
<td>1.943</td>
<td>1.4</td>
<td>51</td>
</tr>
<tr>
<td>10</td>
<td>8</td>
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<td>2.6</td>
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<td>15</td>
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<td>20</td>
<td>4</td>
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<td>25</td>
<td>3</td>
<td>1.396</td>
<td>8.1</td>
<td>4.2</td>
</tr>
</tbody>
</table>
X3.1 Table X3.1 and Table X3.2 provide additional sampling plans that may be useful in situations where the standard plans of Tables 2-6 are unsuitable. The plans of Table X3.1 are to be used when the standard deviation of the process is known. The plans of Table X3.2 are to be used when the standard deviation is not known.

X3.2 The plans in Table X3.1 and Table X3.2 provide AQL’s of 1, 2, 5, and 10 % and LQL’s of 5, 10, 15, 20, and 25 %. To select a plan, go to the table, find the column headed with the desired AQL, read down to the row headed by the desired LQL, and note the sample size \( n \) and \( k \).

<table>
<thead>
<tr>
<th>LQL, %</th>
<th>1</th>
<th>2</th>
<th>5</th>
<th>10</th>
</tr>
</thead>
<tbody>
<tr>
<td>( n )</td>
<td>( k )</td>
<td>50/50</td>
<td>AOQL</td>
<td>( n )</td>
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</table>

### TABLE X4.1 Values of \( z \) Used in Designing Variables Sampling Plans

<table>
<thead>
<tr>
<th>AQL or LQL</th>
<th>% Nonconforming</th>
<th>( z )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2.326</td>
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<tr>
<td>2</td>
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<tr>
<td>12.5</td>
<td>1.150</td>
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<td>15</td>
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</tr>
<tr>
<td>20</td>
<td>0.8416</td>
<td></td>
</tr>
<tr>
<td>25</td>
<td>0.6745</td>
<td></td>
</tr>
</tbody>
</table>

### X4.4.1 Calculate \( n \) as follows:

\[
 n = 8.564/(z_1 - z_2)^2 \quad (X4.5)
\]

Round to 14.

### X4.4.2 Calculate \( k \) as follows:

\[
 k = (\sqrt{4n} + z_2) - 0.3633)/2\sqrt{n} \quad (X4.6)
\]
REFERENCES


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Standard Test Method for Simultaneous Thickness and Electrode Potential Determination of Individual Layers in Multilayer Nickel Deposit (STEP Test)\textsuperscript{1}

This standard is issued under the fixed designation B 764; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (\(\epsilon\)) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method closely estimates the thickness of individual layers of a multilayer nickel electrodeposit and the potential differences between the individual layers while being anodically stripped at constant current density.\textsuperscript{2,3}

1.2 This test method does not cover deposit systems other than multilayer electroplated nickel deposits.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards: \textsuperscript{4}

- B 456 Specification for Electrodeposited Coatings of Copper Plus Nickel Plus Chromium and Nickel Plus Chromium
- B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
- D 1193 Specification for Reagent Water

3. Summary of Test Method

3.1 This procedure is a modification of the well-known coulometric method of thickness testing (Test Method B 504). It is also known as the anodic dissolution or electrochemical stripping method.

3.2 Coulometric thickness testing instruments are based on the anodic dissolution (stripping) of the deposit at constant current, while the time is measured to determine thickness. As commonly practiced, the method employs a small cell that is filled with an appropriate electrolyte, and the test specimen serves as the bottom of the cell. To the bottom of the cell is attached a rubber or plastic gasket whose opening defines the measuring (stripping, anodic) area. If a metallic cell is used, the rubber gasket also electrically insulates the test specimen from the cell. With the specimen as the anode and the cell or agitator tube as the cathode, a constant direct current is passed through the cell until the nickel layer is dissolved. A sudden change in voltage between the electrodes occurs when a different metallic layer starts to dissolve.

3.3 Each different metal or species of the same metal requires a given voltage to keep the current constant while being stripped. As one nickel layer is dissolved away and the next layer becomes exposed, there will be a voltage change (assuming a constant current and difference in the electrochemical characteristics of the two nickel layers). The elapsed time at which this voltage change occurs (relative to the start of the test or previous voltage change) is a measure of the deposit thickness.

3.4 At the same time, the amplitude of the voltage change can be observed. That is, the ease (or difficulty) with which one layer can be dissolved or stripped with reference to another layer can be compared. The lower the voltage needed the more active the metal or the greater the tendency to corrode preferentially to a more noble metal adjacent to it.

3.5 Where the metallic layers are of such a similar nature that change of the stripping voltage is small, there can be problems in detecting this change if the voltage between the deplating cell (cathode) and the sample (anode) is measured. The sample is dissolved anodically, cathodic processes are occurring on the deplating cell (cathode) surface that can also give rise to voltage changes, due to alterations of the cathode surface, thus obscuring the anode voltage change. This difficulty can be avoided by measuring the potential of the dissolving anodic sample with respect to an unpolarized third electrode (reference) placed in the cell. By recording this potential any difference in electrochemical activity between
layers is more readily detected. The equipment may be calibrated against standards with known STEP values.

3.6 The thickness of any specific nickel layer may be calculated from the quantity of electricity used (current multiplied by time), area dissolved, electrochemical equivalent of nickel, anode efficiency, and density of the nickel layer.

3.7 Commercial instruments using this principle are available. They are usually a combination coulometric and STEP instrument. Reference standards are available to calibrate the instrument. The STEP Test, as is the Coulometric Test, is rapid and destructive to the coating.

4. Significance and Use

4.1 The ability of a multilayer nickel deposit to enhance corrosion resistance is a function of the difference in the electrode potentials of the nickel layers (as measured individually at a fixed current density in a given electrolyte versus a reference electrode) and the thicknesses of the layers. The potential differences must be sufficient to cause the bright nickel or top layer to corrode preferentially and sacrificially with respect to the semi-bright nickel layer beneath it.

4.2 This test procedure allows the measurement of these potential differences directly on an electroplated part rather than on separate foil specimens in such a way that time determines the thickness of each layer, while the potential difference between nickel layers is an indication of the corrosion resistance of the total nickel deposit.

4.3 The interpretation and evaluation of the results of this test should be by agreement between the purchaser and the manufacturer.

Note 1—This test may be used as a quality assurance test of the multilayer nickel coatings applied in production. It should be understood that due to many factors that influence the progress of corrosion during actual use of the part, the performance of different multilayer nickel deposits in the test cannot be taken as an absolute indicator of the relative corrosion resistance of these deposits in service.

5. Apparatus

5.1 Composition of the Electrolyte:

- Nickel Chloride (NiCl₂·6H₂O) 300g/L
- Sodium Chloride (NaCl) 50g/L
- Boric Acid (H₃BO₃) 25g/L
- pH 3.0

The pH may be adjusted with diluted hydrochloric acid or sodium hydroxide, as required, and is more critical than the composition of the electrolyte.

Prepared in Purified Water—Type IV or better as specified in Specification D 1193.

5.2 Constant Current Source—This should supply a constant current that can be varied between 0 and 50 mA (typical 25 to 35 mA). A current of 30 mA corresponds to a stripping rate of 7.8 μm/min at 100 % current efficiency when used with a gasket providing 0.08 cm² stripping area. (This is achieved with the solution stated in 5.1.) Most commercial coulometric thickness testers can be used as the current source.

5.3 Electrolyte Agitation Source—All commercial coulometric thickness testers incorporate a means to agitate the solution. It is possible to purchase these types of units separately, if so desired, to be used externally in conjunction with other power supplies.

5.4 Recorder—Any time-based recorder with an input impedance of at least 1.0 MΩ and capable of running at approximately 0.5 mm/s (3 cm/min) can be used.

5.5 Deplating Cell—The cell may be similar in construction to commercially available coulometric deplating cells. It is usually a cup-shaped cell of either 316 stainless steel, copper-nickel alloy, or plastic that engages a round rubber or plastic gasket to the work piece or sample. The opening through the cell and gasket allows contact of the electrolyte to the test specimen and defines the stripping area.

Note 2—A coulometric deplating cell could be constructed of plastic using a cylindrical stainless steel or copper-nickel alloy sheet cathode located in the larger upper area of the cup. The advantages of such a cell are the prevention of whisker growth and the choking off of the small bore opening, and the ease of cathode removal for cleaning or replacement.

5.6 Reference Electrode—Either silver or platinum wire of approximately 1.5 mm in diameter can be used. Silver is probably the better choice due to its ability to form a silver-silver chloride electrode when used in a chloride containing electrolyte. The tip of the reference electrode should extend so that the distance between the tip of the electrode and the bottom of the agitator tube is approximately 5 mm.

Note 3—It is necessary to condition the silver electrode before using in order to form the silver-silver chloride surface. This is easily done by anodically treating approximately a 75-mm length of wire in 1 N hydrochloric acid solution for 10 to 15 s using 35-mA anodic current. This will form a gray film on the wire, which should always be present. Once the gray film is formed, it is not necessary to repeat the conditioning treatment unless the film has been removed. It may be advisable, however, to recondition the electrode after a prolonged period of inactivity or when the electrode has been allowed to remain dry for an extended period of time. Drying off the electrode should be avoided by immersion in either the hydrochloric acid conditioning solution, the step test solution, or distilled water when not in use.

Note 4—A ceramic junction reference electrode that does not require conditioning is available commercially.

5.7 Millivolt Meter (optional)—When using a sensitive and well-calibrated recorder, a millivolt meter is not necessary. If one is desired, however, any sensitive, high-input impedance meter can be used. A standard pH meter with a millivolt setting would be satisfactory. The meter should have a range from 0 to 2000 mV. If a millivolt meter is used which has low-output impedance facilities, it can be used in parallel to drive the recorder and will serve as a buffer amplifier. Most laboratory pH meters have such output terminals.

6. Procedure

6.1 Set up equipment as recommended by the manufacturer. If necessary, turn on the recorder and the millivolt meter and allow them to warm up.

6.2 If chromium is present on the nickel surface, remove it with concentrated hydrochloric acid. Make sure the nickel surface is clean. Rinse well and dry off the surface.
6.3 Position the test specimen in a secure horizontal position so that the chromium-stripped nickel surface is directly beneath the cell gasket.

6.4 Lower the coulometric deplating cell assembly; secure by sealing the gasket to the nickel surface. A flat test area of approximately 10 mm in diameter is desirable but not required. The criterion is that there be no leakage of the electrolyte. If leakage does occur, discontinue test and start a new one.

6.5 Fill the coulometric deplating cell to the appropriate level with the step test solution making sure that no air is trapped within the solution.

6.6 Lower the reference electrode assembly into the coulometric deplating cell, if necessary. The positioning of the reference electrode should be such that the distance from the end of the electrode to the test specimen is reproducible to within 1 mm and be held constant throughout the test.

6.7 Check all electrical connections. Make sure all connections are secure and that no corrosion exists at the contact points and that all contact points are secure.

6.8 Start the recorder (turn on milliamperemeter, if used). The recorder must be calibrated in order to determine the thickness of the nickel layers. This may be accomplished by using commercially available thickness standards or by applying Faraday’s Law. The latter requires information about the current, corroding area, electrochemical equivalent of nickel, density of nickel, efficiency, and the time base of the recorder (see 6.11).

6.9 Turn on the constant current source and agitator, which in turn will start the deplating reaction. Continue recording until the surface underlying the nickel is reached. This end point can be recognized graphically by a sudden change in voltage. If the basis metal is zinc, iron, or steel, the voltage will decrease; if it is copper or brass, the voltage will increase.

6.10 Stop the test by turning off the agitator, constant current source, recorder, and milliamperemeter. Remove the electrode assembly, if necessary, and empty the cell of the stripping solution. Wash the cell three times with purified water (Type IV or better as specified in Specification D 1193) before continuing to the next test.

6.11 This test is based on a measured current-time relationship necessary to remove a given amount of nickel from a specific area.

Example: if the constant current source produces 30 mA, the recorder time base is 30 mm/min, and the deplating area is 0.08 cm², it would take 19.2 s to deplate 2.5 μm of nickel. The chart would travel 9.6 mm. A general equation that may be used is as follows:

\[
\frac{(SL)(A)(I)}{0.303S} = T
\]

where:

\[ SL = \text{chart scan length, mm,} \]
\[ S = \text{chart speed, mm/min,} \]
\[ I = \text{cell current, mA,} \]
\[ A = \text{deplating area, cm,} \]
\[ T = \text{nickel thickness, μm, and} \]
\[ 0.303 = \text{constant calculated from the electrochemical equivalent and density of nickel.} \]

NOTE 7—Commercial units are available that will modify and may simplify the above procedure.

7. Factors Affecting the Accuracy of the Method

7.1 Excessive Metal Build-Up in Coulometric Deplating Cell—Excessive buildup of deposited nickel or the formation of “whiskers” on the inside of the coulometric deplating cell (cathode), especially near the gasket hole, can cause erratic results and produce “noisy” curves. When buildup is observed, remove it completely according to the manufacturer’s instructions or as follows:

7.1.1 If a metallic cell is used as a cathode:

7.1.1.1 Ream with a round, fine file. (A drill or reamer may be used.)

7.1.1.2 Soak for 15 to 20 s in a solution of four parts concentrated sulfuric acid and one part concentrated nitric acid. If 316 stainless steel is used for the cell, it may be soaked in concentrated nitric acid until all nickel is dissolved.

7.1.1.3 Rinse in water (Type IV or better as specified in Specification D 1193) and dry.

7.1.1.4 Repeat 7.1.1.1 to 7.1.1.3 as many times as necessary to remove all metallic buildup. This cleaning process should be done after every ten tests or more frequently, if necessary.

NOTE 8—It has been found that giving the coulometric deplating cell (cathode) a nickel strike prior to using will help prevent erratic buildup or treeing around the gasket hole and cleaning will not be required as often.

7.1.2 If a metallic agitator tube is used as a cathode:

7.1.2.1 Place a stainless steel or nickel plate under the gasket, lower the cell, and rinse it with DI water.

7.1.2.2 Fill the cell with 2 to 2.5 M H₂SO₄, reverse the polarity of the current and strip the nickel from agitator tube. A cleaning current of about 55 mA for about 45 s should suffice. If the nickel is not completely removed, drain the cell, refill it with H₂SO₄, and repeat the cleaning.

7.1.2.3 Wash the cell thoroughly with water.

7.1.2.4 If the tube still looks coated, remove the coating by rubbing the agitation tube with a soft rubber eraser, followed by washing with water.

7.2 Reference Electrode Preparation—If the electrode has not been used for a day or has been allowed to dry for a period of time, one or two conditioning runs will have to be made prior to running a meaningful test. (See Note 2.)
7.3 **Cleanliness of Test Surface**—Make sure the surface area to be tested is free of water breaks, foreign material, etc. Nickel surfaces that have been exposed to air for some time may have become passive. Abrade lightly prior to testing to remove any oxide films present. (Abrading mildly with an eraser usually suffices: if not, clean with dilute sulfuric acid.)

7.4 **Anode Area Variation**—Use only enough pressure on the gasket to seal it to the test area without solution leaks. Excess pressure can distort the gasket and change the anode area affecting the thickness results. If test results vary significantly, examine the resulting depleted area with a magnifying glass to determine if the area has varied in size. Small variations in the anodic area can give large variations in test results. The area defined by the gasket can vary significantly between gaskets. When a different gasket is used, recalibrate the instrumentation.

7.5 **Electrical Noise**—To obtain good, smooth curves, eliminate all electrical noise caused by extraneous voltage fluctuations. Using a buffer amplifier with the shortest leads possible to the cell to drive the recorder may be required to obtain usable results. Shielding the leads to the test cell will also help. If the curves are extremely noisy (erratic), make at least two curves on the same area to determine if the results are meaningful and consistent. It may be necessary to insert an electrical filter in the line source ahead of the constant current supply.

7.6 **Insertion Depth of Agitation Tube**—If the agitation tube contains the reference electrode, insert the tube in the cell to the same depth each time (see 6.6), but not so deep as to “shield” or interfere with the area being stripped.

7.7 **Incomplete Dissolution of the Nickel**—Even though an apparent end point is observed, the nickel may not be completely dissolved. There may be small islands of nickel left, or the periphery of the depleted area may be irregular or uneven. This may be associated with a tilt of the cell relative to the coating surface. Examine the test area with a magnifying lens after each test to insure that all the nickel has been dissolved from the test area. If nickel is present, rerun the test until the test area does not contain any nickel.

8. **Interpretation of Results**

8.1 The data obtained from this test will be shown on the recorded graph, which plots the thickness (stripping time) of nickel on the X-axis versus the millivolt (potential) of the nickel layers on the Y-axis. The thicknesses of the individual layers (or time differential) is measured between the steps or breaks in the curve along the x-axis while the electrode potential difference is determined by the change in amplitude of the curve on the Y-axis. It is desirable that at least two tests, within 6 to 8 mm of each other, be made on each test area and the results averaged.

8.2 **Interpretation of Curve**—Referring to Fig. 1, it can be seen that there are steps or breaks (changing potential) or steps in the curve when moving left to right (increasing thickness).

**Note 9**—The values given in Fig. 1 are included only to simplify the discussion for the Interpretation of Results. The actual values obtained during testing will depend on the nature of the equipment, experimental technique, the specific characteristics of the electroplating processes used to produce the multilayer coatings and other details. It is emphasized that the curve in Fig. 1 is an idealization of an actual result. Although the potential differences are reproducible, the values of the individual potentials may shift depending on the experimental results.

8.2.1 **Microdiscontinuous Nickel**—The first break or step, A to C, in the solid curve is small and occurs at a nickel thickness of approximately 3 µm in Fig. 1. The deposit represented by this curve, from 0 to 3 µm, is a nickel strike which might be used to induce microdiscontinuity in the chromium deposited over this strike, see Note 10. In Fig. 1, the difference in activity between this nickel strike deposit, B (750 mV), and the bright nickel deposit, C (730 mV), is 20 mV. This makes the deposit less active (cathodic) than the bright nickel deposit.

**Note 10**—This nickel deposit is referred to as microdiscontinuous

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**Fig. 1. Ideal STEP test curve for a four-layer nickel coating**

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**FIG. 1 T-Shaped Reference Electrode Assembly**
nickel when it contains inert particles (particle nickel) to produce micro-porous chromium or when it is microcracked (stressed nickel deposit) to produce microcracked chromium. When the nickel strike deposit is not used to produce discontinuities in the chromium, it can be referred to as "noble nickel" if its electrode potential (millivolt activity) is more noble (less active) than the adjacent bright nickel deposit.

8.2.2 Bright Nickel—After the first section of the curve ending at about 3 µm, the curve in Fig. 1 experiences another break or step at the 15 µm thickness mark in the figure, point D. This represents at least 12 µm of bright nickel (see 8.2.4) with a potential of 730 mV.

8.2.3 High Potential Nickel Strike —The line D to F in Fig. 1 between 15 µm and 18 µm represents the shape of the curve of a high potential nickel strike that in this case is 3 µm thick. For it to be classified as a high potential strike, it must have an electrode potential more active than the adjacent bright nickel deposit. In this example, the 705 mV (E) high potential nickel strike is 25 mV more active than the 730 mV bright nickel deposit (D).

8.2.4 Semi-bright Nickel—In Fig. 1, a large potential change occurs, F to H. This slope is the result of the cell transitioning from dissolving the last of the higher activity bright nickel and starting to dissolve the lower activity semi-bright nickel deposits. In Fig. 1, the semi-bright nickel has an electrode potential that is 145 mV less active (cathodic) than the bright nickel deposit. This is the STEP value for this multi-layered nickel coating. Determining the thickness of the bright and semi-bright nickel deposits, as in Fig. 1, normally involves making an estimate. Since the distance along the x-axis between points F and H is when the cell is transiting between the bright and the semi-bright nickel deposits, assigning half of the thickness to both deposits is usually an acceptable procedure. This thickness is usually very small compared to the thickness between C and D for the bright and H and I for the semi-bright nickel. Alternative procedures are presented in 8.2.6 and 8.4.

**NOTE 11**—The step in the curve is never a straight perpendicular line. As the bright nickel is dissolved, the plotted potential is associated only with the bright nickel until the semi-bright nickel is exposed and starts to dissolve. At this point, the measured potential increases and continues to increase until only semi-bright nickel is exposed except for bright nickel on the walls of the pit formed by dissolution of the nickel. From this point on the recorded potential is due primarily to the semi-bright nickel.

**NOTE 12**—Most corrosion studies have demonstrated that for the best corrosion results, if a nickel strike is used between the chromium deposit and the bright nickel, it should have an electrochemical electrode potential equal to, or preferably more noble (less active) than the bright nickel deposit. The semi-bright nickel deposit should also be more noble (less active) than the bright nickel deposit (see Specification B 456).

8.2.5 Substrate—The line after at about 40 µm in Fig. 1 represents the substrate when a steel substrate is under the semi-bright nickel deposit. The curve would turn up if a copper substrate was used.

8.2.6 Thickness and STEP Measurements—Since the rise of the actual STEP curve is normally not a nearly straight line as depicted in Fig. 1, it is best to read the thickness at the midpoint on the rising portion of the curve (see Fig. 2 and 8.4) if it is difficult to assign a millivolt reading due to the shape of the curve, see 8.4 and Notes 13 and 14.

8.3 When a STEP value is referenced, such as in a standard, it refers to the electrode potential in millivolts between the bright nickel and the semi-bright nickel deposits. In Fig. 1, the STEP is 145 mV. When additional STEP values are presented, they must include the deposit being referenced and its relative activity to the deposit in which it is paired. In Fig. 1, the STEP for the high potential nickel strike is 25 mV more active than the bright nickel deposit.

8.4 In some instances, the obtained curves may show irregularities such as drift (deviation from a straight line) in the plot for the semi-bright nickel, the bright nickel deposit, or both. The drift may be more noticeable with thick multilayer deposits. The curve also may exhibit a greater than normal drift or elongation of the step (rising portion of the plot). In order to

**FIG. 2 Midpoint Nickel Thickness Curve**
minimize error in interpreting this type of curve, the millivolt reading for the bright nickel is taken 2 µm before the rise of the curve, point A on Fig. 2 (the point at which the semi-bright nickel is first exposed in the cell), and the mV reading for the semi-bright nickel is taken 2 µm after the rise of the curve, point B on Fig. 2 (the point at which the potential reading is due predominantly to the exposure of only the semi-bright nickel). Where the multilayer nickel contains a thin layer of a high potential deposit, the potential of the bright nickel will be taken at a point 2 µm before the dip in the curve attributed to the high potential layer. Use of an indicating (digital display) meter is helpful if the chart is periodically annotated (every 15 to 30 s) with the display reading.

**NOTE 13**—Due to the much thinner “microdiscontinuous nickel” and “high potential nickel” strikes, compared to the bright and semi-bright nickel deposits, an estimate of their potentials and thickness is sometimes all that is obtainable.

**NOTE 14**—Another method is to determine the point of inflection of the curve, which is the point of maximum slope. If it is not evident to the eye, the use of a straight edge can be helpful. The straight edge is lined up with the step portion of the curve as shown in Fig. 2. The segment of the plot with the maximum slope should be of finite length, line C-D in Fig. 2, and the midpoint of the segment can be taken.

9. **Precision and Bias**

9.1 In the case of multilayer nickel coatings, the measurement uncertainty or precision of the method has been studied on electroplated panels. Results based on measurements made in three laboratories showed that the thickness of semi-bright and bright nickel layers can be determined with a standard deviation of ±1.65 µm with a variance of 9.2 %. The potential difference between semi-bright and bright nickel can be measured with a standard deviation of ±3.7 mV with a variance of 2.6 %.

**NOTE 15**—The relatively high variance in the thickness measurements, 9.2 %, was due to actual variation in coating thickness at various points on the panel. This was confirmed by measuring thickness by the microscopical method at points close or adjacent to the spots used in the STEP tests. The results were in close agreement, that is, the variation in local thickness displayed in the STEP test measurements was also observed in the microscopical measurements. Also, the electrode potential results from tests made at different current-density locations on a single sample can vary greatly due to the different characteristics of nickel electroplated at different current densities.

9.2 Measurements made on primary standard reference materials\(^6\) that certify the thickness, thickness uniformity, and the potential difference between the semi-bright and bright nickel layer showed that thickness could be measured with a standard deviation of ±0.5 µm with a variance of 2.1 %. The potential difference was measured with a standard deviation of ±0.82 mV with a variance of 0.7 %.

9.3 Determination of the bias or accuracy of the method has not been studied in detail. Note 12 suggests, however, that the STEP test provides as accurate a determination of the local nickel thickness as does the microscopical method.

10. **Keywords**

10.1 potential; STEP; thickness

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\(^6\) Primary Standard Reference Material (SRM) Number 2350 for calibrating this method is available from the National Bureau of Standards, Office of Standard Reference Materials, Gaithersburg, MD 20899.
1. Scope

1.1 This guide describes some of the available standard methods for the detection, identification, and measurement of porosity and gross defects in electrodeposited and related metallic coatings and provides some laboratory-type evaluations and acceptances. Some applications of the test methods are tabulated in Table 1 and Table 2.

1.2 This guide does not apply to coatings that are produced by thermal spraying, ion bombardment, sputtering, and other similar techniques where the coatings are applied in the form of discrete particles impacting on the substrate.

1.3 This guide does not apply to beneficial or controlled porosity, such as that present in microdiscontinuous chromium coatings.

1.4 Porosity test results (including those for gross defects) occur as chemical reaction end products. Some occur in situ, others on paper, or in a gel coating. Observations are made that are consistent with the test method, the items being tested, and the requirements of the purchaser. These may be visual inspection (unaided eye) or by 10× magnification (microscope). Other methods may involve enlarged photographs or photomicrographs.

1.5 The test methods are only summarized. The individual standards must be referred to for the instructions on how to perform the tests.

1.6 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.7 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
- B 246 Specification for Tinned Hard-Drawn and Medium-Hard-Drawn Copper Wire for Electrical Purposes
- B 276 Test Method for Apparent Porosity in Cemented Carbides
- B 374 Terminology Relating to Electroplating
- B 537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure
- B 542 Terminology Relating to Electrical Contacts and Their Use
- B 545 Specification for Electrodeposited Coatings of Tin
- B 560 Specification for Electrodeposited Coatings of Tin-Nickel Alloy
- B 605 Specification for Electrodeposited Coatings of Tin
- B 650 Specification for Electrodeposited Engineering Chromium Coatings on Ferrous Substrates
- B 689 Specification for Electroplated Engineering Nickel Coatings
- B 733 Specification for Autocatalytic (Electroless) Nickel-Phosphorous Coatings on Metal
- B 734 Specification for Electrodeposited Copper for Engineering Uses
- B 735 Test Method for Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor
- B 741 Test Method for Porosity in Gold Coatings on Metal Substrates by Paper Electrography
- B 798 Test Method for Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography
- B 799 Test Method for Porosity in Gold and Palladium Coatings by Sulfurous Acid/Sulfur-Dioxide Vapor
- B 809 Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor (Flowers-of-Sulfur)
- B 866 Test Method for Gross Defects and Mechanical Damage in Metallic Coatings by Polysulfide Immersion
- B 877 Test Method for Gross Defects and Mechanical Damage in Metallic Coatings by the Phosphomolybdic Acid (PMA) Method

3. Terminology

3.1 Definitions—Many terms used in this guide are defined in Terminology B 374 or B 542.

3.2 Definitions of Terms Specific to This Standard:
4. Significance and Use

4.1 Porosity tests indicate the completeness of protection or coverage offered by the coating. When a given coating is known to be protective when properly deposited, the porosity serves as a measure of the control of the process. The effects of substrate finish and preparation, plating bath, coating process, and handling, may all affect the degree of imperfection that is measured.

Note 1—The substrate exposed by the pores may be the basis metal, an underplate, or both.

4.2 The tests in this guide involve corrosion reactions in which the products delineate pores in coatings. Since the chemistry and properties of these products may not resemble those found in service environments, these tests are not recommended for prediction of product performance unless correlation is first established with service experience.

5. Applications

5.1 From the viewpoint of both porosity testing and functional significance, it is useful to divide porosity into two broad categories, namely intrinsic porosity and gross defects.

5.1.1 Intrinsic or normal porosity is due primarily to small deviations from ideal plating and surface preparation conditions. As such, it will be present to some degree in all commercial thin platings and will generally follow an inverse relationship with thickness. In addition, scanning electron microscope (SEM) studies have shown that the diameter of such pores at the plating surface is of the order of micrometers, so that only small areas of underlying metal are exposed to the environment.

5.1.2 Gross defects, on the other hand, would result in comparatively large areas of exposed basis metal or underplating. Examples of such defects are mechanical damage to the coating through mishandling or wear. Gross defects can also be found in undamaged coatings in the form of networks of microcracks and as large as-plated pores— with diameters an order of magnitude (or more) greater than intrinsic porosity. Such gross defects indicate such serious deviations from acceptable coating practice as dirty substrates and contaminated or out-of-balance baths.

5.2 Intrinsic porosity and most types of gross defects are too small to be seen except at magnifications so high that a realistic assessment of the overall coating surface in the functional areas of the part cannot be made. Instead, the presence and severity of the porosity is normally determined by some type of pore-corrosion test that will magnify the pore sites by producing visible reaction products in and around the pores or cracks. Tests for gross defects (Section 7), and especially for mechanical damage and wear, are designed to be less severe. Such tests, however, may not detect a sizeable portion of the smaller (intrinsic) pores in a coating. On the other hand, standard tests for intrinsic porosity (Section 6) will easily reveal the presence of gross defects as well.

5.3 Porosity tests are generally destructive in nature and are designed to assess the quality of the coating process in conjunction with the substrate. Therefore, separate test specimens are not ordinarily allowed.

5.4 In the tests summarized in this guide, chemicals react with the exposed substrate through the pore or channel to form a product that is either directly observable or that is made observable by subsequent chemical development.

5.5 Porosity tests differ from corrosion and aging tests. A good porosity test process must clean, depolarize, and activate the substrate metal exposed by the pore, and attack it sufficiently to cause reaction products to fill the pore to the surface of the coating. The corrosive reagent ideally does not react with the coating. Reaction time is limited, particularly with thin

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3.2.1 porosity—for the purpose of this guide, porosity in a coating is defined as any hole, crack, or other defect that exposes the underlying metal to the environment. Differences between the major types of porosity are described in Section 5.

3.2.2 underplate—a metallic coating layer between the basis metal and the topmost metallic coating. The thickness of an underplating is usually greater than 1 µm, in contrast to a strike or flash, which are usually thinner.

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TABLE 1 Applications of Standard Porosity Tests to Metallic Coatings (Section 6)

<table>
<thead>
<tr>
<th>Substrate Metal</th>
<th>Gold</th>
<th>Silver</th>
<th>Nickel</th>
<th>Tin-Nickel</th>
<th>Tin</th>
<th>Tin-Lead</th>
<th>Copper</th>
<th>Palladium</th>
<th>Chromium</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper and Copper</td>
<td>6.1&lt;sup&gt;b&lt;/sup&gt;</td>
<td>6.2&lt;sup&gt;b&lt;/sup&gt;</td>
<td>6.3A</td>
<td>6.4</td>
<td>6.4</td>
<td>6.4</td>
<td>6.4</td>
<td>...</td>
<td>6.2, 6.3A, 6.4,</td>
</tr>
<tr>
<td>Alloys</td>
<td>6.4, 6.5</td>
<td>6.4</td>
<td>6.4</td>
<td>6.4</td>
<td>6.4</td>
<td>6.4</td>
<td>6.4</td>
<td>...</td>
<td>6.2, 6.3A, 6.5</td>
</tr>
<tr>
<td>Nickel</td>
<td>6.1&lt;sup&gt;b&lt;/sup&gt;, 6.2, 6.5</td>
<td>6.3A</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td>6.2, 6.3A, 6.5</td>
<td>...</td>
</tr>
<tr>
<td>Iron or Steel</td>
<td>6.6</td>
<td>...</td>
<td>6.6</td>
<td>6.3B, 6.6</td>
<td>6.3B, 6.6</td>
<td>6.3B, 6.6</td>
<td>6.6</td>
<td>6.6</td>
<td>...</td>
</tr>
<tr>
<td>Silver</td>
<td>6.4</td>
<td>...</td>
<td>6.4</td>
<td>6.4</td>
<td>6.4</td>
<td>6.4</td>
<td>6.4</td>
<td>...</td>
<td>6.4</td>
</tr>
</tbody>
</table>

<sup>a</sup> The substrate may be the basis metal, an underplate, or both (see Note 1).

<sup>b</sup> Thickness restrictions may apply.

TABLE 2 Applications of Tests for Gross Defects and Mechanical Damage (Section 7)

<table>
<thead>
<tr>
<th>Substrate Metal&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Gold</th>
<th>Nickel</th>
<th>Tin-Nickel</th>
<th>Tin</th>
<th>Tin-Lead</th>
<th>Palladium</th>
<th>Silver</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper and Copper Alloys</td>
<td>7.3, 7.5</td>
<td>7.3, 7.4</td>
<td>7.3</td>
<td>7.3</td>
<td>7.3</td>
<td>7.3</td>
<td>7.5</td>
</tr>
<tr>
<td>Nickel</td>
<td>7.5</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td>7.5</td>
</tr>
<tr>
<td>Iron or Steel</td>
<td>7.1</td>
<td>...</td>
<td>7.1</td>
<td>7.1</td>
<td>7.1</td>
<td>7.1</td>
<td>7.1</td>
</tr>
<tr>
<td>Aluminum</td>
<td>...</td>
<td>7.2</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td>...</td>
</tr>
</tbody>
</table>

<sup>a</sup> The substrate may be the basis metal, an underplate, or both (see Note 1).
coatings, since the corrosive will attack the substrate in all directions and, in so doing, undermine the coatings so that false observations may be made. When the corrosion product is soluble in the reagent, a precipitating indicator is used to form the reaction product.

5.6 The substrate exposed by the pores may be the basis metal, an underplate, or both.

6. Outlines of Standard Porosity Tests for Intrinsic Porosity (5.1.1)

NOTE 2—The test methods outlined in this section are only summaries. The specified test standard must be referred to for the instructions on how to perform the tests, as well as for important applications and limitations.

6.1 Nitric Acid Vapor Test (Test Method B 735):

6.1.1 Scope—Gold coatings on nickel, copper, and their alloys, where the gold is at least 0.6 μm (24 μin.) thick.

6.1.2 Summary of Test Method—Test specimens are suspended over concentrated nitric acid in a closed nonreactive vessel for a specified time, usually 0.5 to 2.0 h. After exposure, the specimens are dried in an oven to fix the reaction products. Each reaction product spot indicates a pore in the coating.

6.2 Paper Electrography (Test Method B 741):

6.2.1 Scope—Gold and palladium coatings on nickel, copper, or copper alloys that have flat or nearly flat surfaces, or, with appropriate fixtures, gently curved surfaces.

6.2.2 Summary of Test Method—Electrolyte-soaked paper is pressed against the specimen at controlled pressure. Current, at a constant controlled dc voltage, is passed from an inert anode through the paper, to the specimen (which is made the cathode) for a specified time. Base-metal ions at the pore sites migrate to the paper where an indicator reagent converts them to colored products (the pore indications).

6.3 Sulfur Dioxide Tests:

6.3.1 Scope:

6.3.1.1 Variation A (Test Method B 799, Sulfurous Acid/Sulfur-Dioxide Vapor) applies to gold, palladium, and silver over nickel, copper, and their alloys.

6.3.1.2 Variation B applies to tin and its alloys over iron or steel (Appendixes of Specification B 545 and Specification B 605).

6.3.2 Summary of Test Method—The test specimens are suspended over sulfur-oxide acidic solutions in a sealed chamber. For Variation A (Test Method B 799), the solution is concentrated sulfurous acid. For Variation B it is a 1:4 mixture of 0.1N sulfuric acid and 0.12N sodium thiosulfate solutions. Each reaction product spot on the surface indicates a pore in the coating.

6.4 Humid Sulfur Vapor (“Flowers-of-Sulfur”) (Test Method B 809):

6.4.1 Scope—Primarily for coatings over silver, copper, or copper alloys. Coatings may include nickel, gold, palladium, tin, and any other coating that does not significantly tarnish in reduced sulfur atmospheres.

6.4.2 Summary of Test Method—Test specimens are suspended in a vented closed container over powdered sulfur at controlled humidity and temperature. Black or brown spots indicate porosity.

6.5 Gel Electrography (Test Method B 798):

6.5.1 Scope—Gold and palladium coatings over nickel, copper, or copper alloys.

6.5.2 The test sample is made the anode in a cell of a specific geometry, which contains a solid electrolyte consisting of gelatin, conducting salts and an indicator. Application of a constant dc current causes migration of base-metal ions through the pores to the metallic coating surface where reaction with the indicator produces colored reaction products.


6.6.1 Scope—Metallic coatings included are those that are resistant to ferricyanide and chloride, but are cathodic to iron, steel, or iron-based alloy substrates. Examples of such coatings are gold, tin, nickel, copper, chromium, and their alloys.

6.6.2 Summary of Test Method—Electrolyte-wetted, gel-chloride treated paper strips are placed firmly in contact with test specimen surfaces for a specified time, not to exceed 10 min. After the allotted time, the paper strips are wetted with a ferricyanide indicator solution. Blue spots indicate pores.

6.6.3 Alternate methods involve formation of the blue spots directly on the specimen (Specification B 733, Test Methods, Ferroxyl Test for Iron Base Substrates).

7. Outlines of Porosity Tests for Gross Defects and Mechanical Damage (5.1.2)

NOTE 3—Some of the test methods outlined in this section have been taken from ASTM specifications for specific metallic coatings. As such they are only summaries. ASTM Subcommittee B08.10 is developing complete test method documents for some of those procedures.

7.1 Hot Water Test (Annex of Specification B 689):

7.1.1 Scope—Metallic coatings cathodic to a ferrous substrate; for example, nickel, tin, or gold on steel or iron-based alloy substrates.

7.1.2 Summary of Test Method—The test specimens are immersed for a specified time in neutral, distilled heated (85°C) water that is agitated with clean air. After exposure and drying, black spots and red rust indicate porosity.

7.1.3 Alternative methods involve aerated water at room temperature with longer exposure times.

7.2 Alizarin Test (Specification B 733, Test Methods, “Porosity”):

7.2.1 Scope—Primarily for nickel on aluminum substrate.

7.2.2 Summary of Test Method—The surface of the specimen is swabbed with a sodium hydroxide solution. After rinsing, an alizarin sulfonate solution is applied in two steps followed by glacial acetic acid to remove the background violet color. Any red spots remaining indicate pores.

7.3 Sodium Polysulfide Immersion (Test Method B 866) is an example:

7.3.1 Scope—For detecting gross discontinuities, including wear and mechanical damage, in coatings over copper or its alloys. Applicable coatings include tin, nickel, gold, palladium, or any other coating that does not tarnish in the polysulfide solution.

7.3.2 Summary of Test Method—The test samples are immersed in an alkaline polysulfide solution for 60 s. After rinsing and drying, samples are examined for dark or discolored areas.
7.4 Ferrocyanide Test (Specification B 733, Test Methods, “Porosity”):

7.4.1 Scope—For gross defects in nickel coatings over copper substrates.

7.4.2 Summary of Test Method—Wipe the coated specimen with glacial acetic acid. After 3 min, apply a solution of potassium ferrocyanide and methyl cellulose in boiling distilled water. The appearance of brown spots after 2 min indicates pores.

7.5 Phosphomolybdic Acid (PMA) (Test Method B 877):

7.5.1 Scope—Individual specimens of gold, silver, or palladium, over nickel, copper, or their alloys.

7.5.2 Summary of Test Method—The part is exposed briefly to hydrochloric acid fumes. Then a small drop of aqueous PMA solution is applied to the test area. The presence of any exposed substrate metal is revealed by the formation of an intensely colored molybdenum blue complex.

8. Report

8.1 The results of a porosity test are usually given in terms of one of the following:

8.1.1 Option 1—The number and size of the pore indications in the measurement area. This is converted to a pore density in terms of number of pores per square centimetre. The pore indications are often classified according to size (see the individual test standard).

8.1.2 Option 2—The percentage of the significant surface covered by the pore indications.

8.1.3 Option 3—The area, in square millimetres, of the largest spot on the significant surface.

9. Evaluation

9.1 Procedure—In Option 1, count individual pores at 10× magnification in the significant area of the coating, as defined by the specification or drawing of the product. In Option 2, compare results with panels shown in Test Method B 276 and Practice B 537, or those supplied as criteria by the purchaser. In Option 3, scan for the largest defect(s).

9.2 Criteria (Pass-Fail)—Pass-fail criteria, such as pore count greater than 50/cm² (Option 1), or an area greater than 1 % (Option 2), or a spot or crack with a total area greater than 2.5 mm² (Option 3), should be chosen in conformance to the intended function of the coating and the intended use of the object coated. Such criteria should be agreed upon between supplier and purchaser.

10. Keywords

10.1 electrodeposits; gross defect; mechanical damage; metallic coatings; porosity; porosity testing; wear-through
Standard Specification for Electrodeposited Coatings of Cadmium

1. Scope

1.1 This specification covers the requirements for electrodeposited cadmium coatings on products of iron, steel, and other metals.

Note 1—Cadmium is deposited as a coating principally on iron and steel products. It can also be electrodeposited on aluminum, brass, beryllium copper, copper, nickel, and powder metallurgy parts.

1.2 The coating is provided in various thicknesses up to and including 25 µm either as electrodeposited or with supplementary finishes.

1.3 Cadmium coatings are used for corrosion resistance and for corrosion prevention of the basis metal part. The as-deposited coating (Type I) is useful for the lowest cost protection in a mild or noncorrosive environment where early formation of white corrosion products is not detrimental or harmful to the function of a component. The prime purpose of the supplementary chromate finishes (Types II and III) on the electroplated cadmium is to increase corrosion resistance. Chromating will retard or prevent the formation of white corrosion products on surfaces exposed to various environmental conditions as well as delay the appearance of corrosion from the basis metal.

1.4 Cadmium plating is used to minimize bi-metallic corrosion between high-strength steel fasteners and aluminum in the aerospace industry. Undercutting of threads on fastener parts is not necessary as the cadmium coating has a low coefficient of friction that reduces the tightening torque required and allows repetitive dismantling.

1.5 Cadmium-coated parts can easily be soldered without the use of corrosive fluxes. Cadmium-coated steel parts have a lower electrical contact resistance than zinc-coated steel. The lubricity of cadmium plating is used on springs for doors and latches and for weaving machinery operating in high humidity. Corrosion products formed on cadmium are tightly adherent. Unlike zinc, cadmium does not build up voluminous corrosion products on the surface. This allows for proper functioning during corrosive exposure of moving parts, threaded assemblies, valves, and delicate mechanisms without jamming with debris.

2. Referenced Documents

2.1 The following standards form a part of this document to the extent referenced herein.

2.2 ASTM Standards:

A 165 Specification for Electrodeposited Coatings of Cadmium on Steel

B 117 Practice for Operating Salt Spray (Fog) Apparatus

B 183 Practice for Preparation of Low-Carbon Steel for Electroplating

B 201 Practice for Testing Chromate Coatings on Zinc and Cadmium Surfaces

B 242 Practice for Preparation of High-Carbon Steel for Electroplating

B 253 Guide for Preparation of Aluminum Alloys for Electroplating

B 254 Practice for Preparation of and Electroplating on Stainless Steel

B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings

B 320 Practice for Preparation of Iron Castings for Electroplating

B 322 Practice for Cleaning Metals Prior to Electroplating

B 343 Practice for Preparation of Nickel for Electroplating with Nickel

B 374 Terminology Relating to Electroplating

B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section


2 Discontinued; see 1987 Annual Book of ASTM Standards, Vol 02.05. Replaced by Specification B 766.

3 Annual Book of ASTM Standards, Vol 03.02.

4 Annual Book of ASTM Standards, Vol 02.05.
3. Terminology

3.1 Definitions—Definitions of terms used in this specification are in accordance with Terminology B 374.

4. Classification

4.1 Classes—Electrodeposited cadmium coatings shall be classified on the basis of thickness as follows:

<table>
<thead>
<tr>
<th>Class</th>
<th>Minimum Thickness, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>25</td>
</tr>
<tr>
<td>12</td>
<td>12</td>
</tr>
<tr>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td>5</td>
<td>5</td>
</tr>
</tbody>
</table>

Note 2—Cadmium coatings thicker than 12 µm are normally not economical.

4.2 Types—Electrodeposited cadmium coatings shall be identified by types on the basis of supplementary treatment required as follows:

4.2.1 Type I—As electrodeposited without supplementary treatment.

4.2.2 Type II—With supplementary colored chromate treatment.

4.2.3 Type III—With supplementary colorless chromate treatment.

5. Ordering Information

5.1 In order to make the application of this specification complete, the purchaser needs to supply the following information to the seller in the purchase order or other governing document:

5.1.1 The name, designation, and date of issue of this specification.

5.1.2 Deposit by class and type (4.1 and 4.2).

5.1.3 Composition and metallurgical condition of the substrate to be coated. Application to high-strength steel parts (6.2).

5.1.4 Heat treatment for stress relief, whether it has been performed or is required (6.3).

5.1.5 Additional undercoat, if required (6.5).

5.1.6 Plating process variation, if required (6.6).

5.1.7 Hydrogen embrittlement relief, if required (6.7).

5.1.8 Desired color of the Type II film (6.8.2).

5.1.9 Location of significant surfaces (7.1.2).

5.1.10 Coating luster (7.5).

5.1.11 Whether non-destructive or destructive tests are to be used in cases of choice (Note 14).

5.1.12 Configuration, procedures, and tensile load for hydrogen embrittlement relief test (9.4, 10.6, Supplementary Requirements S2, and S3).

5.1.13 Whether certification is required (Section 12).

5.1.14 Whether supplementary requirements are applicable.

6. Materials and Manufacture

6.1 Nature of Coating—The coating shall be essentially pure cadmium produced by electrodeposition usually from an alkaline cyanide solution.

6.2 High Tensile Strength Steel Parts—Steel parts having an ultimate tensile strength greater than 1650 MPa (approximately 50 HRC) shall not be plated by electrodeposition unless authorized by the purchaser.

6.3 Stress Relief—Steel parts having an ultimate tensile strength of 1050 MPa (approximately 35 HRC) and above, and that have been machined, ground, cold-formed, or cold-straightened shall be heat-treated at 190 ± 15°C for 5 h or more for stress relief before cleaning and coating.

6.4 Preparatory Procedures—The basis metal shall be subjected to such cleaning procedures as necessary to ensure a surface satisfactory for subsequent electroplating. Materials used for cleaning shall have no damaging effects on the basis metal resulting in pits, intergranular attack, stress corrosion cracking, or hydrogen embrittlement. If necessary, cleaning materials for steel parts should be evaluated in accordance with Method F 519.

Note 4—For basis metal preparation, the following standards should be employed depending upon the metallurgical composition: Practices B 183, B 242, B 253, B 254, B 281, B 320, B 322, B 343 and B 558.

6.5 Substrate—Cadmium shall be deposited directly on the basis metal part without an undercoat of another metal except when the part is either stainless steel or aluminum and its alloys. An undercoat of nickel is permissible on stainless steel. With aluminum and aluminum alloys, the oxide layer shall be
removed and replaced by a metallic zinc layer in accordance with Guide B 253. For better adherence, a copper strike or a nickel coating may be applied to the zinc layer before electroplating with the cadmium.

6.6 Plating Process—The plating shall be applied after all basis metal heat treatments and mechanical operations, such as machining, brazing, welding, forming, and perforating of the article, have been completed.

6.7 Hydrogen Embrittlement Relief—Steel parts having a tensile strength of 1200 MPa (approximately 38 HRC) and higher shall be baked at 190 ± 15°C for 8 h or more within 4 h after electroplating to provide hydrogen embrittlement relief. Electroplated springs and other parts subject to flexure shall not be flexed, loaded, or used before the hydrogen embrittlement relief treatment. The baking treatment for hydrogen embrittlement relief shall be done before the application of any supplementary chromate treatment. When specified, freedom from embrittlement shall be determined.

NOTE 5—For high-strength steels, greater than 1300 MPa or approximately 40 HRC, it is strongly recommended that the baking time be extended to 23 h or more to ensure hydrogen embrittlement relief.

NOTE 6—Electroplated steel parts, passivated by the baking operation for hydrogen embrittlement relief, require reactivation before the chromate treatment. This application, immersion in a dilute acid solution, should be done as soon as practical. If the chromating solution contains sulfuric acid, then the reactivating solution should be 1 part of sulfuric acid (sp gr 1.83) by volume added to 99 parts of water. If the chromating solution contains hydrochloric acid, then the reactivating solution should be 1 part of hydrochloric acid (sp gr 1.16) by volume added to 99 parts of water. Duration of immersion should be as brief as is consistent with the nature of the work. Separately racked items can be reactivated in approximately 5 s, whereas a perforated container of barrel-plated parts requires approximately 15 s.

6.8 Chromate Treatment:

6.8.1 Chromate treatments for Types II and III shall be done in or with special aqueous acidic solutions composed of hexavalent chromium along with certain anions that act as catalyst or film-forming compounds to produce a continuous smooth protective film. Chromic acid and nitric acid bright dips shall not be used for treatment to produce chromate coatings. When proprietary materials are used for this treatment, the instructions of the supplier should be followed.

6.8.2 The Type II film color shall range from an iridescent yellow or a thicker, more protective iridescent bronze or brown to the heavier olive drab. It may also be dyed to a desired color. When necessary, the color of the film shall be indicated by the purchaser and specified by the provision of a suitably colored sample or indicated on the drawing for the part.

6.8.3 The absence of color shall not be considered as evidence of lack of Type III film or as a basis for rejection. Presence of clear Type III film shall be determined by a spot test as specified in 10.4.

6.8.4 Waxes, lacquers, or other organic coatings shall not be used as a substitute for, nor may they be used in conjunction with, supplementary treatments when the purpose is to ensure conformance to the salt spray requirements. Waxes and the like, may be used to improve lubricity.

7. Coating Requirements

7.1 Thickness:

7.1.1 The thickness of the coating everywhere on the significant surfaces shall conform to the requirements of the specified class, as defined in 4.1.

7.1.2 Significant surfaces are those normally visible (directly or by reflection) that are essential to the appearance or serviceability of the article when assembled in normal position; or that can be the source of corrosion products that will deface visible surfaces on the assembled article. When necessary, the significant surfaces shall be indicated by the purchaser on applicable drawing of the article, or by the provision of suitably marked samples.

NOTE 7—As heavier coatings are required for satisfactory corrosion resistance than Class 5, allowance should be made in the fabrication of most threaded articles, such as nuts, bolts, and similar fasteners with complementary threads for dimensional tolerances to obtain necessary coating build-up. Flat surfaces and certain shielded or recessed areas, such as root-diameter of threads, have a tendency to exhibit lack of build-up and to be heavier at exposed edges and sharp projections with electrode-deposited coatings. This trend is also found with vacuum-deposited cadmium coatings and is in direct contrast with mechanically deposited coatings.

NOTE 8—The coating thickness requirements of this specification is a minimum requirement. Variation in thickness from point to point on an article is inherent in electroplating. Therefore, the thickness will have to exceed the specified value at some points on the significant surfaces to ensure that it equals or exceeds the specified value at all points. Hence, in most cases, the average coating thickness of an article will be greater than the specified value; how much greater is largely determined by the shape of the article (see Practice B 507) and the characteristics of the electroplated process. In addition, the average coating thickness on articles will vary from article to article within a production lot. Therefore, if all of the articles in a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the average necessary to assure that a single article meets the requirement.

7.1.3 For nonsignificant visible surfaces, the minimum thickness for Classes 25 and 12 shall be Class 8 (8 µm); for Class 8 it shall be Class 5 (5 µm); and for Class 5 it shall be 4 µm.

7.2 Adhesion—The cadmium coating shall be sufficiently adherent to the basis metal to pass the tests detailed in 10.2.

7.3 Abrasion Resistant—The supplementary Type II chromate film shall be adherent, nonpowdery, and abrasion resistant (10.3).

7.4 Corrosion Resistance—Cadmium coatings with supplementary chromate films on iron and steel basis metals shall show neither white corrosion products of cadmium nor basis metal corrosion products at the end of 96 h for Type II film or 12 h for Type III film when tested by continuous exposure to salt spray. The appearance of corrosion products visible to the unaided eye at normal reading distance shall be cause for rejection, except white corrosion products at the edges of specimens shall not constitute failure.

NOTE 9—The hours given are the minimum required to guarantee satisfactory performance. Longer periods before the appearance of white corrosion and basis metal corrosion (rust from iron and steel products) are possible. Salt spray resistance does not vary in exact proportion with increased plating thicknesses of Types II and III coatings. Although specified hours to failure (red rust for iron and steel articles) for Type I coating is not stated, the hours given for Types II and III reflect the added protection of the chromate treatment without requiring impractical testing.
8. Sampling

8.1 Inspection Lot—An inspection lot shall be a collection of coated articles that are of the same kind, that have been produced to the same specifications, that have been coated by a single supplier at one time or at approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

8.2 Selection—A random sample of the size required by Test Method B 602 shall be selected from the inspection lot. The articles in the lot shall be inspected for conformance to the requirements of this specification and the lot shall be classified as conforming to or nonconforming to each requirement in accordance with the criteria of the sampling plan in Test Method B 602.

NOTE 13—Test Method B 602 contains four sampling plans, three of which are to be used with nondestructive test methods, the fourth is to be used with destructive test methods. The three methods for nondestructive tests differ in the quality level they require of the product. Test Method B 602 requires use of the plan with the intermediate quality level unless the purchaser specifies otherwise. It is recommended that the purchaser compare the plans with his needs and state which plan is to be used. If the plans in Test Method B 602 do not serve the needs, additional plans are given in Guide B 697. Both Method B 602 and Guide B 697 list references where additional information on sampling inspection and additional plans are given.

9. Specimen Preparation

9.1 Electroplated Parts or Separate Specimens—When the electroplated parts are of such form, shape, size, and value as to prohibit use, or are not readily adaptable to a test, or when destructive tests of small lot size are required, the test shall be made by the use of separate specimens electroplated concurrently with the articles represented. The permission or the requirement to use test specimens, their number, the material from which they shall be made, and their shape and size shall be stated in the purchase order or other governing document. The separate specimens shall be of a basis metal equivalent to that of article represented, of the same metallurgical condition, and shall have the same surface condition. These separate specimens shall be introduced into the lot at regular intervals before the cleaning operation, preliminary to electroplating, and shall not be separated therefrom until after completion of the electroplating. Conditions affecting the electroplating of specimens, including the spacing, plating media, bath agitation, temperature, etc., in respect to the other objects being electroplated, shall correspond as nearly as possible to those affecting the significant surfaces of the articles represented. Unless a need can be demonstrated, separately prepared specimens shall not be used in place of production items for nondestructive and visual examinations.

9.2 Thickness, Adhesion, Abrasion Resistance and Presence of Clear (Type III) Coating Specimens—If separate specimens for thickness, adhesion, abrasion resistance, and the presence of clear (Type III) coating tests are required, they shall be strips approximately 100 mm long, 25 mm wide, and 1 mm thick, or cylindrical pieces with the cross-section area approximately equal to that of the production item.

9.3 Corrosion Resistance Specimens—If separate specimens for the corrosion resistance test are required, they shall be panels, approximately 150 mm long, 100 mm wide, and 1 mm thick.
9.4 Hydrogen Embrittlement Relief Specimens—If separate specimens are required for the hydrogen embrittlement relief test, the configuration shall be specified by the purchaser.

NOTE 15—When required for testing, the manufacturer of the basis metal parts should provide the coating facility with the stated test specimens, loading bolts, loading rings, and the like.

10. Test Methods

10.1 Thickness:
10.1.1 The thickness of electrodeposited cadmium coatings shall be determined by Test Methods B 487, B 499, B 504, B 567, or B 568, as applicable. Other methods may be used, if it can be demonstrated that the uncertainty of measurements with these methods is less than 10%.

10.1.2 The thickness measurements of Types II and III deposits shall be made after application of the supplementary treatments. Whenever Test Method B 504 or B 567 is used, remove the supplementary treatment from the test area before measuring the thickness. Remove by using a mild abrasive (a paste of levigated alumina or magnesium oxide) and rubbing gently with the finger.

10.2 Adhesion—The electrodeposited cadmium coating shall be tested for adhesion to the basis metal or substrate layer in a manner that is consistent with the service requirements of the coated article. When examined at a magnification of approximately 4× diameters, the coating shall not show separation at the interface. The formation of cracks in the coating caused by rupture of the basis metal that does not result in flaking, peeling, or blistering of the coating shall not be considered as nonconformance. Use one of the following methods for determining adhesion.

10.2.1 The surface of the coated article shall be scrapped or sheared with a sharp edge, knife, or razor blade through the coating down to the basis metal and examined at 4× magnification for evidence of nonadhesion.

10.2.2 The part shall be plastically deformed, if possible, by clamping in a vise and bending the projecting portion back and forth until rupture occurs.

10.2.3 Any suitable test procedure, such as the burnishing test, the draw test, the peel test, or the scribe test in accordance with Test Methods B 571 shall be used.

NOTE 16—There is no single satisfactory test for evaluating the adhesion of electrodeposited coatings. Those given (10.2.1, 10.2.2, and 10.2.3) are widely used; however, other tests may prove more applicable in special cases.

10.3 Abrasion Resistance—The abrasion resistance of the supplementary chromate films (Types II and III) shall be determined in accordance with Practice B 201.

10.4 Clear (Colorless) Type III Coatings—The presence of a clear Type III coating shall be determined in accordance with Practice B 201.

10.5 Salt Spray Corrosion Resistance—The 5% neutral salt spray (fog) test as defined in Test Method B 117 shall be used when there is a steel or iron substrate. Parts with supplementary chromate film shall be aged at room temperature for at least 24 h before subjecting to the test.

10.6 Hydrogen Embrittlement—The test to indicate freedom from hydrogen embrittlement failure of coated parts or specimens need not be conducted unless the method is specified in the purchase order. The method description shall include specimen configuration, means of applying a load to the part, stress or load level, and duration of test. Parts shall not crack or fail by fracture when tested.

10.7 Visual Examination—The coating shall be examined for compliance with the requirements of luster, workmanship, and finish.

11. Rejection and Rehearing

11.1 Coatings that fail to conform to the requirements of this specification or authorized modifications may be rejected. They may be reconsidered for acceptance by rectifying inspection in accordance with Methods B 602 and B 697 when allowed by the purchaser. Rejection should be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of the test, the producer or supplier may make claims for a rehearing. Coatings that show imperfection during subsequent manufacturing operations may be rejected.

12. Certification

12.1 When specified in the purchase order or contract, the producer’s or supplier’s certification shall be furnished to the purchaser stating that samples representing each lot have been processed, tested, and inspected in accordance with this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

13. Packaging and Packing

13.1 Preservation, packaging and packing methods for electrodeposited cadmium parts or articles employed by a supplier shall be such as to preclude damaging during shipment and handling.

14. Keywords

14.1 cadmium; electrodeposited coatings; electrodeposited coatings; cadmium
S1. Responsibility for Inspection

S1.1 The producer or supplier shall be responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein unless disapproved by the purchaser. The purchaser retains the right to perform any of the inspections and tests set forth in this specification where such inspections and tests are deemed necessary to ensure that the supplies and services conform to the prescribed requirements.

S2. Specimens for Hydrogen Embrittlement Relief

S2.1 Four specimens shall be used to represent the lot. The specimens for the test shall be round notched specimens with the axis of the specimen (load direction) perpendicular to the short transverse grain flow direction. The configuration shall be in accordance with the figure pertaining to 12.7 6 0.025 mm (0.5 6 0.001 in.) round tension test specimens with 50.8 6 0.13 mm (2.0 6 0.05 in.) gage length and examples of small-size specimens proportional to the standard specimen in Method E 8. Specimens shall have a 60 6 1° V-notch located approximately at the center of the gage length. The cross section area at the root of the “V” shall be approximately equal to half the area of the full cross-section area of the specimen’s reduced section. The “V” shall have a 0.254 6 0.013 mm radius of curvature at the base of the notch.

S3. Embrittlement Relief Test

S3.1 The notched specimens shall be subjected to a sustained tensile load equal to 75 6 2 % of the ultimate notch tensile strength of the material. The articles, parts, or specimens shall be held under load for at least 200 h and then examined for cracks or fracture.

S3.2 Parts such as spring pins, lock rings, and the like, that are installed in holes or rods shall be similarly assembled using the applicable parts specifications or drawing tolerances which impose the maximum sustained tensile load on the plated part. The selected samples shall be subjected to a sustained tensile load equal to 115 % of the maximum design yield load for which the part was designed. Fastener hardware, where the maximum yield load is not known or given, shall be tested in accordance with MIL-STD 1312, Test 5. Parts that require special fixtures, extreme loads to comply with the above requirements, or where the maximum design yield load is not known, may be represented by separate notched specimens.

Note S00017—The manufacturer of the basis metal parts should provide the coating facility with notched tensile specimens when required for testing.

APPENDIXES

X1. Electrodeposited Cadmium Coating

X1.1 Use—Electrodeposited cadmium coatings, mainly on iron and steel products, are widely used in many industries. Electrodeposition of the cadmium on other metal products being used to a greater extent in both the aerospace and communication industries. The corrosion rate of cadmium plated parts is much lower than that of unplated parts in most atmospheres as well as in water. In addition, the electrochemical relationship between cadmium and a basis metal, for example, steel, is such that the corrosion or rusting is suppressed by galvanic action even where the cadmium coating may be damaged or worn through. The Type I, as-deposited coating, may be used to improve solderability, to lower electrical contact resistance, to provide surface conductivity on electronic equipment, and to prevent seizing of moving parts, bolts, nuts, and latches.

X1.2 Limitations—The Type I electrodeposited coatings should not be used on production items that in service will reach a temperature of 225°C or higher or will come in contact with other parts that reach these temperatures. Cadmium coated parts that may be subjected to heat from soldering, brazing, or welding operations should be so labeled and tagged to indicate being so coated because of the danger from poisonous cadmium oxide vapors during these operations.
X2. Supplementary Treatments

X2.1 Purpose—The clear chromate film (Type III) is generally used to prevent fingerprintings and corrosion of parts that may occur at room temperature during assembly and storage or will be exposed to a mild environmental condition. The purpose of the colored chromate supplementary finish (Type II) on cadmium coatings is to retard the weathering to form white corrosion products on the surfaces exposed to stagnant water, sea water, marine atmospheres, high humidity, or cyclic condensation and drying. Most of the iridescent yellow to live drab Type II chromate films will be satisfactory as an undercoat if production items are to receive an organic paint system application, as generally required by the aerospace industry.

X2.2 Limitations—Supplementary chromate treatments should not be applied to production items that will not be painted and will be continuously exposed to temperatures in excess of 65°C or intermittently exposed for short periods to a temperature of about 150°C. The protective value of the supplementary coating decreases under these conditions. Temperatures above 65°C will accelerate the deterioration of the chromate film especially in drier environments, such that the chromate coating can become so powdery that no protection is provided.

X2.3 Handling Precautions—Chromate treated articles that involve only dipping in chemical solutions normally require 24 h at 20 to 30°C to render them suitable for handling without damage to the finish coating while the latter is in the gelatinous form. It is important with such coatings that the supplement film is not damaged while wet in order to comply with the workmanship requirements.

X3. Service Conditions

X3.1 The service life of a cadmium coating, based upon atmospheric exposure tests, has supported the generalization that the protective value of the deposit is proportional to its thickness and to the environment to which it is exposed. Conditions of exposure and basis metals are so varied that it is not possible to predict the exact service life of a coating of a given thickness, with or without a supplementary finish. Table X3.1 provides recommended thicknesses with a supplementary finish on typical applications (mainly iron and steel articles) that are suitable under certain service conditions.

<table>
<thead>
<tr>
<th>Service Condition</th>
<th>Class</th>
<th>Treatment</th>
<th>Exposure</th>
<th>Typical Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Severe</td>
<td>25* or 12</td>
<td>Type II</td>
<td>To condensation, perspiration, infrequent wetting by rain, cleaners</td>
<td>Military hardware, electronic parts for tropical service, washing machinery</td>
</tr>
<tr>
<td>Moderate</td>
<td>8</td>
<td>Type II</td>
<td>Mostly to dry indoor atmospheres but subjected to occasional condensation, wear, or abrasion</td>
<td>Threaded parts, screws, bolts, radio parts instruments, TV, and radio chassis</td>
</tr>
<tr>
<td>Mild</td>
<td>5</td>
<td>Type II or Type III</td>
<td>Indoor atmospheres and with rare condensation and subject to minimum wear or abrasion</td>
<td>Springs, locks, washers, tools, fasteners, electronic and electrical parts</td>
</tr>
</tbody>
</table>

* Cadmium coatings thicker than 12 µm are normally not economical.
X4. Toxicity

X4.1 Cadmium, because of its toxicity, should not be used on articles intended for use as food containers, cooking utensils, or for other objects likely to come in contact with food.

X5. Packaging Limitations

X5.1 Electrodeposited cadmium coated articles without supplementary protective films should not be packaged or packed in nonventilated containers either together or in contact with electrical and electronic equipment, because of the danger of deleterious effects on the cadmium coating from unstable electrical insulation. In addition to organic electrical insulation, phenolic resinous substances and other containing unsaturated carbon-to-carbon linkages, such as oil paints and impregnated papers, cause an abnormal attack on the cadmium by setting free in the presence of moisture, formic acid, butyric acid, and the like. Corrosion of cadmium coated articles have been noted when they have been packaged in direct contact with container materials such as wood or cardboard. Corrosion has been especially severe if the container materials have become wet or have been stored under conditions of high humidity.

X6. Cross Reference

X6.1 The correlation between the classes and types used in this specification, Specification A 165, Federal Specification QQ-P-416, and International Standard ISO 2082 are indicated in Table X6.1

<table>
<thead>
<tr>
<th>CLASS</th>
<th>THICKNESS, µm</th>
<th>TYPE</th>
<th>THICKNESS, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>25^A</td>
<td>NS</td>
<td>12</td>
</tr>
<tr>
<td>12</td>
<td>12</td>
<td>OS</td>
<td>8</td>
</tr>
<tr>
<td>8</td>
<td>8</td>
<td>TS</td>
<td>5</td>
</tr>
</tbody>
</table>

Supplementary Finish:

Type:

I  None, as deposited
II With a colored chromate treatment
III With a colorless chromate treatment

Specification QQ-P-416

<table>
<thead>
<tr>
<th>CLASS</th>
<th>THICKNESS, in. (µm):</th>
<th>SERVICE CONDITION</th>
<th>CLASS</th>
<th>THICKNESS, µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0005 (13)</td>
<td>3</td>
<td>Fe/Cd25c</td>
<td>25</td>
</tr>
<tr>
<td>2</td>
<td>0.0003 (8)</td>
<td>2</td>
<td>Fe/Cd12c</td>
<td>12^A</td>
</tr>
<tr>
<td>3</td>
<td>0.0002 (5)</td>
<td>1</td>
<td>Fe/Cd5c</td>
<td>5</td>
</tr>
</tbody>
</table>

Supplementary Finish:

Type:

I  None, as deposited
II With chromate treatment (colored)
III With phosphate treatment

Specification ISO 2082

A  Cadmium coatings thicker than 12 µm are normally not economical.
Standard Guide for Determining Mass Per Unit Area of Electrodeposited and Related Coatings by Gravimetric and Other Chemical Analysis Procedures

This standard is issued under the fixed designation B 767; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide outlines a general method for determining the mass per unit area of electrodeposited, electroless, mechanically-deposited, vacuum-deposited, anodized, and chemical conversion coatings by gravimetric and other chemical analysis procedures.

1.2 This guide determines the average mass per unit area over a measured area.

1.3 The stripping methods cited are described in specifications or in the open literature or have been used routinely by at least one laboratory.

1.4 The procedures outlined can be used for many coating-substrate combinations. They cannot be used where the coating cannot be separated from the substrate by chemical or physical means as would be the case if white brass were plated with yellow brass.

1.5 In principle, these procedures can be used to measure very thin coatings or to measure coatings over small areas, but not thin coatings over small areas. The limits depend on the required accuracy. For example, 2.5 mg/cm² of coating might require 2.5 mg of coating covering 1 cm², but 0.1 mg/cm² of coating would require 25 cm² to obtain 2.5 mg of coating.

1.6 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
A 90/A 90 M Test Method for Weight (Mass) of Coating on Iron and Steel Articles with Zinc or Zinc-Alloy Coatings
A 309 Test Method for Weight and Composition of Coating on Terne Sheet by the Triple-Spot Test
A 428 Test Method for Weight (Mass) of Coating on Aluminum-Coated Iron or Steel Articles
B 137 Test Method for Measurement of Coating Mass Per Unit Area on Anodically Coated Aluminum
B 449 Specification for Chromates on Aluminum

2.2 British Standards Institution Documents:
BS 729 Hot Dip Galvanized Coatings on Iron and Steel Articles, Specification for:
BS 1706 Electroplated Coatings of Cadmium and Zinc on Iron and Steel, Specification for:
BS 1872 Electroplated Coatings of Tin, Specification for:
BS 3189 Phosphate Treatment of Iron and Steel, Specification for:
BS 3382 Electroplated Coatings on Threaded Components, Specification for:
BS 3597 Electroplated Coatings of 65/35 Tin-Nickel Alloy, Specification for:

2.3 Government Standards:
DOD-P-16232F Phosphate Coatings, Heavy, Manganese or Zinc Base (for Ferrous Metals)

2.4 Federal Standards:
FED-STD 151b Metals; Test Methods: Test 513.1 for Weight of Coating on Hot Dip Tin Plate and Electrolytic Tin Plate
RR-T-51D Tableware and Flatware—Silverplated

2.5 Military Standard:
MIL-M-45202C Magnesium Alloys, Anodic Treatment of

2.6 ISO Standards:
ISO 2081 Metallic Coatings—Electroplated Coatings of Zinc on Iron or Steel
ISO 2082 Metallic Coatings—Electroplated Coatings on Cadmium on Iron or Steel
ISO 2093 Metallic Coatings—Electrodeposited Coatings of Tin, Annex B
ISO 2106 Anodizing of Aluminum and its Alloys—Determination of Mass Per Unit Area (Surface Density) of
Anodic Oxide Coatings—Gravimetric Method
ISO 3892 Conversion Coatings on Metallic Materials—Determination of Coating Mass Per Unit Area—Gravimetric Methods
ISO 4522/1 Metallic Coatings—Test Methods for Electrodeposited Silver and Silver Alloy Coatings—Part 1: Determination of Coating Thickness

3. Summary of Guide
3.1 The mass of a coating over a measured area is determined by the following:
3.1.1 Weighing the test specimen before and after dissolving the coating in a reagent that does not attack the substrate.
3.1.2 Weighing the coating after dissolving the substrate in a reagent that does not attack the coating, or
3.1.3 Dissolving both the coating and the substrate and quantitatively analyzing the resulting solution.
3.2 The mass per unit area is calculated from the mass and area measurements, the thickness from the mass, area, and density of the coating materials.

4. Significance and Use
4.1 The thickness of a coating is critical to its performance and is specified in many specifications calling for coatings.
4.2 These procedures are used for acceptance testing and appear in a few specifications.
4.3 Coating thickness instruments are often calibrated with thickness standards that are based on mass and area measurements.
4.4 The average thickness of a coating on the measured area can be calculated from its mass per unit area only if the density of the coating material is known.

5. Apparatus
5.1 In addition to normal chemical laboratory equipment for handling small amounts of corrosive and toxic chemicals, an accurate ruler or vernier caliper and a good balance are required. See sections 7 and 8.

6. Specimen Preparation
6.1 Size—The specimen must be large enough to permit area and mass measurement of adequate accuracy. (Section 7 and 8.2.)
6.2 Shape—The shape of the test specimen must be such that the surface area can be easily measured. A rectangular or circular test specimen is usually suitable.
6.3 Edge Condition—If the area to be measured is small and needs to be known accurately, the edges must be dressed to remove smeared coating, to remove loose burrs, and to provide well-defined and (for rectangles) straight edges. This should be considered for areas less than 100 mm². One method of dressing the edges of a rectangular specimen is to clamp the specimen between two plastic or metal blocks with the edge of the specimen flush with the edges of the blocks and then to grind and polish the edges metallographically.
6.4 Heat Treatment—If the substrate is to be dissolved leaving the coating intact, it is desirable to first heat-treat the test specimen so that the coating will not curl up tightly or fall apart. Some gold deposits of 1.5 mg/cm² will fall apart when their substrates are dissolved, but after heat treatment at 120°C for 3 h will support themselves. If the thickness of a coating (instead of its mass per unit area) is being determined, one should not use a heat treatment that might change the density of the coating material.

7. Measurement of Coated Area:
7.1 Measurement Method—The accuracy of the area measurement must be better than the desired accuracy of the mass per unit area measurement. Hence the method of measuring the area will depend on the desired accuracy and the specimen size.
7.2 Equipment—The area can be measured with a planimeter, but it is usually determined by linear measurements. Often a micrometer or vernier caliper is used. For large areas, however, a ruler may do. For maximum accuracy, a measuring microscope is used.
7.3 Number of Measurements—Because circular or rectangular specimens will not be perfectly circular or rectangular, it is desirable to measure each dimension in three places. For a rectangle, one would measure the length of each edge and the length and width through the center and obtain an average for each dimension.

Note 1—In the case of a cylinder one would normally measure the diameter and length. In one specification for galvanized wire (fencing), the length of the wire specimen is not measured, but in effect is calculated from the mass (which is measured anyway), the radius, and the density of the steel substrate. \( \rho = \frac{m}{\pi r^2 L} \)

8. Gravimetric Determination of Mass of Coating:
8.1 Specimen Size—The accuracy of the mass measurement must be better than the desired accuracy of the mass per unit area measurement. Hence, the test specimen must be large enough that the coating can be weighed with the desired accuracy.
8.2 Equipment—A balance is required, but the required sensitivity of the balance depends on the size of the test specimen, the coating thickness (coating mass), and the required accuracy of the measurement. A balance that weighs to 0.01 g is sometimes satisfactory, though a good analytical balance weighing to 0.0001 g is more versatile. A microbalance is required for small specimens of thin coatings, but it is limited to small samples.

9. Procedure
9.1 The mass of coating may be determined \( \ell \) by weighing the test specimen before and after dissolving the coating (see Annex A1) and taking the difference or \( b \) by dissolving the substrate (see Annex A1) and weighing the coating directly.
9.1.1 By Difference—The test specimen is first cleaned of any foreign material and finally rinsed with alcohol, blown dry with clean air, and weighed. The specimen is immersed in the appropriate reagent (see Annex A1) to dissolve the coating, rinsed with water, rinsed with alcohol, blown dry with clean air, and weighed again. The loss of mass is the mass of the coating. To determine if there was any dissolution of the substrate, repeat the process with the stripped substrate making
sure that it is in the reagent just as long as before. Any loss of mass enables one to make a judgment of a possible error due to any dissolution of the substrate with the coating during the stripping process.

9.1.2 By Direct Weighing—The substrate is dissolved in the appropriate reagent (see Annex A1. The coating is rinsed with water, rinsed with alcohol, blown dry with clean air, and weighed. To determine if there was any dissolution of the coating, submit the isolated coating to the same stripping process making sure that the coating is in the stripping reagent for the same length of time as it was during the stripping process. Any loss of mass enables one to make a judgment of a possible error due to any dissolution of the coating with the substrate during the stripping process.

NOTE 2—The test procedure given at the end of 9.1.1 and 9.1.2 should be conducted to evaluate a gravimetric method the first time it is used.

9.2 Determination of Mass of Coating by Chemical Analysis—This method is by nature very general. Both the coating and substrate are dissolved in a suitable reagent and then the resulting solution is analyzed for the coating material. For each coating-substrate-reagent combination, there are several analytical methods. For possible analytical methods see vols 03.05 and 03.06 of the Annual Book of ASTM Standards.

10. Calculation

10.1 Calculate the mass per unit area as follows:

\[
\text{Mass per unit area} = \frac{m}{A} \quad (\text{mg/cm}^2)
\]

where:

\( m \) = mass of coating (mg), and

\( A \) = area covered by coating (cm²)

10.2 Calculate the thickness as follows:

\[
\text{Thickness} = 10 \times \frac{M}{D} \quad (\mu\text{m})
\]

where:

\( M \) = mass per unit area (mg/cm²), and

\( D \) = density (g/cm³).

NOTE 3—The density of a coating metal is usually not the same as the handbook value or the theoretical value. For example, the density of electrodeposited gold is generally less than 19.3 g/cm³ and sometimes as low or lower than 17 g/cm³. The densities of some electrodeposited metals are given by W. H. Safranek.⁸

ANNEX

(Mandatory Information)

A1. REAGENTS FOR SELECTIVE DISSOLUTION OF METAL LAYERS

NOTE A1.1—The specific issues of standards are cited in this table and included in the literature as references because they contain the information from which this table is based.

A1.1 With many of the reagents given in Table A1.1, there may be some dissolution of the layer other than the one being stripped. Often the dissolution is not significant, but the possibility should be tested for as suggested in 9.1.1, and 9.1.2.

A1.2 Dissolution is carried out at room temperature unless otherwise indicated. All test pieces are rinsed and dried (see 9.1.1 and 9.1.2) before weighing.

<table>
<thead>
<tr>
<th>Coating</th>
<th>Substrate</th>
<th>Reagents</th>
<th>Remarks—Sources</th>
</tr>
</thead>
<tbody>
<tr>
<td>aluminum</td>
<td>steel</td>
<td>(1) 20 parts by mass NaOH, 80 parts water (2) concentrated HCl (sp gr 1.19)</td>
<td>Immers e a few min (avoid longer time) at about 90°C. While rinsing, scrub with a sponge to remove loose material. Drain off water, immerse 3 s in concentrated HCl at room temperature, scrub again in running water, and repeat entire process until there is no visible reaction in the HCl. Two or three cycles are required normally. A more detailed description is given in the 1981 issue of Test Method A 428.</td>
</tr>
<tr>
<td>aluminum</td>
<td>steel</td>
<td>(1) 200 g SbCl₅ in 1L concentrated HCl (2) 100 g SnCl₂·2H₂O in 1L concentrated HCl plus a few granules of tin</td>
<td>Mix equal volume of (1) and (2), immerse until evolution of hydrogen stops, about 1–4 min. Keep below 38°C, rinse and scrub with soft cloth. This test procedure appears in Ref (1) and in the 1981 issue of Test Method A 428.</td>
</tr>
<tr>
<td>anodized aluminum</td>
<td>aluminum</td>
<td>35 mL 85 % phosphoric acid plus 20 g/L CrO₃</td>
<td>Immerse 5 min at 100°C, rinse, dry, weigh. Repeat cycle until weight is constant. This procedure appears in the 1945 issue of Method B 137 and the 1982 issue of ISO Standard 2106.</td>
</tr>
<tr>
<td>anodized magnesium (HAE)</td>
<td>magnesium</td>
<td>300 g/L CrO₃</td>
<td>Immerse at room temperature, rinse, dry, weigh, and repeat until weight loss is less than 3.9 mg/dm². Keep piece of commercially pure aluminum in solution but not in contact with magnesium. This procedure appears in Military Standard MIL-M-45202C.</td>
</tr>
<tr>
<td>brass</td>
<td>steel</td>
<td>500 g/L CrO₃, 50 g/L H₂SO₄</td>
<td>Immers e at room temperature with mild agitation.</td>
</tr>
<tr>
<td>cadmium</td>
<td>steel</td>
<td>300 g/L NH₄NO₃</td>
<td></td>
</tr>
<tr>
<td>Coating</td>
<td>Substrate</td>
<td>Reagents</td>
<td>Remarks—Sources</td>
</tr>
<tr>
<td>-------------------------</td>
<td>-----------------</td>
<td>--------------------------------------------------------------------------</td>
<td>----------------------------------------</td>
</tr>
<tr>
<td>cadmium</td>
<td>steel</td>
<td>20g Sb2O3 in 1L concentrated HCl or 20g Sb2O3 in 800 mL concentrated HCl + 200 mL water</td>
<td>This procedure appears in the 1961 issue of British Standard 3382 and the 1986 issue of ISO Standard 2082. Immerse until evolution of gas practically stops. (2)</td>
</tr>
<tr>
<td>cadmium</td>
<td>steel</td>
<td>5% (NH₄)₂S₄O₆ plus 10% by volume of concentrated NH₄OH solution</td>
<td>Immersion. (2) This procedure appears in the 1986 issue of ISO Standard 2082.</td>
</tr>
<tr>
<td>chromate</td>
<td>aluminum</td>
<td>(1) NaNO₃</td>
<td>Immers in molten NaNO₃ at 326 to 354°C for 2 min, rinse in cold water, immerse in (2) for 30 s at room temperature. This procedure appears in the 1967 issue of Practice B 449.</td>
</tr>
<tr>
<td>chromate (aged)</td>
<td>aluminum and its alloys</td>
<td>(1) 98% NaNO₃, 2% NaOH (2) 1 part by volume water and 1 part concentrated HNO₃</td>
<td>Immerse in (1) for 2 to 5 min at 370 to 500°C (Some coatings may require the higher temp.) Rinse in water, immerse in (2) for 15 to 30 s at room temperature.</td>
</tr>
<tr>
<td>chromate (fresh)</td>
<td>aluminum and its alloys</td>
<td>1 part by volume water and 1 part concentrated HNO₃</td>
<td>Immerse 1 min at room temperature within 3 hr of application of coating. This procedure appears in the 1980 issue of ISO Standard 3892.</td>
</tr>
<tr>
<td>chromate</td>
<td>cadmium or zinc</td>
<td>50 g/L NaCN or KCN</td>
<td>Dissolve cathodically at 15 A/dm² at room temperature. This procedure appears in the 1980 issue of ISO Standard 3892. Conversion Coatings on Metallic Materials—Determination of Coating Mass per Unit Area—Gravimetric Methods</td>
</tr>
<tr>
<td>chromium</td>
<td>nickel or steel</td>
<td>12 g/L NaOH (1) dissolve 200 g Na₂S in 3/4 L water, heat to boiling with 20 g S, dilute to 1 L.</td>
<td>Chromium dissolves anodically at about 20 mA/cm². (3) Immerse in (1). When copper becomes black copper sulfide and begins to peel off, rinse and immerse in 20% NaCN to dissolve copper sulfide. (4)</td>
</tr>
<tr>
<td>copper</td>
<td>nickel or steel</td>
<td>(2) 20% NaCN</td>
<td>Dissolves about 1.2 µm/min. (3, 5)</td>
</tr>
<tr>
<td>copper</td>
<td>zinc alloys</td>
<td>500 g/L CrO₃, 50 g/L H₂SO₄</td>
<td>Dissolves zinc alloy substrate. Cool initial reaction to prevent dissolution of copper. (4)</td>
</tr>
<tr>
<td>gold</td>
<td>steel, copper, Ni-Co</td>
<td>1 part by volume water, 1 part concentrated HNO₃</td>
<td>Substrate is dissolved by immersion. Heat as required. Keep free of halides. Nickel may passivate: make contact with nickel wire to increase area of the nickel. This procedure appears in the 1985 issue of ISO Standard 4524/1. See terne plate.</td>
</tr>
<tr>
<td>lead-tin alloys</td>
<td>steel, brass</td>
<td>90% H₃PO₄</td>
<td>Immerse at 180 to 190°C, do not add water. 2.5 µm nickel dissolves in about 10 min. This procedure appears in the 1965 issue of British Standard 3382, Parts 3 and 4; Appendix F.</td>
</tr>
<tr>
<td>nickel</td>
<td>brass</td>
<td>500 g/L CrO₃, 50 g/L H₂SO₄</td>
<td>Dissolves brass substrate by immersion at room temperature with mild agitation.</td>
</tr>
<tr>
<td>nickel</td>
<td>steel</td>
<td>(1) fuming HNO₃ with mild agitation or (2) 1 part fuming 1 part concentrated HNO₃ (3) 10% CrO₃</td>
<td>Attack of steel is insignificant. Transfer quickly to CrO₃ to remove HNO₃, then rinse with water. Ni dissolves more rapidly in (2). (3, 6)</td>
</tr>
<tr>
<td>nickel</td>
<td>steel</td>
<td>(1) sodium meta-nitrobenzene sulphonate 65 g NaOH 10 g NaCN 100 g water to 1 L (2) sodium nitrobenzoic acid 65 g NaOH 20 g NaCN 100 g water to 1 L</td>
<td>Immerse in (1) or (2) at 75 to 85°C 7 µm nickel dissolves in about 30 min and dissolves copper undercoat. See British Standard 3382, Parts 3 and 4; 1965: Appendix F.</td>
</tr>
<tr>
<td>nickel or nickel over copper</td>
<td>zinc alloys</td>
<td>1 part concentrated HCl + 4 parts water</td>
<td>Dissolves zinc alloy substrate. Cool initial reaction to prevent dissolution of copper. Check for dissolution of nickel. To remove copper from nickel, see copper on nickel. (4)</td>
</tr>
<tr>
<td>phosphate (amorphous)</td>
<td>aluminum and its alloys</td>
<td>1 part by volume water and 1 part 65 to 70% (m/m) HNO₃</td>
<td>Immerse 1 min at room temperature. This procedure appears in the 1980 issue of ISO Standard 3892, Conversion Coatings on Metallic Materials—Determination of Coating Mass per Unit Area—Gravimetric Methods</td>
</tr>
<tr>
<td>phosphate (crystalline)</td>
<td>aluminum and its alloys</td>
<td>65 to 70% (m/m) HNO₃</td>
<td>Immerse 5 min at 75 ± 5°C or 15 min at room temperature. This procedure appears in the 1980 issue of ISO Standard 3892, Conversion Coatings on Metallic Materials—Determination of Coating Mass per Unit Area—Gravimetric Methods.</td>
</tr>
<tr>
<td>phosphate</td>
<td>cadmium or zinc</td>
<td>20g (NH₄)₂CrO₇ in 25 to 30% (m/m) NH₄OH</td>
<td>Immerse 3 to 5 min at room temperature. This procedure appears in the 1980 issue of ISO Standard 3892, Conversion Coatings on Metallic Materials—Determination of Coating Mass per Unit Area—Gravimetric Methods.</td>
</tr>
<tr>
<td>phosphate</td>
<td>steel</td>
<td>20 g/L Sb₂O₃ in concentrated HCl</td>
<td>Immerse at room temperature. Rub off any loose material. This procedure appears in the 1973 issue of British Standard 31899, Appendix E.</td>
</tr>
<tr>
<td>phosphate (manganese)</td>
<td>steel</td>
<td>50 g/L CrO₃</td>
<td>Immerse at least 15 min at 75 ± 5°C, rinse, dry, weigh, and repeat until weight is constant.</td>
</tr>
<tr>
<td>Coating Substrate</td>
<td>Reagents</td>
<td>Remarks—Sources</td>
<td></td>
</tr>
<tr>
<td>-------------------</td>
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<td></td>
</tr>
<tr>
<td>phosphate (zinc base) steel</td>
<td>100 g/L NaOH</td>
<td>See standard DOD-P-16232F. This procedure appears in the 1980 issue of ISO standard 3892, Conversion Coatings on Metallic Materials—Determination of Coating Mass per Unit Area—Gravimetric Methods. Immerse 5 min at 70 ± 5°C.</td>
<td></td>
</tr>
<tr>
<td>phosphate (zinc base) steel</td>
<td>90 g/L EDTA tetra sodium salt</td>
<td>This procedure appears in the 1980 issue of ISO standard 3892, Conversion Coatings on Metallic Materials—Determination of Coating Mass per Unit Area—Gravimetric Methods.</td>
<td></td>
</tr>
<tr>
<td>phosphate (zinc base) steel</td>
<td>4 g/L triethanolamine</td>
<td></td>
<td></td>
</tr>
<tr>
<td>silver nickel brass</td>
<td>19 parts by volume concentrated H$_2$SO$_4$</td>
<td>Immerse at least 10 min, rinse, dry, weight, and repeat until weight is constant.</td>
<td></td>
</tr>
<tr>
<td>silver copper alloys (1)</td>
<td>19 parts by volume concentrated H$_2$SO$_4$</td>
<td>Immerse at 60 to 70°C until silver dissolves, dip in concentrated H$_2$SO$_4$, rinse. This procedure appears in British Standard 3382, Parts 5 and 6; 1967: Appendixes H and K. See the 1985 issue of ISO standard 4522/1.</td>
<td></td>
</tr>
<tr>
<td>silver nickel and steel</td>
<td>90 g/L NaCN</td>
<td>Dissolve anodically at 2–6 V. This procedure appears in the 1985 issue of ISO Standard 4522/1.</td>
<td></td>
</tr>
<tr>
<td>silver</td>
<td>15 g/L NaOH</td>
<td>Dissolve anodically at 3 to 4 V. See Federal Specification RR-T-51D and the 1985 issue of ISO Standard 4522/1.</td>
<td></td>
</tr>
<tr>
<td>silver corrosion-resisting steel and tin alloys</td>
<td>30 g/L NaCN</td>
<td></td>
<td></td>
</tr>
<tr>
<td>terne plate long terne sheet (1)</td>
<td>100 g/L NaOH</td>
<td>Strip anodically in NaOH solution, 77 to 88°C, 12 A/dm$^2$, reverse current 5 to 15 s, rinse, dip in HCl solution for 1 to 2 s, rinse. See Test Method A 309.</td>
<td></td>
</tr>
<tr>
<td>terne plate long terne sheet (2)</td>
<td>1 volume concentrated HCl + 3 volume water</td>
<td></td>
<td></td>
</tr>
<tr>
<td>tin copper alloys</td>
<td>concentrated HCl</td>
<td>Immerse, silver replaces terne metal, remove by scrubbing in water and examine for residual terne metal. This procedure appears in the 1981 issue of Test Method A 309. Immerse and simmer until tin is dissolved. This procedure appears in British Standard 3382, Parts 5 and 6; 1967: Appendixes H and K.</td>
<td></td>
</tr>
<tr>
<td>tin copper or brass</td>
<td>20 g/L Sb$_2$O$_3$ in concentrated HCl</td>
<td>Immerse. This procedure appears in the 1964 issue of British Standard 1872, Appendix A and the 1973 issue of ISO Standard 2093, Annex B.</td>
<td></td>
</tr>
<tr>
<td>tin steel</td>
<td>40 % NaOH</td>
<td>Immerse and heat until gas evolution stops.</td>
<td></td>
</tr>
<tr>
<td>tin steel</td>
<td>120 g SbCl$_3$ in 1 L concentrated HCl</td>
<td>Immerse until evolution of gas stops and then wait 15 to 30 s. This procedure appears in Federal Test Method Standard No. 151b.</td>
<td></td>
</tr>
<tr>
<td>tin steel</td>
<td>20 g Sb$_2$O$_3$ in 1 L concentrated HCl</td>
<td>Immerse until 1 min after evolution of gas stops. (7) This procedure appears in the 1973 issue of ISO Standard 2093, Annex B. Alloy dissolves at about 0.1 µm/min. Copper dissolves at about 0.5 mg/dm$^2$/min. (8)</td>
<td></td>
</tr>
<tr>
<td>tin-lead alloy copper</td>
<td>10 mL concentrated HNO$_3$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>tin-lead alloy copper</td>
<td>15 g urea</td>
<td></td>
<td></td>
</tr>
<tr>
<td>tin-lead alloy copper</td>
<td>10 mL H$_2$O$_2$(10 volume)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>tin-lead alloy copper</td>
<td>80 mL water</td>
<td></td>
<td></td>
</tr>
<tr>
<td>tin-lead alloy copper</td>
<td>20 g/L Sb$_2$O$_3$ or 32 g SbCl$_3$ in 1 L concentrated HCl</td>
<td>Dissolve anodically at near-boiling temperature. If current density is too high, coating passivates and gas is evolved. To reactivate, make cathodic for a few seconds. This procedure appears in the 1963 issue of British Standard 3597, Appendix B.</td>
<td></td>
</tr>
<tr>
<td>zinc steel (1)</td>
<td>20 g Sb$_2$O$_3$ or 32 g SbCl$_3$ in 1 L concentrated HCl</td>
<td>Immerse in solution (2), keep below 38°C until violent evolution of hydrogen has stopped and only a few bubbles are being evolved. This procedure appears in the 1981 issue of Test Method A 90.</td>
<td></td>
</tr>
<tr>
<td>zinc steel (2)</td>
<td>5 mL of (1) in 100 mL concentrated HCl</td>
<td>Immerse until effervescence ceases. See British Standard 1706: 1960: Appendix B and the 1986 issue of ISO Standard 2081.</td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>20 g Sb$_2$O$_3$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>800 mL concentrated HCl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>200 mL water</td>
<td></td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>3.2 g SbCl$_3$ or 2 g Sb$_2$O$_3$ in 500 mL concentrated HCl</td>
<td>Immerse until vigorous reaction virtually ceases. Brush off loose deposits. This procedure appears in the 1971 issue of British Standard 729, Appendix.</td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>water to 1 L</td>
<td></td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>1-to-1 HCl</td>
<td>Immerse in 1-to-1 HCl until violent evolution of hydrogen has stopped and only a few bubbles are being evolved. Keep below 38°C. This procedure appears in the 1981 issue of Test Method A 90.</td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>ammonium persulfate 5 g</td>
<td>This procedure appears in the 1961 issue of British Standard 3382.</td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>ammonium hydroxide (sp. gr. 0.880)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>10 mL</td>
<td>Immerse.</td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>water 90 mL</td>
<td></td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>500 mL conc HCl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>300 g/L NH$_4$NO$_3$</td>
<td>This procedure appears in the 1986 issue of ISO Standard 2081.</td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>500 mL conc HCl</td>
<td>Immerse.</td>
<td></td>
</tr>
<tr>
<td>zinc steel</td>
<td>1 g propin-2-ol-1 (C$_3$H$_4$O)</td>
<td>This procedure appears in the 1986 issue of ISO Standard 2081.</td>
<td></td>
</tr>
</tbody>
</table>

A The boldface numbers in parentheses refer to the list of references at the end of this guide.
REFERENCES

Standard Test Method for Short Rod Fracture Toughness of Cemented Carbides

This standard is issued under the fixed designation B 771; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the fracture toughness of cemented carbides ($K_{IcSR}$) by testing slotted short rod or short bar specimens.

1.2 Values stated in SI units are to be regarded as the standard. Inch-pound units are provided for information only.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

E 399 Test Method for Plane-Strain Fracture Toughness of Metallic Materials
E 616 Terminology Relating to Fracture Testing

3. Terminology Definitions

3.1 stress intensity factor, $K_I$ (dimensional units $FL^{-3/2}$)—the magnitude of the ideal-crack-tip stress field for mode I in a linear-elastic body.

Note 1—Values of $K_I$ for mode I are given by:

$$K_I = \lim_{r \to 0} \left[ \sigma_y \sqrt{2\pi r} \right]$$

where:

$r$ = distance directly forward from the crack tip to a location where the significant stress $\sigma_y$ is calculated, and

$\sigma_y$ = principal stress normal to the crack plane.

3.2 Abbreviations: fracture toughness of cemented carbide, $K_{IcSR}$ (dimensional units $FL^{-3/2}$)—the material-toughness property measured in terms of the stress-intensity factor $K_I$ by the operational procedure specified in this test method.

4. Summary of Test Method

4.1 This test method involves the application of an opening load to the mouth of the short rod or short bar specimen which contains a chevron-shaped slot. Load versus displacement across the slot at the specimen mouth is recorded autographically. As the load is increased, a crack initiates at the point of the chevron slot and slowly advances longitudinally, tending to split the specimen in half. The load goes through a smooth maximum when the width of the crack front is about one third of the specimen diameter (short rod) or breadth (short bar). Thereafter, the load decreases with further crack growth. Two unloading-reloading cycles are performed during the test to measure the effects of any macroscopic residual stresses in the specimen. The fracture toughness is calculated from the maximum load in the test and a residual stress parameter which is evaluated from the unloading-reloading cycles on the test record.

5. Significance and Use

5.1 The property $K_{IcSR}$ determined by this test method is believed to characterize the resistance of a cemented carbide to fracture in a neutral environment in the presence of a sharp crack under severe tensile constraint, such that the state of stress near the crack front approaches tri-tensile plane strain, and the crack-tip plastic region is small compared with the crack size and specimen dimensions in the constraint direction. A $K_{IcSR}$ value is believed to represent a lower limiting value of fracture toughness. This value may be used to estimate the relation between failure stress and defect size when the conditions of high constraint described above would be expected. Background information concerning the basis for development of this test method in terms of linear elastic fracture mechanics may be found in Refs (1-4).

5.2 This test method can serve the following purposes:

5.2.1 To establish, in quantitative terms significant to service performance, the effects of fabrication variables on the fracture toughness of new or existing materials, and

5.2.2 To establish the suitability of a material for a specific application for which the stress conditions are prescribed and for which maximum flaw sizes can be established with confidence.

6. Specimen Configuration, Dimensions, and Preparation

6.1 Both the round short rod specimen and the rectangular shaped short bar specimen are equally acceptable and have been found to have the same calibration (5). The short rod dimensions are given in Fig. 1; the short bar in Fig. 2.

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1. This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.06 on Cemented Carbides.


3. The boldface numbers in parentheses refer to the list of references at the end of this standard.
6.2 Grip Slot—Depending on the apparatus used to test the specimen, a grip slot may be required in the specimen front face, as shown in Fig. 3. The surfaces in the grip slot shall have a smooth ground finish so that the contact with each grip will be along an essentially continuous line along the entire grip slot, rather than at a few isolated points or along a short segment within the grip slot.

6.3 Crack-Guiding Slots—These may be ground using a diamond abrasive wheel of approximately 124.63 mm (4.960 in.) diameter, with a thickness of 0.36 ± 0.01 mm (0.0140 ± 0.0005 in.). The resulting slots in the specimen are slightly thicker than the diamond wheel (0.38 ± 0.02 mm, or 0.015 ± 0.001 in.). A diamond concentration number of 50, and a grit size of 150 are suggested. Dimensions are given in Fig. 1 and Fig. 2 for two slotting options: (1) Specimens with curved slot bottoms made by plunge feeding the specimen onto a diamond cutting wheel of a given radius, and (2) Specimens with straight slot bottoms made by moving the specimen by a cutting wheel. The values of \( a_o \) and \( \theta \) for the two slot configurations are chosen to cause the specimen calibration to remain constant.

7. Apparatus

7.1 The procedure involves testing of chevron-slotted specimens and recording the load versus specimen mouth opening displacement during the test.

7.2 Grips and Fixtures for Tensile Test Machine Loading—Grip slots are required in the specimen face for this test method, as shown in Fig. 3. Fig. 4 shows the grip design. Grips shall have a hardness of 45 HRC or greater, and shall be capable of providing loads to at least 1560 N (350 lbf). The grips are attached to the arms of tensile test machine by the pin and clevis arrangement shown in Fig. 5. The grip lips are
inserted into the grip slot in the specimen, and the specimen is loaded as the test machine arms apply a tensile load to the grips. A transducer for measuring the specimen mouth opening displacement during the test, and means for automatically recording the load-displacement test record, such as an X-Y recorder, are also required when using the tensile test machine apparatus. A suggested design for the specimen mouth opening displacement gage appears in Fig. 6. The gage shall have a displacement resolution of 0.25 µm (10⁻⁶ in.) or better. However, it is not necessary to calibrate the displacement axis of the test record since only displacement ratios are used in the data analysis.

7.3 Distributed Load Test Machine—An alternative special purpose machine that has been found suitable for the test requires no grip slot in the front face of the specimen. A thin stainless steel inflatable bladder is inserted into the chevron slot in the mouth of the specimen. Subsequent inflation of the bladder causes it to press against the inner surfaces of the slot, thus producing the desired loading. The machine provides load and displacement outputs, which must be recorded externally on a device such as an X-Y recorder.

7.4 Testing Machine Characteristics—It has been observed that some grades of carbides show a “pop-in” type of behavior in which the load required to initiate the crack at the point of the chevron slot is larger than the load required to advance the crack just after initiation, such that the crack suddenly and audibly jumps ahead at the time of its initiation. Occasionally, the load at crack initiation can exceed the load maximum which occurs as the crack passes through the critical location in the specimen. When this occurs, a very stiff machine with controlled displacement loading is necessary in order to allow the crack to arrest well before passing beyond the critical location. The large pop-in load is then ignored, and the subsequent load maximum as the crack passes through the critical location is used to determine $K_{IC}$-SR. Stiff machine loading is also required in order to maintain crack growth stability to well beyond the peak load in the test, where the second unloading-reloading cycle is initiated.

8. Procedure

8.1 Number of Tests—A minimum of 3 replicate tests shall be made.

8.2 Specimen Measurement:

8.2.1 Measure and record all specimen dimensions. If the dimensions are within the tolerances shown in Fig. 1 and Fig. 2, no correction to the data need be made for out-of-tolerance dimensions. If one or more of the parameters $a_o$, $W$, $\theta$ or $\tau$ are out of tolerance by up to 3 times the tolerances shown in Fig. 1 and Fig. 2, valid tests may still be made by the application of the appropriate factors to account for the deviation from standard dimensions (see 9.3). If the slot centering is outside the indicated tolerance, the crack is less likely to follow the chevron slots. However, the test may still be considered successful if the crack follows the slots sufficiently well, as discussed in 9.2.

8.2.2 The slot thickness measurement is critical on specimens to be tested on a Fractometer. It should be measured to within 0.013 mm (0.0005 in.) at the outside corners of the slot using a feeler gage. If a feeler gage blade enters the slot to a depth of 1 mm or more, the slot is said to be at least as thick as the blade. Because the saw cuts forming the chevron slot overlap somewhat in the mouth of the specimen, and because the cuts may not meet perfectly, the slot width near the center of the mouth may be larger than the width at the outside corners. If the slot width near the center exceeds the slot width

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4 Fractometer, a trademark of Terra Tek Systems, 360 Wakara Way, Salt Lake City, UT 84108, has been found satisfactory for this purpose.
at the corners by more than 0.10 mm (0.004 in.), a test of that specimen by a Fractometer is invalid.

8.3 Specimen Testing Procedure:
8.3.1 Load Transducer Calibration:
8.3.1.1 Calibrate the output of the load cell in the test machine to assure that the load cell output, as recorded on the load versus displacement recorder, is accurately translatable into the actual force applied to the specimen. In those cases in which a distributed load test machine is used (see 7.3), the calibration shall be performed according to the instructions in Annex A1.

8.3.1.2 Install the specimen on the test machine. If using the tensile test machine (see 7.2), operate the test machine in the “displacement control” mode. Bring the grips sufficiently close together such that they simultaneously fit into the grip slot in the specimen face. Then increase the spacing between the grips very carefully until an opening load of 10 to 30 N (2 to 7 lb) is applied to the specimen. Check the alignment of the specimen with respect to the grips, and the alignment of the grips with respect to each other. The grips shall be centered in the specimen grip slot to within 0.25 mm (0.010 in.). The vertical offset between the grips shall not exceed 0.13 mm (0.005 in.). Using a magnifying glass, observe the grips in the grip slot from each side of the specimen to assure that the specimen is properly installed. Correct any deviations from the desired specimen alignment.

8.3.1.3 Install the specimen mouth opening displacement gage on the specimen. The gage must sense the mouth opening no farther than 1 mm (0.040 in.) from the front face of the specimen. If the gage design of Fig. 6 is used, the contact force between the gage arms and the specimen can be adjusted with a rubber elastic band so the gage will support itself, as indicated in Fig. 5. However, the contact force must not be more than 2 N (0.5 lb), as it increases the measured load to fracture the specimen.

8.3.1.4 Adjust the displacement (x-axis) sensitivity of the load-displacement recorder to produce a convenient-size data trace. A 70° angle between the x-axis and the initial elastic loading trace of the test is suggested. A quantitative calibration of the displacement axis is not necessary.

8.3.1.5 With the load-displacement recorder operating, test the specimen by causing the specimen mouth to open at a rate of 0.0025 to 0.0125 mm/s (0.0001 to 0.0005 in./s). The specimen is unloaded by reversing the motion of the grips twice during the test. The first unloading is begun when the slope of the unloading line on the load-displacement record will be approximately 70% of the initial elastic loading slope. (For estimating the point at which the unloadings should be initiated, it can be assumed that the unloading paths will be linear and will point toward the origin of the load-displacement record.) The second unloading is begun when the unloading slope will be approximately 35% of the initial elastic loading slope. Each unloading shall be continued until the load on the specimen has decreased to less than 10% of the load at the initiation of the unloading. The specimen shall be immediately reloaded and the test continued after each unloading. The test record generated by the above procedure should be similar to that of Fig. 7.

8.3.2 Crack-Pop-In—If a sudden load drop occurs simultaneously with an audible “pop” or “tick” sound from the specimen during the initial part of the test when the load is rising most rapidly, a crack pop-in has occurred at the point of
the chevron slot. If the pop-in is large, such that the first unloading slope that can be drawn is less than half of the initial elastic loading slope, the test is invalid.

9. Calculation and Interpretation of Results

9.1 Remove the specimen from the apparatus. If the two halves are still joined, break them apart with a wedge. Examine the fracture surfaces for any imperfections that may have influenced the measured peak load. Any imperfections (such as a void, a surface irregularity, or a piece of foreign matter) that is visible to the naked eye may influence the measurement if the imperfection is located between 7.6 mm (0.30 in.) and 14.2 mm (0.56 in.) from the mouth of the specimen. Imperfections outside this region do not affect the peak load unless they are very large. Discard the data whenever the peak load may have been affected by an imperfection in the fracture plane.

9.2 Examine the fracture surface to determine how well the crack followed the chevron slots in splitting the specimen apart. If the "crack follow" was imperfect, the crack will have cut substantially farther into one half of the specimen than the other, and the crack surface will not intersect the bottom of the chevron slots, as shown in Fig. 8. The size of the lip overhanging the slot bottom determines whether the crack follow was sufficiently good for a valid test. Measure the "overhang" of the fracture surface over the slot bottom on each side of the chevron at a distance of 10.8 mm (0.425 in.) from the mouth of the specimen (Fig. 8). If the sum, $\Delta b$, of the overhangs on each side of the chevron exceeds 0.25 mm (0.010 in.), the test is invalid.

NOTE 2—Imperfect crack follow often results from poor centering of the chevron slot in the specimen. However, it can also result from strong residual stresses in the test specimen.

9.3 Out-of-Tolerance Dimension Corrections—If the specimen dimensions are all within the tolerances specified in Fig. 1 and Fig. 2, assign $C_c = 1$, where $C_c$ is the specimen configuration correction factor. If $a_o$, $W$, $\theta$, or $\tau$ differ from their specified tolerance by more than 3 times the tolerance specified in Fig. 1 or Fig. 2, the sample is invalid. If $a_o$, $W$, $\theta$, or $\tau$ differ from their specified tolerance by less than or equal to three times the tolerance specified in Fig. 1 or Fig. 2, compensation can be made using the correction factors defined in 9.3.1 through 9.3.5 (7). The subscript $nom$ refers to the nominal dimension specified in Fig. 1 or Fig. 2.

9.3.1 If $a_o$ is within tolerance, assign $C_a = 1$. However, if $a_o$ is out of tolerance, calculate:

$$C_a = 1 + 1.8(a_o - a_{nom})/B.$$  \hspace{1cm} (2)

9.3.2 If $W$ is within tolerance, assign $C_w = 1$. However, if $W$ is out of tolerance, calculate:

$$C_w = 1 - 0.7(W - W_{nom})/B.$$  \hspace{1cm} (3)

9.3.3 If $\theta$ is within tolerance, assign $C_\theta = 1$. However, if $\theta$ is out of tolerance, calculate:

$$C_\theta = 1 - 0.015(\theta - \theta_{nom})$$  \hspace{1cm} (4)

where $\theta$ is in degrees.

9.3.4 If $\tau$ is within tolerance, or if $\tau$ is out of tolerance and grip loading was used, assign $C_\tau = 1$. However, if $\tau$ is out of tolerance and distributed loading (Fractometer) was used, calculate:

$\text{FIG. 7 Sample Load-Displacement Test Record with Data Analysis Constructions and Definitions}$

$\text{FIG. 8 Short Rod or Short Bar Tested Specimen Half with Imperfect Crack Guidance by the Slots}$
9.3.5 Calculate \( C_c \) from:
\[
C_c = C_c C_c C_c C_c
\]  
(6)

9.4 Analyze the test record to obtain \( p \), the residual stress parameter. The basis for the use of \( p \) to compensate for the effects of any macroscopic longitudinal residual stresses in the specimen is given in Ref (3).

9.4.1 Locate the “high” and “low” points on each unloading-reloading cycle. A high point is the point at which the mouth opening displacement started decreasing to unload the specimen, and the corresponding low point is on the reloading part of the unloading-reloading cycle at half the load of the high point. The high and low points are labeled H and L, respectively, in Fig. 7.

9.4.2 Draw the ideal elastic release path approximations through the high and low points of each unloading-reloading cycle (slanted dashed lines of Fig. 7).

9.4.3 Draw the horizontal “average load” line between the two ideal elastic release lines (Fig. 7). The average load line is drawn at the level of the average load on the data trace between the two unloading-reloading cycles. It must be drawn horizontal, but the choice of the average load can vary by \( \pm 5\% \) from the correct value without materially affecting the results.

9.4.4 Measure \( \Delta X \) (the distance between the ideal elastic release lines at the average load line) and \( \Delta X_r \) (the distance between the ideal elastic release lines at the zero load line). Calculate \( p = \Delta X / \Delta X_r \). If the release lines cross before reaching the zero load axis, \( \Delta X_r \), and therefore \( p \), are considered to be negative. The analysis is nevertheless valid. However, the test is considered invalid unless \( -0.15 < p < +0.15 \), inasmuch as the theory assumes relatively small values of \( p \).

9.5 From the test record, measure the maximum load in the experiment, \( F_c \).

9.6 Calculate \( K_{ESR} \).

9.6.1 If grips and fixtures for tensile test machine loading are used, calculate:
\[
K_{QSR} = A F_c C_c (1 + p) B^{1/2}
\]  
(7)

in which \( A = 22.0 \) and \( B \) is the specimen diameter (short rod) or breadth (short bar) in the system of units in which \( F_c \) and \( K_{QSR} \) are expressed. \( A \) is the dimensionless specimen configuration calibration constant defined in Ref (1) and evaluated in Ref (6). It is not a function of machine stiffness, material properties, nor absolute specimen size, so long as the scaled specimen configuration, including the location of the applied load on the specimen, remains constant. The calibrated value of \( A \) is uncertain by about \( 5\% \).

9.6.2 If the distributed load test machine is used, calculate:
\[
K_{QSR} = C_c (l + p) K_{DL}
\]  
(8)

where:

\[
C_c = 1 - 12.5(\tau - \tau_{nom})/B
\]  
(5)

9.6.3 If all of the validity requirements of the test are satisfied, then:
\[
K_{ESR} = K_{QSR}
\]  
(9)

Validity requirements are specified in 6.4, 6.5, 8.2, 8.3.6, 9.1, 9.2, and 9.4.4.

10. Report

10.1 The report shall include the following for each specimen tested:

10.1.1 Specimen identification,

10.1.2 Environment of test, if other than normal atmosphere and room temperature,

10.1.3 Diameter, \( B \) (short rod) or Breadth, \( B \) (short bar),

10.1.4 Length, \( W \),

10.1.5 Height, \( H \) (short bar only),

10.1.6 Chord angle, \( \theta \),

10.1.7 Slot thickness, \( \tau \),

10.1.8 Crack overhang sum, \( \Delta b \), in accordance with 9.2.

10.1.9 Comments on any unusual appearance of the fracture surface, and

10.1.10 \( K_{ESR} \) or \( K_{QSR} \) with a summary of the invalidities.

11. Precision and Bias

11.1 Precision is the closeness of agreement between individual test results. The precision of a \( K_{ESR} \) determination is a function of the precision and bias of the various measurements of the specimen and testing fixtures, the precision and bias of the load and displacement measuring and recording devices used to produce the test record, and the precision of the constructions made on the record.

11.2 The precision of \( K_{ESR} \) measurements is estimated based on a round robin test series reported in a research report.\(^5\)

Six laboratories participated in the round robin, in which five different grades of cemented carbides were tested. Each laboratory tested approximately five short rod specimens of each grade of material. The average within-laboratory percent standard deviation (the repeatability) was 2.9 %. This pertains to tests done on the same material by the same operator using the same equipment within a short time. The average between-laboratory percent standard deviation (the reproducibility) was 5.0 %.

11.3 Bias is a systematic error that contributes to the difference between a population mean of the measurements and an accepted reference or true value. Since there is no accepted method for determining the true fracture toughness of cemented carbides, no statement on bias can be made.

\(^5\) Supporting data are available from ASTM Headquarters. Request RR: B09-1005.
ANNEX

A1. CALIBRATION OF THE DISTRIBUTED LOAD TEST MACHINE

A1.1 The equation for the fracture toughness (critical stress intensity factor) for the specimen geometries of this test method and for the loading configuration used by the distributed load (DL) test machine is:

\[ K_{DL} = 8.26 P_c \sqrt{B} \]  (A1.1)

where:

\( P_c \) = the peak pressure in the inflatable bladder during the test.

The factor 8.26 is a dimensionless constant for the specimen configuration and the loading configuration of the distributed load test machine. It is entirely comparable to the dimensionless constant \( A = 22.0 \) which applies for the grip loading configuration of this test method (see 9.6.1).

A1.2 The machine is normally calibrated to display the signal from the pressure transducer in units of pressure (MPa or ksi) times 8.26 = 22.0, where \( B = 0.0127 \text{ m} \) for the SI read-out, or \( B = 0.500 \text{ in.} \) for the inch-pound read-out. Thus, the peak reading displayed in a test is the \( K_{DL} \) for the specimen.

A1.3 The machine shall be calibrated in accordance with the manufacturer’s instructions before testing each specimen. This involves switching a shunt resistor into the bridge circuit of the pressure transducer to provide the same bridge balance offset as a known pressure. The amplifier gain is then adjusted to cause the display to read the correct value.

A1.4 The equivalent pressure signal obtained by switching the shunt resistor into the bridge circuit should be checked yearly, or more often by the manufacturer, or as follows:

A1.4.1 Disconnect the pressure tube from the intensifier, zero the display, and connect a pressure tube from a pressure standard to the intensifier.

A1.4.2 Apply an accurately known pressure of about 14 MPa (2 ksi). Adjust the amplifier gain to obtain an output display of 0.931 MPa \( \sqrt{m} \) per MPa of applied pressure (SI), or 5.84 ksi \( \sqrt{m} \) per ksi of applied pressure (inch-pound). Check that the display returns to zero for zero applied pressure.

A1.4.3 With zero applied pressure and the display reading zero, switch the calibrating resistor into the bridge circuit and note the display. This displayed reading should be used in subsequent test-to-test calibrations.

Note A1.1—In performing the above calibration, considerable care must be exercised to prevent air from entering the machine’s pressure system, as air destroys the stiffness of the system.

REFERENCES

Standard Specification for Iron Graphite Sintered Bearings (Oil-Impregnated)\textsuperscript{1}

This standard is issued under the fixed designation B 782; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (\(\epsilon\)) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers iron-base sintered metal powder, oil-impregnated bearings. There are two grades, depending on the level of combined carbon (see Table 1):

1.1.1 Grade 1, Iron Graphite—low combined carbon and

1.1.2 Grade 2, Iron Graphite—high combined carbon.

1.2 The values stated in inch-pound units are the standard. The metric equivalents of inch-pound units may be approximate.

\textbf{NOTE 1}—Definitions of powder metallurgy terms can be found in Definitions B 243. Additional useful information is available in the Related Material section of Vol 02.05 of the Annual Book of ASTM Standards.

\textbf{NOTE 2}—Information on design, permissible loads, dimensional tolerances, and recommended press fits and running clearances are provided in Appendix X1.

2. Referenced Documents

2.1 \textit{ASTM Standards}:

- B 243 Terminology of Powder Metallurgy\textsuperscript{2}
- B 328 Test Method for Density, Oil Content, and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings\textsuperscript{3}
- E 9 Test Methods of Compression Testing of Metallic Materials at Room Temperature\textsuperscript{3}
- E 1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel and in Iron, Nickel, and Cobalt Alloys\textsuperscript{4}

3. Ordering Information

3.1 Orders for material under this specification shall include the following information:

3.1.1 Grade (Table 1),

3.1.2 Dimensions, and

3.1.3 Certification (see Section 13).

\textbf{TABLE 1 Chemical Requirements}

<table>
<thead>
<tr>
<th>Element</th>
<th>Grade 1</th>
<th>Grade 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Iron, min</td>
<td>94.5</td>
<td>94.5</td>
</tr>
<tr>
<td>Graphite</td>
<td>2.0 to 3.0</td>
<td>1.5 to 2.5</td>
</tr>
<tr>
<td>Total other elements by difference, max</td>
<td>2.0</td>
<td>2.0</td>
</tr>
<tr>
<td>Combined carbon</td>
<td>0 to 0.5</td>
<td>0.5 to 1.0</td>
</tr>
</tbody>
</table>

4. Materials and Manufacture

4.1 Bearings shall be made by cold compacting and sintering metal powders, with or without sizing, to produce finished parts conforming to the requirements of this specification.

5. Chemical Composition

5.1 The material shall conform to the requirements prescribed in Table 1.

6. Physical Properties

6.1 \textit{Density}—The density of bearings supplied fully impregnated with lubricant shall be within the limits prescribed in Table 2. Density determinations shall be made in accordance with Test Method B 328.

6.2 \textit{Oil Content}—Oil content of bearings shall not be less than 18 \% for each grade when determined in accordance with Test Method B 328.

7. Mechanical Properties

7.1 \textit{Radial Crushing Force}:

7.1.1 Radial crushing force shall be determined by compressing the test specimen between two flat steel surfaces at a “no load” speed no greater than 0.2 in./min (5 mm/min), the direction of the load being normal to the longitudinal axis of the specimen. The point at which the load drops as a result of the first crack shall be considered the crushing strength. This test shall be applied to plain cylindrical bearings. Flanged bearings shall be tested by cutting off the flange and compressing the two sections separately. Each section shall meet the minimum strength requirements prescribed in Table 3.

7.1.2 Radial crushing force shall not be less than the value calculated as follows:

\[ P = \frac{KLT^2}{(D - T)^2} \]  

(1)
where:

\[ P = \text{radial crushing force, lbf (N)}; \]
\[ D = \text{outside diameter of bearing, in. (mm)}; \]
\[ T = \text{wall thickness of bearing, in. (mm)}; \]
\[ K = \text{strength constant as shown in Table 3 for the grade specified, psi (MPa (N/mm\(^2\))}; \]
\[ L = \text{length of bearings, in. (mm)}; \]

7.1.3 Concerning spherical bearings, sample parts from a lot will be machined to a straight wall and radially crushed to calculate the \( K \) value. Sample parts from the same lot will be radially crushed as is (whole part). By correlation, the minimum radial crush value will be established on the whole bearing and so specified as the minimum radial crush value for the part.

8. Workmanship, Finish, and Appearance

8.1 Bearings shall be uniform in composition. When cut or fractured, the exposed surface shall be of uniform appearance.

9. Sampling

9.1 Lot—Unless otherwise specified, a lot shall consist of parts of the same form and dimensions made from powders of the same composition, formed and sintered under the same conditions, and submitted for inspection at one time.

9.2 Sample for Chemical Analysis—If required, at least one sample for chemical analysis shall be taken from each lot. A representative sample of chips may be obtained by milling, drilling, or crushing at least two pieces with clean dry tools without lubrication. If the parts are not completely dry, the parts selected for test shall have the oil extracted in accordance with Test Method B 328.

9.3 Mechanical Tests—The manufacturer and purchaser shall agree on a representative number of specimens for mechanical tests.

10. Analytical Methods

10.1 Carbon Analysis—Carbon analysis is a procedure for determining the total, the graphitic, and the combined carbon in iron-graphite sintered bearings.

10.1.1 Oil Extraction—Parts must be dry and free of oil before running tests. The preferred method of oil extraction is by the Soxhlet apparatus method specified in Test Method B 328. Upon agreement between purchaser and supplier, a low-temperature furnace (approximately 1000°F) with a nitrogen or inert atmosphere may be used.

10.1.2 Total Carbon—Obtain total carbon in accordance with Test Method E 1019 with the exception of a \( \frac{1}{4}-\text{g} \) sample may be used upon agreement between customer and supplier.

10.1.3 Graphitic Carbon—The amount of graphitic carbon is found using the following procedure: Weigh and transfer a \( \frac{1}{4}-\text{g} \) sample to a 400-mL beaker. Add 25 mL of distilled water. Carefully add 25 mL of concentrated nitric acid and gently boil until all the iron is in solution. At this point, add five to ten drops of 48% hydrofluoric acid to ensure complete solubility of all carbides, silicates, and so forth. Filter the solution through a porous combustion crucible, wash with hot water until free of acid, then once with ethyl alcohol. Dry at 100°C for 1 h. After drying, add approximately 1 g of carbon-free iron chips and 1 g of copper chips (or another approved accelerator) and follow the procedure for determining the total carbon.

10.1.4 Combined Carbon—To obtain the amount of combined carbon, subtract the amount of graphitic carbon from the total carbon.

10.1.5 Alternative Method of Determining Combined Carbon—The combined carbon may be a metallographic estimate of the carbon in the material.

11. Inspection

11.1 Unless otherwise specified, inspection of parts supplied on contract shall be made by the purchaser at the destination.

12. Rejection

12.1 Rejection based on tests made in accordance with this specification shall be reported in writing to the manufacturer within 30 days of receipt of shipment; however, the rejected parts should not be returned without authorization from the producer.

13. Certification

13.1 A certification based on the manufacturer’s quality control that the material conforms to the requirements of this specification shall be the basis of shipment of the material. A certificate covering the conformance of the material to these specifications shall be furnished by the manufacturer upon request of the purchaser.

14. Keywords

14.1 density; iron graphite bearings; \( K \) strength constant; oil content; oil-impregnated; porosity
X1. EXPLANATORY INFORMATION

X1.1 Design Information

X1.1.1 In calculating permissible loads, the operating conditions, housing conditions, and construction should be considered. In general, this material has less resistance to seizure and corrosion than copper-base material. The maximum static bearing load should not exceed 11 000-psi (76-MPa (N/mm²)) Grade 1 or 15 000-psi (104-MPa (N/mm²)) Grade 2 of projected bearing area (length times inside diameter of bearing) for this material. This figure is 75 % of the value for the compression deformation limit (yield strength permanent set of 0.001 in. (0.025 mm) for specimens 11 ⁄8 in. (28.6 mm) in diameter and 1 in. (25.4 mm) in length) as determined in accordance with Test Methods E 9.

X1.2 Permissible Loads

X1.2.1 Permissible loads for various operating conditions are given in Table X1.1.

X1.3 Dimensional Tolerances

X1.3.1 Commercial dimensional tolerances are given in Table X1.2.

X1.4 Press Fits

X1.4.1 Plain cylindrical journal bearings are commonly installed by press fitting the bearing into a housing with an insertion arbor. For housings rigid enough to withstand the press fit without appreciable distortion and for bearings with wall thickness approximately one eighth of the bearing outside diameter, the press fits shown in Table X1.3 are recommended.

X1.5 Running Clearance

X1.5.1 Proper running clearance for sintered bearings depends to a great extent on the particular application. Therefore, only minimum recommended clearances are listed in Table X1.4. It is assumed that ground steel shafting will be used and that all bearings will be oil impregnated.

X1.6 Flange and Thrust Bearing Specifications

X1.6.1 Diameter and thickness specifications for flange and thrust washers are shown in Table X1.5.

X1.7 Lubrication

X1.7.1 It was found that a circulating-type oil containing rust and oxidation inhibitors is the most desirable type of oil to be used. The viscosity should be specified by the user in accordance with the application.

---

**TABLE X1.1 Permissible Loads**

<table>
<thead>
<tr>
<th>Shaft Velocity, ft/min (m/min)</th>
<th>Permissible Loads, psi (MPa (N/mm²))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Slow and intermittent</td>
<td>3600 (25)</td>
</tr>
<tr>
<td>25 (7.6)</td>
<td>1800 (12)</td>
</tr>
<tr>
<td>50 to 100 (15.2 to 30.4), incl</td>
<td>450 (3.1)</td>
</tr>
<tr>
<td>Over 100 to 150 (30.4 to 45.7), incl</td>
<td>300 (2.1)</td>
</tr>
<tr>
<td>Over 150 to 200 (45.7 to 61), incl</td>
<td>225 (1.6)</td>
</tr>
<tr>
<td>Over 200 (61)</td>
<td></td>
</tr>
</tbody>
</table>

Grade 1, 2

---

**TABLE X1.2 Dimensional Tolerances**

<table>
<thead>
<tr>
<th>Inside Diameter and Outside Diameter</th>
<th>Total Diameter Tolerance^a</th>
</tr>
</thead>
<tbody>
<tr>
<td>in. mm</td>
<td>in. mm mm</td>
</tr>
<tr>
<td>Up to 0.760 up to 19.31</td>
<td>0.001 0.025 0.001 0.025</td>
</tr>
<tr>
<td>0.761 to 1.510 19.32 to 38.36</td>
<td>0.0015 0.025 0.0015 0.04</td>
</tr>
<tr>
<td>1.511 to 2.510 38.37 to 63.76</td>
<td>0.002 0.05 0.002 0.05</td>
</tr>
<tr>
<td>2.511 to 3.010 63.77 to 76.46</td>
<td>0.003 0.08 0.002 0.08</td>
</tr>
<tr>
<td>3.011 to 4.010 76.47 to 101.86</td>
<td>0.004 0.10 0.004 0.10</td>
</tr>
<tr>
<td>4.011 to 5.010 101.87 to 127.26</td>
<td>0.005 0.13 0.005 0.13</td>
</tr>
<tr>
<td>5.011 to 6.010 127.27 to 152.65</td>
<td>0.006 0.15 0.006 0.15</td>
</tr>
</tbody>
</table>

Length Total Length Tolerance^b|

<table>
<thead>
<tr>
<th>in. mm</th>
<th>mm</th>
<th>in. mm</th>
<th>mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Up to 1.495 up to 37.97</td>
<td>0.010 0.25</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.496 to 1.990 37.98 to 50.54</td>
<td>0.015 0.38</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.991 to 2.990 50.55 to 75.96</td>
<td>0.020 0.51</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.991 to 4.985 75.97 to 126.61</td>
<td>0.030 0.76</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Outside Diameter Wall Thickness, max Concentricity Tolerance^c|

<table>
<thead>
<tr>
<th>in. mm</th>
<th>mm</th>
<th>in. mm</th>
<th>mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Up to 1.510 up to 38.36</td>
<td>up to 0.355 9.02 0.003 0.08</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.511 to 2.010 38.37 to 51.06</td>
<td>up to 0.505 12.83 0.004 0.10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.011 to 4.010 51.07 to 101.86</td>
<td>up to 1.010 25.65 0.005 0.13</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4.011 to 5.010 101.87 to 127.26</td>
<td>up to 1.510 38.35 0.006 0.15</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5.011 to 6.010 127.27 to 152.65</td>
<td>up to 2.010 51.05 0.007 0.18</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

^aTotal tolerance on the inside diameter and outside diameter is a minus tolerance only.

^bTotal tolerance is split into plus and minus.

^cTotal indicator reading.

**TABLE X1.3 Recommended Press Fits**

<table>
<thead>
<tr>
<th>Outside Diameter of Bearing</th>
<th>Press Fit</th>
</tr>
</thead>
<tbody>
<tr>
<td>in. mm</td>
<td>Min</td>
</tr>
<tr>
<td>in. mm</td>
<td>in. mm</td>
</tr>
<tr>
<td>Up to 0.760 up to 19.31</td>
<td>0.001 0.025 0.003 0.08</td>
</tr>
<tr>
<td>0.761 to 1.510 19.32 to 38.36</td>
<td>0.0015 0.04 0.004 0.10</td>
</tr>
<tr>
<td>1.511 to 2.510 38.37 to 63.76</td>
<td>0.002 0.05 0.005 0.13</td>
</tr>
<tr>
<td>2.511 to 3.010 63.77 to 76.45</td>
<td>0.002 0.05 0.006 0.15</td>
</tr>
<tr>
<td>Over 3.010 over 76.45</td>
<td>0.002 0.05 0.007 0.18</td>
</tr>
</tbody>
</table>

^aFor shaft velocities over 200 ft/min, the permissible loads may be calculated as follows:

\[
P = 50 000/V
\]

where:

\[
P = \text{safe load, psi of projected area and}
\]

\[
V = \text{shaft velocity, ft/min (m/min)}.
\]
TABLE X1.4 Running Clearances

<table>
<thead>
<tr>
<th>Shaft Size</th>
<th>Clearance, min</th>
</tr>
</thead>
<tbody>
<tr>
<td>in.</td>
<td>mm</td>
</tr>
<tr>
<td>mm</td>
<td>in.</td>
</tr>
<tr>
<td>Up to 0.760</td>
<td>up to 19.31</td>
</tr>
<tr>
<td>0.761 to 1.510</td>
<td>19.32 to 38.36</td>
</tr>
<tr>
<td>1.511 to 2.510</td>
<td>38.37 to 63.76</td>
</tr>
<tr>
<td>Over 2.510</td>
<td>over 63.76</td>
</tr>
</tbody>
</table>

TABLE X1.5 Flange and Thrust Bearings Diameter and Thickness Tolerances

**Flange Bearings, Flange Diameter Tolerances**

<table>
<thead>
<tr>
<th>Diameter Range</th>
<th>Standard</th>
<th>Special</th>
</tr>
</thead>
<tbody>
<tr>
<td>in.</td>
<td>mm</td>
<td>in.</td>
</tr>
<tr>
<td>0 to 1 1/2</td>
<td>0 to 38</td>
<td>±0.005  ±0.13 ±0.0025 ±0.06</td>
</tr>
<tr>
<td>Over 1 1/2 to 3</td>
<td>39 to 76</td>
<td>±0.010  ±0.25 ±0.005 ±0.13</td>
</tr>
<tr>
<td>Over 3 to 6</td>
<td>77 to 152</td>
<td>±0.025  ±0.64 ±0.010 ±0.25</td>
</tr>
</tbody>
</table>

**Flange Bearings, Flange Thickness Tolerances**

<table>
<thead>
<tr>
<th>Diameter Range</th>
<th>Standard</th>
<th>Special</th>
</tr>
</thead>
<tbody>
<tr>
<td>in.</td>
<td>mm</td>
<td>in.</td>
</tr>
<tr>
<td>0 to 1 1/2</td>
<td>0 to 38</td>
<td>±0.005  ±0.13 ±0.0025 ±0.06</td>
</tr>
<tr>
<td>Over 1 1/2 to 3</td>
<td>39 to 76</td>
<td>±0.010  ±0.25 ±0.007 ±0.18</td>
</tr>
<tr>
<td>Over 3 to 6</td>
<td>77 to 152</td>
<td>±0.015  ±0.38 ±0.010 ±0.25</td>
</tr>
</tbody>
</table>

**Thrust Bearings (1/4 in. (6.35 mm) Thickness Tolerances, All Diameters)**

<table>
<thead>
<tr>
<th>Diameter Range</th>
<th>Standard</th>
<th>Special</th>
</tr>
</thead>
<tbody>
<tr>
<td>in.</td>
<td>mm</td>
<td>in.</td>
</tr>
<tr>
<td>±0.005</td>
<td>±0.13</td>
<td>±0.0025 ±0.06</td>
</tr>
</tbody>
</table>

**Parallelism on Faces, max**

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*Standard and special tolerances are specified for diameters, thickness, and parallelism. Special tolerances should not be specified unless required since they require additional or secondary operations and, therefore, are costlier.

*Outside diameter tolerances same as for flange bearings.*

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Standard Specification for Materials for Ferrous Powder Metallurgy (P/M) Structural Parts

This standard is issued under the fixed designation B 783; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers a variety of ferrous P/M structural materials and includes a classification system or material designation code. The classification system used in this specification includes chemical composition, minimum tensile; 0.2% offset yield strength for as-sintered materials and minimum ultimate tensile strength for heat-treated materials (sinter hardened or quenched and tempered). It also contains minimum density and maximum coercive field strength requirements for iron-phosphorus materials. Material classification is governed by the designation code which is explained in Appendix X1. The data provided display typical mechanical properties achieved under commercial manufacturing procedures. Physical and mechanical property performance characteristics can change as a result of subsequent processing steps beyond those designated in this standard. These changes could improve or degrade the properties.

1.2 Property values stated in inch-pound units are the standard. Conversion factors to SI units may be approximate.

2. Referenced Documents

2.1 ASTM Standards:
- B 243 Terminology of Powder Metallurgy
- B 328 Test Method for Density, Oil Content, and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings
- B 528 Test Method for Transverse Rupture Strength of Metal Powder Specimens
- E 8 Test Methods for Tension Testing of Metallic Materials
- E 1019 Test Methods for Determination of Carbon, Nitrogen, and Oxygen in Iron, Nickel, and Cobalt Alloys

2.2 Other Standard:
- MPIF Standard 35 Materials Standard for P/M Structural Parts

3. Terminology

3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243. Additional descriptive information is available in the Related Materials section of Vol 02.05 of the Annual Book of ASTM Standards.

4. Ordering Information

4.1 Materials for parts conforming to this specification shall be ordered by material designation code.

4.2 Orders for parts under this specification may include the following information:
- Certification, if required (see Section 11),
- Test methods and mechanical properties other than strength (see 8.2 and 8.3),
- Density (see 7.1),
- Porosity or oil content (see 7.2), and
- Special packaging if required.

5. Materials and Manufacture

5.1 Structural parts shall be made by pressing and sintering metal powders with or without subsequent heat treating. Parts may also be made by repressing or repressing and resintering sintered parts, if necessary, with or without subsequent heat treatment to produce finished parts conforming to the requirements of this specification.

6. Chemical Composition

6.1 The material shall conform to the requirements of Table 1.

6.2 Chemical analysis, if required, shall be made by methods agreed upon by the producer and the user.

6.3 Various analytical test methods are used to determine the chemical composition (see ASTM standards for the appropriate test methods) of P/M materials. Combustion-infra-red

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1 This specification is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.05 on Structural Parts.


3 Available from MPIF, 105 College Road East, Princeton, NJ 08540.
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absorption and inert gas fusion methods (Test Methods E 1019) are used for the specific elements of carbon, nitrogen, oxygen and sulfur.

6.4 The Chemical Composition Requirements Table (Table 1) designates the limits of metallurgically combined carbon for each alloy. The combined carbon level can be estimated metallographically for sintered P/M steels. When a clear pearlite to ferrite ratio cannot be estimated metallographically, total carbon can be determined using analytical methods (Test Methods E 1019). This would include very low carbon levels (<0.08 %), heat treated steels and materials made from prealloyed base powders or diffusion alloyed powders. When reporting carbon levels, the report should identify whether the carbon is metallurgically combined carbon or total carbon and the test method should be identified. While total carbon will approximate the combined carbon in many materials, free graphite and other carbonaceous material will raise the total carbon level above the level of combined carbon, possibly causing the total carbon content to exceed the combined carbon level specified for the material.

7. Physical Properties

7.1 Density:

7.1.1 The user and producer may agree upon a minimum average density for the part or minimum densities for specific regions of the part, or both, except soft magnetic materials, which require a minimum average density as part of the material specification.

7.1.2 Density shall be determined in accordance with Test Method B 328.

7.2 Porosity:

7.2.1 The producer and the user may also agree upon a minimum volume oil content for parts that are to be self-lubricating.

7.2.2 Porosity or oil content, or both, shall be determined in accordance with Test Method B 328.

7.2.3 The producer and the user may agree upon a functional test for porosity in parts that are to be self-lubricating, or for permeability where fluid flow must be restricted.

8. Mechanical Properties

8.1 The guaranteed properties shown in Tables 2-11 are included in the suffix of the material designation code. The code is adopted from MPIF Standard 35. All tensile strengths are read as 10^3 psi, and are defined as the 0.2 % offset yield strength for as-sintered materials and the ultimate tensile strength for heat-treated materials (sinter hardened or quenched and tempered). Iron-phosphorus materials (Table 3) contain an alphanumeric suffix and are an exception to this rule. The iron-phosphorus suffix is related to the minimum density and

### TABLE 1

Continued

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<th>Molybdenum</th>
<th>Chromium</th>
<th>Manganese</th>
<th>Silicon</th>
<th>Sulfur</th>
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<th>Nitrogen</th>
<th>Columbium</th>
<th>Oxygen</th>
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<td>0.0</td>
<td>0.0</td>
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<td>Bal.</td>
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<td>0.0</td>
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<td>...</td>
<td>...</td>
<td>2.0</td>
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</tbody>
</table>

A Carbon, on basis of iron only, may be a metallographic estimate.

B At least 1 % of the nickel is admixed as elemental powder.

C At least 2 % of the nickel is admixed as elemental powder.

D LE = L grade with extended chemical composition.

8.1.1 Materials that are heat treated (sinter-hardened or quenched and tempered) have the numeric value followed by HT in the suffix.

8.2 The producer and the user should agree upon the method to be used to verify the minimum strength characteristics of the finished parts. Since it is usually impossible to machine tensile test specimens from these parts, alternative strength tests are advisable. An example would be measuring the force needed to break teeth off a gear with the gear properly fixtured.

8.3 If the tensile properties of the materials are required, standard test bars shall be molded from the same mixed powder lot, at the density of a critical region in the part, and processed along with the parts. When a P/M part has a larger ruling section than the test bar being used, the test bar may not be representative of the part. The following procedures are listed with the preferred method first.

8.3.1 Transverse rupture strength (see Test Method B 528) can be related to the minimum tensile strength by the ratio of typical transverse rupture strength to typical tensile strength at the same density as the part, as shown in, or interpolated from the tables contained in Appendix X1.

8.3.2 For as-sintered material, flat unmachined tension test specimens (see Test Methods E 8) should be used for determination of 0.2 % offset yield strength.

8.3.3 For determining the tensile strength of heat-treated (sinter-hardened or quenched and tempered) material, round

### Table 2: Minimum Tensile Strength for Iron and Carbon Steel

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Minimum Strength</th>
</tr>
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<td>Yield 10^3 psi</td>
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<td>-20</td>
<td>...</td>
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<tr>
<td>F-0005-15</td>
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<tr>
<td>-20</td>
<td>...</td>
</tr>
<tr>
<td>-25</td>
<td>...</td>
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### Table 3: Minimum Density and Maximum Coercive Field Strength for Iron-Phosphorus

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<th>Maximum Coercive Field Strength Oe</th>
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<tr>
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<td>1.7</td>
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### Table 4: Minimum Tensile Strength for Copper Infiltrated Iron and Steel

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<td>-24</td>
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<td>FC-0205-30</td>
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</table>

^1 10^3 psi = 6.895 MPa (6.895 N/mm²)

A These materials are frequently used in magnetic applications and are specified with minimum density and maximum coercive field strength. One oersted is equal to 79.6 A/m in SI units. Typical magnetic properties can be found in Specification A 839.
test bars should be machined from specially molded, assintered bars because heat treated, unmachined specimens yield lower values. The machined tension test specimens (see Test Methods E 8) shall be heat-treated with the production parts.

9. Sampling

9.1 Lot—Unless otherwise specified, a lot shall consist of parts of the same form and dimensions made from powders of the same composition, molded, and processed under the same conditions, and submitted for inspection at one time.

9.2 Chemical Analysis—When requested on the purchase order, at least one sample for chemical analysis shall be taken from each lot. The analysis shall be performed by a mutually agreed upon method.

9.3 Mechanical Tests—The producer and the user shall agree on a representative number of specimens for mechanical tests.

10. Rejection and Rehearing

10.1 Parts that fail to conform to the requirements of this specification may be rejected. Rejection should be reported to the producer or supplier promptly and in writing.

11. Certification

11.1 When specified in the purchase order or contract, a producer’s certification shall be furnished to the user that the parts were manufactured, sampled, tested, and inspected in accordance with this specification and have been found to meet the requirements. When specified in the purchase order or contract, a report of the test results shall be furnished.

12. Keywords

12.1 ferrous powder metallurgy; ferrous structural parts; powder metallurgy (P/M); structural parts
### TABLE 9 Minimum Tensile Strength for Diffusion Alloyed Steel

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*10^3 psi = 6.895 MPa (6.895 N/mm²)*

### TABLE 10 Minimum Tensile Strength for Austenitic Stainless Steel

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</thead>
<tbody>
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<td>Minimum Strength</td>
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<tr>
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<td>Yield 10^3 psi</td>
</tr>
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*10^3 psi = 6.895 MPa (6.895 N/mm²)*

TABLE 11 Minimum Tensile Strength for Ferritic and Martensitic Stainless Steel

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Minimum Strength</th>
<th>Minimum Elongation</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>Yield 10^3 psi</td>
<td>Ultimate 10^3 psi</td>
</tr>
<tr>
<td>SS-410-90HT</td>
<td>...</td>
<td>90</td>
</tr>
<tr>
<td>SS-410L-20</td>
<td>20</td>
<td>10.0</td>
</tr>
<tr>
<td>SS-430N2-28</td>
<td>28</td>
<td>...</td>
</tr>
<tr>
<td>SS-430L-24</td>
<td>24</td>
<td>...</td>
</tr>
<tr>
<td>SS-434N2-28</td>
<td>28</td>
<td>...</td>
</tr>
<tr>
<td>SS-434L-24</td>
<td>24</td>
<td>...</td>
</tr>
</tbody>
</table>

\(^{a} 10^3 \text{ psi} = 6.895 \text{ MPa} (6.895 \text{ N/mm}^2)\)


APPENDIX

(Nonmandatory Information)

X1. USE OF THIS SPECIFICATION

X1.1 PM/Material Code Designation:

X1.1.1 The P/M material code designation or identifying code for structural P/M parts defines a specific material as to chemical composition and minimum strength expressed in 10^3 psi (6.895 MPa). For example, FC-0208-60 is a P/M copper steel material containing nominally 2 % copper and 0.8 % combined carbon possessing a minimum yield strength of 60 \times 10^3 psi (60 000 psi) in the as-sintered condition.

X1.1.2 The system offers a convenient means for designating both the chemical composition and minimum strength value of any standard P/M material. The density is given for each standard material as one of the typical values and is no

### TABLE X1.1 Iron and Carbon Steel

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Minimum Strength</th>
<th>Tensile Properties</th>
<th>Un-notched Charpy Impact</th>
<th>Compressive Yield Strength</th>
<th>Hardness</th>
<th>Microindentation (conventional)</th>
<th>Fatigue Limit</th>
<th>Density</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Yield 10^3 psi</td>
<td>Ultimate Strength</td>
<td>Elongation (0.2 %)</td>
<td>Young’s Modulus</td>
<td>Poisson’s Ratio</td>
<td>Transverse Rupture Strength</td>
<td>Rockwell</td>
<td>g/cm³</td>
</tr>
<tr>
<td>F-0000-10</td>
<td>10</td>
<td>18</td>
<td>13</td>
<td>1.5</td>
<td>15.0</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>-15</td>
<td>15</td>
<td>18</td>
<td>25</td>
<td>2.5</td>
<td>17.5</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>-20</td>
<td>20</td>
<td>...</td>
<td>38</td>
<td>7.0</td>
<td>23.5</td>
<td>0.28</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>F-0005-15</td>
<td>15</td>
<td>24</td>
<td>18</td>
<td>&lt;1.0</td>
<td>15.0</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>-20</td>
<td>20</td>
<td>...</td>
<td>32</td>
<td>1.0</td>
<td>16.5</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>-25</td>
<td>25</td>
<td>...</td>
<td>38</td>
<td>1.5</td>
<td>19.5</td>
<td>0.27</td>
<td>0.27</td>
<td>0.27</td>
</tr>
<tr>
<td>F-0005-50HT</td>
<td>...</td>
<td>50</td>
<td>60</td>
<td>&lt;0.5</td>
<td>16.5</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>-60HT</td>
<td>...</td>
<td>60</td>
<td>70</td>
<td>&lt;0.5</td>
<td>18.5</td>
<td>0.27</td>
<td>0.27</td>
<td>0.27</td>
</tr>
<tr>
<td>-70HT</td>
<td>...</td>
<td>70</td>
<td>80</td>
<td>&lt;0.5</td>
<td>20.5</td>
<td>0.27</td>
<td>0.27</td>
<td>0.27</td>
</tr>
<tr>
<td>F-0008-20</td>
<td>20</td>
<td>29</td>
<td>25</td>
<td>&lt;0.5</td>
<td>12.5</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>-25</td>
<td>25</td>
<td>35</td>
<td>30</td>
<td>&lt;0.5</td>
<td>16.0</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>-30</td>
<td>30</td>
<td>42</td>
<td>35</td>
<td>&lt;1.0</td>
<td>18.5</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>F-0008-55HT</td>
<td>...</td>
<td>55</td>
<td>65</td>
<td>&lt;0.5</td>
<td>16.5</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>-65HT</td>
<td>...</td>
<td>65</td>
<td>75</td>
<td>&lt;0.5</td>
<td>16.5</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>-75HT</td>
<td>...</td>
<td>75</td>
<td>85</td>
<td>&lt;0.5</td>
<td>19.5</td>
<td>0.27</td>
<td>0.27</td>
<td>0.27</td>
</tr>
<tr>
<td>-85HT</td>
<td>...</td>
<td>85</td>
<td>95</td>
<td>&lt;0.5</td>
<td>21.5</td>
<td>0.27</td>
<td>0.27</td>
<td>0.27</td>
</tr>
</tbody>
</table>

\(^{a} \text{Suffix numbers represent minimum strength values in } 10^3 \text{ psi; yield in the as-sintered condition and ultimate in the heat-treated condition.}\)

\(^{b} \text{Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.}\)

\(^{c} \text{Tempering temperature for heat-treated (HT) materials: } 350°F (177°C).\)

\(^{d} \text{Yield and ultimate tensile strength are approximately the same for heat-treated materials.}\)

N/D—Not Determined for the purposes of this standard.
longer a requirement of the specification, with the exception of the iron-phosphorus materials as detailed in Tables X1.3 and X1.4.

Table X1.2 Iron and Carbon Steel (SI)

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Minimum Strength</th>
<th>Tensile Properties</th>
<th>Elastic Constants</th>
<th>Unnotched Charpy Impact Energy</th>
<th>Transverse Rupture Strength</th>
<th>Compressive Yield Strength</th>
<th>Hardness</th>
<th>Fatigue Limit</th>
<th>Density</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Yield</td>
<td>Ultimate</td>
<td>Yield Strength</td>
<td>Elongation (in 25.4 mm)</td>
<td>Young’s Modulus</td>
<td>Poisson’s Ratio</td>
<td>J</td>
<td></td>
<td>g/cm³</td>
</tr>
<tr>
<td></td>
<td>Ultimate</td>
<td>Strength</td>
<td>(0.2 %)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>MPa</td>
<td>MPa</td>
<td>%</td>
<td>GPa</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>F-0000-10</td>
<td>70</td>
<td>...</td>
<td>120</td>
<td>105</td>
<td>105</td>
<td>0.25</td>
<td>25</td>
<td>110</td>
<td>40</td>
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<tr>
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<td>100</td>
<td>...</td>
<td>120</td>
<td>120</td>
<td>8</td>
<td>340</td>
<td>120</td>
<td>60</td>
<td>N/D</td>
</tr>
<tr>
<td>-20</td>
<td>140</td>
<td>...</td>
<td>260</td>
<td>170</td>
<td>160</td>
<td>28</td>
<td>47</td>
<td>660</td>
<td>80</td>
</tr>
<tr>
<td>F-0005-15</td>
<td>100</td>
<td>...</td>
<td>170</td>
<td>105</td>
<td>105</td>
<td>0.25</td>
<td>4</td>
<td>330</td>
<td>125</td>
</tr>
<tr>
<td>-20</td>
<td>140</td>
<td>...</td>
<td>220</td>
<td>115</td>
<td>5</td>
<td>440</td>
<td>160</td>
<td>40</td>
<td>N/D</td>
</tr>
<tr>
<td>-25</td>
<td>170</td>
<td>...</td>
<td>260</td>
<td>135</td>
<td>7</td>
<td>520</td>
<td>190</td>
<td>55</td>
<td>100</td>
</tr>
<tr>
<td>F-0005-50HT</td>
<td>410</td>
<td>...</td>
<td>410</td>
<td>&lt;0.5</td>
<td>115</td>
<td>0.27</td>
<td>7</td>
<td>720</td>
<td>300</td>
</tr>
<tr>
<td>-60HT</td>
<td>410</td>
<td>...</td>
<td>410</td>
<td>&lt;0.5</td>
<td>115</td>
<td>0.27</td>
<td>5</td>
<td>830</td>
<td>360</td>
</tr>
<tr>
<td>-70HT</td>
<td>480</td>
<td>...</td>
<td>480</td>
<td>&lt;0.5</td>
<td>140</td>
<td>0.27</td>
<td>5</td>
<td>970</td>
<td>420</td>
</tr>
<tr>
<td>F-0008-20</td>
<td>200</td>
<td>...</td>
<td>200</td>
<td>&lt;0.5</td>
<td>85</td>
<td>0.25</td>
<td>3</td>
<td>350</td>
<td>190</td>
</tr>
<tr>
<td>-25</td>
<td>240</td>
<td>...</td>
<td>240</td>
<td>&lt;0.5</td>
<td>110</td>
<td>0.25</td>
<td>4</td>
<td>420</td>
<td>210</td>
</tr>
<tr>
<td>-30</td>
<td>290</td>
<td>...</td>
<td>290</td>
<td>&lt;1.0</td>
<td>115</td>
<td>0.25</td>
<td>5</td>
<td>510</td>
<td>210</td>
</tr>
<tr>
<td>-35</td>
<td>390</td>
<td>...</td>
<td>390</td>
<td>1.0</td>
<td>140</td>
<td>0.27</td>
<td>7</td>
<td>690</td>
<td>250</td>
</tr>
<tr>
<td>F-0008-55HT</td>
<td>450</td>
<td>...</td>
<td>450</td>
<td>&lt;0.5</td>
<td>115</td>
<td>0.25</td>
<td>4</td>
<td>690</td>
<td>480</td>
</tr>
<tr>
<td>-65HT</td>
<td>520</td>
<td>...</td>
<td>520</td>
<td>&lt;0.5</td>
<td>115</td>
<td>0.25</td>
<td>5</td>
<td>790</td>
<td>550</td>
</tr>
<tr>
<td>-75HT</td>
<td>590</td>
<td>...</td>
<td>590</td>
<td>&lt;0.5</td>
<td>135</td>
<td>0.27</td>
<td>6</td>
<td>900</td>
<td>620</td>
</tr>
</tbody>
</table>

A Suffix numbers represent minimum strength values in 10³ psi; yield in the as-sintered condition and ultimate in the heat-treated condition.

TABLE X1.3 Iron-Phosphorus

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>g/cm³</td>
<td>Oe</td>
<td>10⁵ psi</td>
<td>10³ psi</td>
<td>%</td>
<td>10⁶ psi</td>
<td>ft-lbf</td>
<td>Rockwell</td>
</tr>
<tr>
<td>FY-4500</td>
<td>-20V</td>
<td>6.7</td>
<td>2.0</td>
<td>40.0</td>
<td>30.0</td>
<td>5</td>
<td>18.5</td>
<td>0.27</td>
</tr>
<tr>
<td></td>
<td>-20W</td>
<td>6.9</td>
<td>2.0</td>
<td>45.0</td>
<td>32.0</td>
<td>7</td>
<td>20.5</td>
<td>0.27</td>
</tr>
<tr>
<td></td>
<td>-17W</td>
<td>6.9</td>
<td>1.7</td>
<td>45.0</td>
<td>32.0</td>
<td>10</td>
<td>20.5</td>
<td>0.27</td>
</tr>
<tr>
<td></td>
<td>-20X</td>
<td>7.1</td>
<td>2.0</td>
<td>50.0</td>
<td>35.0</td>
<td>12</td>
<td>22.5</td>
<td>0.28</td>
</tr>
<tr>
<td>-20Y</td>
<td>7.3</td>
<td>2.0</td>
<td>55.0</td>
<td>38.0</td>
<td>9</td>
<td>24.5</td>
<td>0.28</td>
<td>65 HRB</td>
</tr>
<tr>
<td>-17Y</td>
<td>7.3</td>
<td>1.7</td>
<td>60.0</td>
<td>41.0</td>
<td>15</td>
<td>24.5</td>
<td>0.28</td>
<td>65 HRB</td>
</tr>
<tr>
<td>FY-8000</td>
<td>-17V</td>
<td>6.7</td>
<td>1.7</td>
<td>48.0</td>
<td>40.0</td>
<td>2</td>
<td>18.5</td>
<td>0.27</td>
</tr>
<tr>
<td></td>
<td>-17W</td>
<td>6.9</td>
<td>1.7</td>
<td>50.0</td>
<td>45.0</td>
<td>3</td>
<td>20.5</td>
<td>0.27</td>
</tr>
<tr>
<td></td>
<td>-15W</td>
<td>6.9</td>
<td>1.5</td>
<td>53.0</td>
<td>45.0</td>
<td>4</td>
<td>20.5</td>
<td>0.27</td>
</tr>
<tr>
<td></td>
<td>-17X</td>
<td>7.1</td>
<td>1.7</td>
<td>55.0</td>
<td>50.0</td>
<td>3</td>
<td>22.5</td>
<td>0.28</td>
</tr>
<tr>
<td></td>
<td>-15X</td>
<td>7.1</td>
<td>1.5</td>
<td>57.0</td>
<td>48.0</td>
<td>4</td>
<td>22.5</td>
<td>0.28</td>
</tr>
<tr>
<td></td>
<td>-15Y</td>
<td>7.3</td>
<td>1.5</td>
<td>62.0</td>
<td>53.0</td>
<td>4</td>
<td>24.5</td>
<td>0.28</td>
</tr>
</tbody>
</table>

A Suffix numbers represent maximum coercive field strength values (oersteds × 10³), the letter suffix indicates the minimum density in g/cm³.

B Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.

C Additional data in preparation will appear in subsequent editions of this standard.

N/D—Not Determined for the purposes of this standard.
material designation coding system is intended for use only with such materials and should not be used to create non-standard materials. However, the use of designations such as FC-0208 or FN-0205 to denote materials of a specified composition is permitted. The explanatory notes, property values, and other contents of this standard have no application to any other materials.

X1.1.4 In the coding system, the prefix letters denote the general type of material. For example, the prefix FC represents iron (F) and copper (C), which is known as iron-copper and copper steel. The prefix letter codes are as follows:

- **C**—Copper.
- **F**—Iron.
- **FY**—Iron-phosphorus.
- **FC**—Iron-copper and Copper Steel.
- **FN**—Iron-nickel and Nickel Steel.
- **FX**—Infiltrated Iron or Steel.
- **FL**—Prealloyed Ferrous material except Stainless Steel.
- **FD**—Diffusion Alloyed Steel.
- **N**—Nickel.
- **SS**—Stainless Steel.

X1.2 Prefix and Four-Digit Code:

X1.2.1 In ferrous materials, the major alloying elements (except combined carbon) are included in the prefix letter code. Other elements are excluded from the code but are represented in the chemical composition that appears with each standard material. The first two digits of the four-digit code indicate the percentage of the major alloying constituent present. In the iron-phosphorus material, the first two digits represent the percentage of phosphorus multiplied by 100 to more accurately indicate the nominal amount of phosphorus.

X1.2.2 Combined carbon content in ferrous materials is designated by the last two numbers in the four-digit series. There are three carbon ranges designated as follows:

<table>
<thead>
<tr>
<th>Carbon Ranges Code Designation</th>
<th>Range Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>A 00</td>
<td>0.0 % to 0.3 %</td>
</tr>
<tr>
<td>B 05</td>
<td>0.3 % to 0.6 %</td>
</tr>
<tr>
<td>C 08</td>
<td>0.6 % to 0.9 %</td>
</tr>
</tbody>
</table>

A: Iron-phosphorus material carbon range is 0.00 % to 0.03 % when designated as “00.”
B: Carbon range for the low alloy series is 0.4 % to 0.7 % when designated as “05.”

X1.3 Suffix Digit Code—The two- or three-digit suffix represents the minimum strength value, expressed in 10^3 psi (6.895 MPa) that the user can expect from the P/M material possessing that chemical composition. In the as-sintered condition the strength is tensile yield; in the heat-treated condition, it is ultimate tensile (see Minimum Value, Tables X1.1-X1.20). An exception to this is found in the soft magnetic “FY” material in which the suffix represents the minimum density and maximum coercive field strength. The suffix number represents the maximum coercive field strength (ten times the value in oersteds) instead of the yield or tensile strength. For example FY-4500-20W would represent an iron-0.45 % phosphorus alloy.
### TABLE X1.5 Copper Infiltrated Iron and Steel

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Minimum Strength</th>
<th>Tensile Properties</th>
<th>Elastic Constants</th>
<th>Unnotched Charpy Impact Energy</th>
<th>Transverse Rupture Strength</th>
<th>Compressive Yield Strength (0.1 %)</th>
<th>Hardness</th>
<th>Fatigue Limit</th>
<th>Survival</th>
<th>Density</th>
</tr>
</thead>
<tbody>
<tr>
<td>FX-1000-25</td>
<td>25</td>
<td>32</td>
<td>7.0</td>
<td>23.5</td>
<td>0.28</td>
<td>25.0</td>
<td>132</td>
<td>33</td>
<td>65 HRC</td>
<td>7.3</td>
</tr>
<tr>
<td>FX-1005-40</td>
<td>40</td>
<td>50</td>
<td>4.0</td>
<td>23.5</td>
<td>0.28</td>
<td>13.0</td>
<td>158</td>
<td>53</td>
<td>62 HRC</td>
<td>7.3</td>
</tr>
<tr>
<td>FX-1005-110HT</td>
<td>...</td>
<td>120</td>
<td>7</td>
<td>23.5</td>
<td>0.28</td>
<td>7.0</td>
<td>210</td>
<td>110</td>
<td>38 HRC</td>
<td>7.3</td>
</tr>
<tr>
<td>FX-1008-50</td>
<td>50</td>
<td>87</td>
<td>60</td>
<td>3.0</td>
<td>23.5</td>
<td>10.0</td>
<td>166</td>
<td>71</td>
<td>89 HRC</td>
<td>7.3</td>
</tr>
<tr>
<td>FX-1008-110HT</td>
<td>...</td>
<td>120</td>
<td>0.5</td>
<td>23.5</td>
<td>0.28</td>
<td>6.5</td>
<td>189</td>
<td>115</td>
<td>43 HRC</td>
<td>7.3</td>
</tr>
<tr>
<td>FX-2000-25</td>
<td>25</td>
<td>46</td>
<td>37</td>
<td>21.0</td>
<td>0.24</td>
<td>15.0</td>
<td>144</td>
<td>41</td>
<td>66 HRC</td>
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<td>74</td>
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</table>

- Suffix numbers represent minimum strength values in 10^3 psi; yield in the as-sintered condition and ultimate in the heat-treated condition.
- Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.
- Tempering temperature for heat-treated (HT) materials: 350°F (177°C).
- Yield and ultimate tensile strength are approximately the same for heat-treated materials.
- N/D—Not Determined for the purposes of this standard.

### TABLE X1.6 Copper Infiltrated Iron and Steel (SI)

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Minimum Strength</th>
<th>Tensile Properties</th>
<th>Elastic Constants</th>
<th>Unnotched Charpy Impact Energy</th>
<th>Transverse Rupture Strength</th>
<th>Compressive Yield Strength (0.1 %)</th>
<th>Hardness</th>
<th>Fatigue Limit</th>
<th>Survival</th>
<th>Density</th>
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</table>

- Suffix numbers represent minimum strength values in 10^3 psi (see page 2); yield in the as-sintered condition and ultimate in the heat-treated condition.
- Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.
- Yield and ultimate tensile strength are approximately the same for heat-treated materials (see page 3).
- N/D—Not Determined for the purposes of this standard.

### X1.4 Suffix Letter Code—When the code designation HT appears after the suffix digits it is understood that the P/M material specified has been heat-treated (sinter hardened or quenched and tempered) and that the strength represented is ultimate tensile in 10^3 psi (6.895 MPa (6.895 N/mm^2)). The letter suffix in the iron-phosphorus materials represents the density. The density is represented alphabetically by the following letters:

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<th>Suffix Letter</th>
<th>Minimum Density (g/cm^3)</th>
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Note: X1.1—The example used in X1.3 (FY-4500-20W) now becomes an iron-0.45 % phosphorus alloy with a minimum density of 6.9 g/cm^3.
### P/M Material Properties

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Minimum Strength*&lt;sup&gt;A&lt;/sup&gt;,&lt;sup&gt;C&lt;/sup&gt;</th>
<th>Tensile Properties</th>
<th>Elastic Constants</th>
<th>Unnotched Charpy Impact Energy</th>
<th>Transverse Rupture Strength</th>
<th>Compressive Yield Strength (0.1 %)</th>
<th>Hardness</th>
<th>Fatigue Limit 90 % Survival</th>
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<td>Ultimate Strength</td>
<td>Yield Strength (0.2 %)</td>
<td>Elongation</td>
<td>Young’s Modulus</td>
<td>Poisson’s Ratio</td>
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</table>

*Suffix numbers represent minimum strength values in 10<sup>3</sup> psi; yield in the as-sintered condition and ultimate in the heat-treated condition.

Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.

*Tempering temperature for heat-treated (HT) materials: 350°F (177°C).

Yield and ultimate tensile strength are approximately the same for heat-treated materials.

N/D—Not Determined for the purposes of this standard.

**Note 1:** 10<sup>3</sup> psi = 6.895 MPa (6.895 N/mm<sup>2</sup>).

**Note 2:** 1 in. = 25.4 mm.

**Note 3:** 1 ft-lbf = 1.356 J.

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X1.5 Data Source—Information used in compiling this specification was contributed by the membership of ASTM Committee B09 on Metal Powders and Metal Powder Products and the MPIF Standards Committee. These technical data are on file at MPIF Headquarters, Princeton, NJ, and are reproduced in this specification with the permission of the Metal Powder Industries Federation.
<table>
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<tr>
<th>Material Designation Code</th>
<th>Minimum Strength&lt;sup&gt;a,c&lt;/sup&gt;</th>
<th>Tensile Properties</th>
<th>Elastic Constants</th>
<th>Unnotched Charpy Impact Energy</th>
<th>Transverse Rupture Strength</th>
<th>Compressive Yield Strength (0.1 %)</th>
<th>Hardness</th>
<th>Fatigue Limit</th>
<th>90 % Survival</th>
<th>Density&lt;sup&gt;b&lt;/sup&gt;</th>
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<sup>a</sup> Suffix numbers represent minimum strength values in 10<sup>3</sup> psi; yield in the as-sintered condition and ultimate in the heat-treated condition.

<sup>b</sup> Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.

<sup>c</sup> Tempering temperature for heat-treated (HT) materials: 350°F (177°C).

<sup>d</sup> Yield and ultimate tensile strength are approximately the same for heat-treated materials.

N/D—Not Determined for the purposes of this standard.
TABLE X1.9 Iron-Nickel and Nickel Steel

NOTE 1—10^3 psi = 6.895 MPa (6.895 N/mm²).
NOTE 2—1 in. = 25.4 mm.
NOTE 3—1 ft-lbf = 1.356 J.

### P/M Material Properties

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<sup>a</sup> Suffix numbers represent minimum strength values in 10^3 psi; yield in the as-sintered condition and ultimate in the heat-treated condition.
<sup>b</sup> Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.
<sup>c</sup> Tempering temperature for heat-treated (HT) materials: 500°F (260°C).
<sup>d</sup> Yield and ultimate tensile strength are approximately the same for heat-treated materials.
N/D—Not Determined for the purposes of this standard.
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<sup>a</sup> Suffix numbers represent minimum strength values in 10<sup>3</sup> psi; yield in the as-sintered condition and ultimate in the heat-treated condition.

<sup>b</sup> Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.

<sup>c</sup> Tempering temperature for heat-treated (HT) materials: 500°F (260°C).

<sup>d</sup> Yield and ultimate tensile strength are approximately the same for heat-treated materials.

N/D—Not Determined for the purposes of this standard.
### Table X1.11 Low Alloy Steel

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<th>Compressive Yield Strength (0.1 %)</th>
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A Suffix numbers represent minimum strength values in 10³ psi; yield in the as-sintered condition and ultimate in the heat-treated condition.

B Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.

C Tempering temperature for heat-treated (HT) materials: 350°F (177°C).

D Yield and ultimate tensile strength are approximately the same for heat-treated materials.

N/D—Not Determined for the purposes of this standard.

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Note 1—10³ psi = 6.895 MPa (6.895 N/mm²).

Note 2—1 in. = 25.4 mm.

Note 3—1 ft-lbf = 1.356 J.
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\(^a\) Suffix numbers represent minimum strength values in 10\(^3\) psi; yield in the as-sintered condition and ultimate in the heat-treated condition.
\(^b\) Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.
\(^c\) Tempering temperature for heat-treated (HT) materials: 177°C (350°F).
\(^d\) Yield and ultimate tensile strength are approximately the same for heat-treated materials.
N/D—Not Determined for the purposes of this standard.
### TABLE X1.13 Sinter Hardened Steel

**Note 1**—10³ psi = 6.895 MPa (6.895 N/mm²).

**Note 2**—1 in. = 25.4 mm.

**Note 3**—1 ft-lbf = 1.356 J.

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Minimum Strength</th>
<th>Tensile Properties</th>
<th>Elastic Constants</th>
<th>Fatigue Limit 90 % Survival</th>
<th>Density</th>
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¹ Suffix numbers represent minimum strength values in 10³ psi; yield in the as-sintered condition and ultimate in the heat-treated condition.
² Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.
³ Tempering temperature for heat-treated (HT) materials: 177°C (350°F).
⁴ Yield and ultimate tensile strength are approximately the same for heat-treated materials.
⁵ Additional data in preparation will appear in subsequent editions of this standard.

### TABLE X1.14 Sinter Hardened Steel (SI)

**Note 1**—10³ psi = 6.895 MPa (6.895 N/mm²).

**Note 2**—1 in. = 25.4 mm.

**Note 3**—1 ft-lbf = 1.356 J.

<table>
<thead>
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¹ Suffix numbers represent minimum strength values in 10³ psi; yield in the as-sintered condition and ultimate in the heat-treated condition.
² Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.
³ Tempering temperature for heat-treated (HT) materials: 177°C (350°F).
⁴ Yield and ultimate tensile strength are approximately the same for heat-treated materials.
⁵ Additional data in preparation will appear in subsequent editions of this standard.
### TABLE X1.15 Diffusion Allooided Steel

#### P/M Material Properties

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<th>Compressive Yield Strength (0.1 %)</th>
<th>Hardness</th>
<th>Fatigue Limit</th>
<th>90 % Survival(^E)</th>
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\(^A\) Suffix numbers represent minimum strength values in 10\(^3\) psi; yield in the as-sintered condition and ultimate in the heat-treated condition.

\(^B\) Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.

\(^C\) Alloys containing copper additions may have lower impact and fatigue limit as compared to the values in the data tables.

\(^D\) Tempering temperature for heat-treated (HT) materials: 177 °C (350 °F).

\(^E\) Yield and ultimate tensile strength are approximately the same for heat-treated materials.

N/D—Not Determined for the purposes of this standard.
TABLE X1.16 Diffusion Alloyed Steel (SI)

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<th>Material Designation Code</th>
<th>Minimum Strength[A,C]</th>
<th>Ultimate Strength</th>
<th>Yield Strength (0.2 %)</th>
<th>Elongation</th>
<th>Young’s Modulus</th>
<th>Poisson’s Ratio</th>
<th>Unnotched Charpy Impact Energy[C]</th>
<th>Transverse Rupture Strength</th>
<th>Compressive Yield Strength (0.1 %)</th>
<th>Hardness</th>
<th>Fatigue Limit 90 % Survival[D]</th>
<th>Density g/cm³</th>
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<td></td>
</tr>
<tr>
<td>-55</td>
<td>380</td>
<td>430</td>
<td>&lt;1.0</td>
<td>135</td>
<td>0.27</td>
<td>12</td>
<td>1070</td>
<td>430</td>
<td>83</td>
<td>N/D</td>
<td>230</td>
<td>6.90</td>
</tr>
<tr>
<td>-60</td>
<td>410</td>
<td>470</td>
<td>1.0</td>
<td>150</td>
<td>0.28</td>
<td>24</td>
<td>1380</td>
<td>500</td>
<td>90</td>
<td>260</td>
<td>7.20</td>
<td></td>
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<tr>
<td>-65</td>
<td>450</td>
<td>500</td>
<td>2.0</td>
<td>170</td>
<td>0.28</td>
<td>30</td>
<td>1590</td>
<td>550</td>
<td>95</td>
<td>320</td>
<td>7.40</td>
<td></td>
</tr>
</tbody>
</table>

A Suffix numbers represent minimum strength values in 10³ psi; yield in the as-sintered condition and ultimate in the heat-treated condition.

B Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.

C Temperatures for heat-treated (HT) materials: 177 °C (350 °F).

D Yield and ultimate tensile strength are approximately the same for heat-treated materials.

Alloys containing copper additions may have lower impact and fatigue limit as compared to the values in the data tables.

N/D—Not Determined for the purposes of this standard.
### TABLE X.17 Austenitic Stainless Steel

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Minimum Values&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Typical Values&lt;sup&gt;b&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Minimum Strength&lt;sup&gt;a&lt;/sup&gt;</td>
<td>Tensile Properties</td>
</tr>
<tr>
<td></td>
<td>Yield</td>
<td>Ultimate</td>
</tr>
<tr>
<td>SS-303N1-25</td>
<td>25 ...</td>
<td>0.0</td>
</tr>
<tr>
<td>SS-303N2-35</td>
<td>35 ...</td>
<td>3.0</td>
</tr>
<tr>
<td>SS-303N2-38</td>
<td>38 ...</td>
<td>6.0</td>
</tr>
<tr>
<td>SS-303L-12</td>
<td>12 ...</td>
<td>12.0</td>
</tr>
<tr>
<td>SS-303L-15</td>
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<td>15.0</td>
</tr>
<tr>
<td>SS-304N1-30</td>
<td>30 ...</td>
<td>0.0</td>
</tr>
<tr>
<td>SS-304N2-33</td>
<td>33 ...</td>
<td>5.0</td>
</tr>
<tr>
<td>SS-304N2-38</td>
<td>38 ...</td>
<td>8.0</td>
</tr>
<tr>
<td>SS-304H-20</td>
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<td>7.0</td>
</tr>
<tr>
<td>SS-304L-13</td>
<td>13 ...</td>
<td>15.0</td>
</tr>
<tr>
<td>SS-304L-18</td>
<td>18 ...</td>
<td>18.0</td>
</tr>
<tr>
<td>SS-316N1-25</td>
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<tr>
<td>SS-316N2-33</td>
<td>33 ...</td>
<td>5.0</td>
</tr>
<tr>
<td>SS-316N2-38</td>
<td>38 ...</td>
<td>8.0</td>
</tr>
<tr>
<td>SS-316H-20</td>
<td>20 ...</td>
<td>5.0</td>
</tr>
<tr>
<td>SS-316L-15</td>
<td>15 ...</td>
<td>12.0</td>
</tr>
<tr>
<td>SS-316L-22</td>
<td>22 ...</td>
<td>15.0</td>
</tr>
</tbody>
</table>

N1—Nitrogen alloyed. Good strength, low elongation.
N2—Nitrogen alloyed. High strength, medium elongation.
*—Sintered at 2350°F (1288°C) in dissociated ammonia
H—Low carbon. Lower strength, high elongation.
—Sintered at 2100°F (1149°C) in 100% hydrogen.
L—Low carbon. Lower strength, highest elongation.
Sintered at 2350°F (1288°C) in partial vacuum.
Cooled to avoid nitrogen absorption.

<sup>a</sup> Suffix numbers represent minimum strength values in 10<sup>3</sup> psi; yield in the as-sintered condition and ultimate in the heat-treated condition.
<sup>b</sup> Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.
<sup>c</sup> Additional data in preparation will appear in subsequent editions of this standard.

N/D—Not Determined for the purposes of this standard.
# Austenitic Stainless Steel (SI)

## P/M Material Properties

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Minimum Values&lt;sup&gt;A&lt;/sup&gt;</th>
<th>Typical Values&lt;sup&gt;B&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Minimum Strength&lt;sup&gt;A&lt;/sup&gt;</td>
<td>Tensile Properties</td>
</tr>
<tr>
<td></td>
<td>Yield Ultimate</td>
<td>Ultimate Strength</td>
</tr>
<tr>
<td>SS-303N1-25</td>
<td>170 0.0 270 220 0.5 105 0.25 5 590 260</td>
<td>82 HRB</td>
</tr>
<tr>
<td>SS-303N2-35</td>
<td>240 3.0 380 290 5.0 115 0.25 26 680 320</td>
<td>63 HRB</td>
</tr>
<tr>
<td>SS-303N2-38</td>
<td>260 6.0 470 310 10.0 140 0.27 47 N/D 320</td>
<td>70 HRB</td>
</tr>
<tr>
<td>SS-303L-12</td>
<td>80 12.0 270 120 17.5 120 0.25 54 570 140</td>
<td>21 HRB</td>
</tr>
<tr>
<td>SS-303L-15</td>
<td>100 15.0 300 170 20.0 140 0.27 75 N/D 200</td>
<td>35 HRB</td>
</tr>
<tr>
<td>SS-304N1T-30</td>
<td>210 0.0 390 280 0.5 105 0.25 5 770 280</td>
<td>81 HRB</td>
</tr>
<tr>
<td>SS-304N2-33</td>
<td>230 5.0 390 280 10.0 115 0.25 34 880 320</td>
<td>82 HRB</td>
</tr>
<tr>
<td>SS-304N2-38</td>
<td>260 8.0 480 310 13.0 140 0.27 75 N/D 320</td>
<td>68 HRB</td>
</tr>
<tr>
<td>SS-304H-20</td>
<td>140 7.0 280 170 10.0 120 0.25 27 590 170</td>
<td>35 HRB</td>
</tr>
<tr>
<td>SS-304L-13</td>
<td>90 15.0 300 120 23.0 120 0.25 61 N/D 150</td>
<td>35 HRB</td>
</tr>
<tr>
<td>SS-304L-18</td>
<td>120 18.0 390 180 26.0 140 0.27 108 N/D 190</td>
<td>45 HRB</td>
</tr>
<tr>
<td>SS-316N1-25</td>
<td>170 0.0 280 230 0.5 105 0.25 7 740 250</td>
<td>59 HRB</td>
</tr>
<tr>
<td>SS-316N2-33</td>
<td>230 5.0 410 270 10.0 115 0.25 38 860 300</td>
<td>62 HRB</td>
</tr>
<tr>
<td>SS-316N2-38</td>
<td>260 8.0 480 310 13.0 140 0.27 65 N/D 320</td>
<td>65 HRB</td>
</tr>
<tr>
<td>SS-316H-20</td>
<td>140 5.0 240 170 7.0 120 0.25 27 590 170</td>
<td>35 HRB</td>
</tr>
<tr>
<td>SS-316L-15</td>
<td>100 12.0 280 140 18.5 120 0.25 47 550 150</td>
<td>20 HRB</td>
</tr>
<tr>
<td>SS-316L-22</td>
<td>150 15.0 390 210 21.0 140 0.27 88 N/D 200</td>
<td>45 HRB</td>
</tr>
</tbody>
</table>

<sup>A</sup> Suffix numbers represent minimum strength values in 10<sup>3</sup> psi (see page 2); yield in the as-sintered condition and ultimate in the heat-treated condition.

<sup>B</sup> Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.

<sup>C</sup> Additional data in preparation will appear in subsequent editions of this standard.

N/D Not Determined for the purposes of this standard.

---

N1—Nitrogen alloyed. Good strength, low elongation.
N2—Nitrogen alloyed. High strength, medium elongation.
H—Low carbon. Lower strength, high elongation.
L—Low carbon. Lower strength, highest elongation.
Sintered at 1149°C (2100°F) in dissociated ammonia
Sintered at 1288°C (2350°F) in dissociated ammonia
Sintered at 1149°C (2100°F) in 100 % hydrogen.
Sintered at 1288°C (2350°F) in partial vacuum.
Cooled to avoid nitrogen absorption.

Processing parameters used to generate these data, other conditions could be used.

SS-303 Austenitic Machining Grades—SS-303 is preferred for parts requiring extensive secondary machining. Strength and hardness are high and corrosion resistance is good. SS-303 is non-magnetic.

SS-304 General Purpose Austenitic Grades—SS-304 has good strength properties and corrosion resistance. A general purpose grade used in many applications. SS-304 is non-magnetic.

SS-316 General Purpose Austenitic Grades—SS-316 has the best combination of properties in a P/M stainless steel alloy. Corrosion resistance is better than SS-303. First choice for general purpose applications. Non-magnetic.
### TABLE X1.19 Ferritic and Martensitic Stainless Steel

**NOTE 1**—$10^3$ psi = 6.895 MPa (6.895 N/mm²)

**NOTE 2**—1 in. = 25.4 mm

**NOTE 3**—1 ft-lbf = 1.356 J.

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Minimum Values A</th>
<th>Typical Values B</th>
<th>P/M Material Properties</th>
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</thead>
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<tr>
<td></td>
<td>Minimum Strength</td>
<td>Tensile Properties</td>
<td>Elastic Constants</td>
</tr>
<tr>
<td></td>
<td>Minimum</td>
<td>Ultimate</td>
<td>Strength (0.2 %) c</td>
</tr>
<tr>
<td></td>
<td>psi</td>
<td>%</td>
<td>psi</td>
</tr>
<tr>
<td>SS-410-90HT</td>
<td>... 90</td>
<td>0.0</td>
<td>105</td>
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<td>SS-410L-20</td>
<td>... 20</td>
<td>10.0</td>
<td>48</td>
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<tr>
<td>SS-430N2-28</td>
<td>28 ...</td>
<td>3.0</td>
<td>60</td>
</tr>
<tr>
<td>SS-430L-24</td>
<td>24 ...</td>
<td>14.0</td>
<td>50</td>
</tr>
<tr>
<td>SS-434N2-28</td>
<td>28 ...</td>
<td>4.0</td>
<td>60</td>
</tr>
<tr>
<td>SS-434L-24</td>
<td>24 ...</td>
<td>10.0</td>
<td>50</td>
</tr>
</tbody>
</table>

N2—Nitrogen alloyed. High strength, medium elongation.
* Sintered at 2350°F (1288°C) in dissociated ammonia.
L—Low carbon. Lower strength, highest elongation.
Sintered at 2350°F (1288°C) in partial vacuum.
Cooled to avoid nitrogen absorption.
HT—Martensitic grade, heat treated. Highest strength.
*Sintered at 2100°F (1149°C) in dissociated ammonia.
*Processing parameters used to generate these data, other conditions could be used.

SS-409L **Standard Ferritic Grade-SS-409** is used in applications requiring welding.

SS410-90HT **Standard Martensitic Grade-SS-410** is used in applications requiring hardness and wear resistance. Carbon is added for increased heat treatment response. As-sintered, it is in a hardened state due to the effect of furnace cooling. A secondary quench and temper heat treatment will give increased hardness. It has fair corrosion resistance and poor machinability in comparison to the 300 series SS grades. SS-410 is ferromagnetic.

SS-410L **Standard Ferritic Grade-SS-410** is used for soft magnetic properties and environments requiring the least corrosion resistance in a ferritic grade.

SS-430 **Standard Ferritic Grade-SS-430** is used for soft magnetic properties and environments requiring somewhat better corrosion resistance than SS-410. Magnetic response lower than seen with SS-410.

SS-434 **Standard Ferritic Grade-SS-434** is used for soft magnetic properties and environments requiring slightly better corrosion resistance than SS-430. Magnetic response is similar to SS-430.

A Suffix numbers represent minimum strength values in $10^3$ psi; yield in the as-sintered condition and ultimate in the heat-treated condition.

B Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.

C Tempering temperature for heat treated (HT) material; 350°F (177°C).

D Yield and ultimate tensile strength are approximately the same for heat treated materials.

N/D—Not Determined for the purposes of this standard.
### TABLE X1.20 Ferritic and Martensitic Stainless Steel (SI)

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Minimum Values&lt;sup&gt;A&lt;/sup&gt;</th>
<th>Tensile Properties</th>
<th>Elastic Constants</th>
<th>Unnotched Charpy Impact Energy</th>
<th>Transverse Rupture Strength</th>
<th>Compressive Yield Strength (0.1 %)</th>
<th>Hardness</th>
<th>10&lt;sup&gt;7&lt;/sup&gt; Cycle Fatigue Strength 90 % Survival</th>
<th>Density&lt;br&gt;g/cm&lt;sup&gt;3&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>SS-410L-90HT</td>
<td>620 MPa</td>
<td>0.0 %</td>
<td>720 MPa</td>
<td>&lt;0.5 %</td>
<td>125 MPa</td>
<td>0.25 GPa</td>
<td>15 MPa</td>
<td>23 HRC 55 HRC</td>
<td>240 MPa</td>
</tr>
<tr>
<td>SS-410L-20</td>
<td>140 MPa</td>
<td>10.0 %</td>
<td>330 MPa</td>
<td>16.0 %</td>
<td>165 MPa</td>
<td>0.27 GPa</td>
<td>66 N/D</td>
<td>45 HRB N/D</td>
<td>125 MPa</td>
</tr>
<tr>
<td>SS-430N2-28</td>
<td>190 MPa</td>
<td>3.0 %</td>
<td>410 MPa</td>
<td>5.0 %</td>
<td>170 MPa</td>
<td>0.27 GPa</td>
<td>34 N/D</td>
<td>70 HRB N/D</td>
<td>170 MPa</td>
</tr>
<tr>
<td>SS-430L-24</td>
<td>170 MPa</td>
<td>14.0 %</td>
<td>340 MPa</td>
<td>20.0 %</td>
<td>170 MPa</td>
<td>0.27 GPa</td>
<td>108 N/D</td>
<td>45 HRB N/D</td>
<td>170 MPa</td>
</tr>
<tr>
<td>SS-434N2-28</td>
<td>190 MPa</td>
<td>4.0 %</td>
<td>410 MPa</td>
<td>8.0 %</td>
<td>165 MPa</td>
<td>0.27 GPa</td>
<td>20 N/D</td>
<td>68 HRB N/D</td>
<td>150 MPa</td>
</tr>
<tr>
<td>SS-434L-24</td>
<td>170 MPa</td>
<td>10.0 %</td>
<td>340 MPa</td>
<td>15.0 %</td>
<td>165 MPa</td>
<td>0.27 GPa</td>
<td>88 N/D</td>
<td>50 HRB N/D</td>
<td>150 MPa</td>
</tr>
</tbody>
</table>

N<sub>2</sub>—Nitrogen alloyed. High strength, medium elongation.
*—Sintered at 1288°C (2350°F) in dissociated ammonia
L—Low carbon. Lower strength, highest elongation.
Sintered at 1288°C (2350°F) in partial vacuum.
Cooled to avoid nitrogen absorption.
HT—Martensitic grade, heat treated. Highest strength.
*—Sintered at 1149°C (2100°F) in dissociated ammonia
*—Processing parameters used to generate these data, other conditions could be used.

SS-409L  Standard Ferritic Grade-SS-409 is used in applications requiring high strength and wear resistance. Carbon is added for increased heat treatment response. As-sintered, it is in a hardened state due to the effect of furnace cooling. A secondary quench and temper heat treatment will give increased hardness. It has poor corrosion resistance and poor machinability in comparison to the 300 series SS grades. SS-410 is ferromagnetic.

SS-410L  Standard Ferritic Grade-SS-410 is used for soft magnetic properties and environments requiring the least corrosion resistance in a ferritic grade.

SS-430L  Ferritic Grade-SS-430L is used for soft magnetic properties and environments requiring somewhat better corrosion resistance than SS-410L. Magnetic response is lower than seen with SS-410L.

SS-434L  Ferritic Grade-SS-434L is used for soft magnetic properties and environments requiring slightly better corrosion resistance than SS-430L. Magnetic response is similar to SS-430L.

A Suffix numbers represent minimum strength values in 10<sup>3</sup> psi; yield in the as-sintered condition and ultimate in the heat-treated condition.
B Mechanical property data derived from laboratory prepared test specimens sintered under commercial manufacturing conditions.
C Tempering temperature for heat treated (HT) material; 177°C (350°F).
D Yield and ultimate tensile strength are approximately the same for heat treated materials.
N/D Not Determined for the purposes of this standard.

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Standard Test Method for
Determining the Percentage of Alloyed or Unalloyed Iron Contamination Present in Powder Forged (P/F) Steel Parts

1. Scope
1.1 This test method covers a metallographic procedure for determining the percentage of alloyed or unalloyed iron contamination present in powder forged low-alloy steel parts and the percentage of alloyed iron contamination in powder-forged iron and carbon steel parts.

1.2 Property values stated in SI units are the standard. Conversion factors to inch-pound units may be approximate.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents
2.1 ASTM Standards:
   B 243 Terminology of Powder Metallurgy
   E 3 Practice for Preparation of Metallographic Specimens

3. Terminology
3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243. Additional descriptive information is available in the Related Material Section of Vol 02.05 of the Annual Book of ASTM Standards.

3.2 Description of Term Specific to This Standard:
   3.2.1 cross product contamination—the unintentional mixing of powders with distinct differences in chemical composition.

4. Summary of Test Method
4.1 A section representing the core region of the part is taken from the powder forged part and prepared for metallographic examination.

4.2 The polished and etched sample is examined microscopically at a magnification of 100X and a systematic point count made of features with etching characteristics different from that of the matrix.

4.3 The amount of contaminant is reported as a percentage to the nearest 0.1%.

5. Significance and Use
5.1 Cross product contamination occurs whenever alloy steel powders are processed in the same equipment as iron powders.

5.2 Unalloyed iron particles, because they may not harden upon heat treatment, are a potential source of soft spots in low-alloy steel parts.

5.3 Alloyed iron particles, having higher hardenability than an iron or carbon steel matrix, are a potential source of hard spots.

5.4 Hard or soft spots may cause problems in service or machining.

5.5 The results of the tests may be used to qualify parts for shipment in accordance with guidelines agreed between purchaser and manufacturer.

6. Apparatus
6.1 Equipment for the metallographic preparation of test specimens.

6.2 A metallographic microscope permitting observation and measurement at a magnification of 100X.

7. Sampling
7.1 Take a metallographic specimen from the powder-forged part. The polished surface of the specimen should be not less than that required to superimpose 2500 grid points at a magnification of 100X. Multiple sections are permitted in order to obtain the necessary area for measurement on small parts.

7.2 The polished surface shall be parallel to the direction of forging, that is, parallel to the direction of travel of the forging
punch, or as specified in the contract or purchase order, and shall represent an area away from the surface of the part.

8. Procedure

8.1 Preparation of Specimens:

8.1.1 Polishing—In polishing the specimens, it is highly important that the polished surface be free from artifacts and debris. It is recommended that the procedures described in Practice E 3 be followed. Automated grinding and polishing procedures are recommended.

8.1.2 Etching—Lightly etch the freshly polished specimen with 2 % nital (2 mL nitric acid, 98 mL ethyl alcohol). Next, etch the polished and lightly etched specimen by immersion in a freshly prepared aqueous solution containing 3 g potassium metabisulfite and 10 g sodium thiosulfate per 100 mL. Rinse the specimen in running water, then rinse with low residue alcohol and dry with a blast of dry air.

8.1.2.1 The etching time will depend on alloy type, carbon content, and microstructure. The greater the alloy content, the slower the etching rate; the greater the carbon content, the faster the etching rate.

8.1.2.2 A good contrast is developed between the matrix and the contaminant because of a combination of etching and staining. The areas containing the highest alloy content are the least affected. Unalloyed iron will become darkened in a low-alloy matrix and low-alloy particles will remain light in an iron or carbon steel matrix. In a low-alloy matrix, contaminant particles of another low-alloy powder can be distinguished from unalloyed iron contamination because the particles etch differently (see Fig. 1 and Fig. 2).

8.2 Examination—Superimpose a grid of between 100 and 250 systematically placed points upon a 100× magnified image (that is, a field of view) of the polished and etched specimen. Count and record the number of grid points falling upon contaminant particles; if necessary, a separate count may be kept to distinguish between alloy contamination and unalloyed iron contamination in low-alloy steel parts, or, types of alloy contaminant in iron or carbon steel parts. (See Note 1.) Counting of randomly selected discrete fields should be continued until at least 2500 grid points have been superimposed on the specimen. The total number of points falling on contaminant particles for all fields counted shall be divided by the total number of grid points superimposed and multiplied by 100 to determine the area percentage of contamination.

Note: 1—Any grid point that falls on a contaminant particle boundary should be counted as one half. To avoid bias, questionable points should be counted as one half.

9. Report

9.1 Report the area percentage of contaminant to the nearest 0.1 %.

10. Precision and Bias

10.1 The precision and bias that can be expected through the use of this test method is currently under review by Subcommittee B09.11 on Near Full Density Powder Metallurgy Materials.

11. Keywords

11.1 cross product contamination; powder forging (P/F); powder forged (P/F) parts; powder forged (P/F) steels

FIG. 1 Illustration of Iron and Low-Alloy Contaminants in P/F-4650
FIG. 2 Illustration of Low-Alloy Contaminant in P/F-1060

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Designation: B 796 – 02

Standard Test Method for Nonmetallic Inclusion Content of Powders Intended for Powder Forging (P/F) Applications

This standard is issued under the fixed designation B 796; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a metallographic method for determining the nonmetallic inclusion level of powders intended for powder forging (P/F) applications.

1.2 The test method covers repress powder forged test specimens in which there has been minimal lateral material flow (< 1%). The core region of the powder forged test specimen shall contain no porosity detectable at 100×.

1.3 This test method is not suitable for determining the nonmetallic inclusion level of powder forged test specimens that have been forged such that the core region contains porosity. At the magnification used for this test method residual porosity is hard to distinguish from oxide inclusions. Too much residual porosity makes a meaningful assessment of the inclusion population impossible.

1.4 The test method may be applied to materials that contain manganese sulfide (admixed or prealloyed) provided the near neighbor separation distance is changed from 30 µm to 15 µm.

NOTE 1—The test method may be applied to powder forged parts where there has been a greater amount of material flow provided:
- The near neighbor separation distance is changed, or
- The inclusion sizes agreed between the parties are adjusted for the amount of material flow.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
- E 3 Practice for Preparation of Metallographic Specimens
- E 768 Guide for Preparing and Evaluating Specimens for Automatic Inclusion Assessment of Steel

3. Summary of Test Method

3.1 A section representing the core region is cut from the powder forged test specimen and mounted for metallographic grinding and polishing.

3.2 The polished sample is examined microscopically at a magnification of 100× and a note made of inclusions larger than a predetermined size.

3.3 The maximum Feret’s diameter is used to determine inclusion size. A Feret’s diameter is a caliper diameter as illustrated in Fig. 1.

3.4 The fragmented nature of some inclusions means that their size determination is somewhat complicated. The concept of near neighbor separation is used in determining inclusion size. If an inclusion is within a certain distance of its neighboring particles, it is considered a member of an inclusion cluster or agglomerate. Detected features within 30 µm of one another are considered part of the same inclusion. The concept is illustrated schematically in Fig. 2.

3.5 The nonmetallic inclusion level of the test specimen is reported as the number of inclusions per 100 mm² greater than or equal to the predetermined size.

4. Significance and Use

4.1 The extensive porosity present in pressed and sintered ferrous materials masks the effect of inclusions on mechanical properties. In contrast, the properties of material powder forged to near full density are strongly influenced by the composition, size, size distribution, and location of nonmetallic inclusions.

4.2 The test for nonmetallic inclusions in powder forged steels is useful as the following:
- Characteristic to classify or differentiate one grade of powder from another.
- Means of quality comparison of powders intended for powder forging, lot to lot.
- Significant variations in nonmetallic inclusion content will occur if:
  - The powder used to form the test specimen does not meet powder forging quality standards for nonmetallic inclusion content.

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1 This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.11 on Near Full Density Powder Metallurgy Parts.

2 Annual Book of ASTM Standards, Vol 03.01.
4.3.2 Processing of the powder forged test specimen has been carried out under conditions that do not permit oxide reduction or allow oxidation of the test specimen, or both.

5. Apparatus
5.1 Equipment for the metallographic preparation of test specimens.
5.2 A metallographic microscope permitting observation and measurement up to a magnification of 100× using light with a wavelength of 544 nm (green filter), an objective lens with a magnification of from 8× to 12.5×, and a numerical aperture between 0.16 and 0.20.

NOTE 2—Defining the light optics used is important because this determines the features that will be resolved, and all detected features are included in the assessment of inclusion size.

6. Sampling
6.1 A metallographic sample shall be removed from the powder forged test specimen, austenitized, and quenched.
6.2 The polished surface of the sample to be examined shall be not less than 350 mm² (0.54 in²) in area. Multiple sections are permitted in order to obtain the necessary area for measurement.
6.3 The polished surface shall be parallel to the direction of forging, that is, parallel to the direction of travel of the forging punch, and shall represent the core region of the test specimen.

7. Procedure
7.1 Preparation of Specimens—In polishing the specimens, it is highly important that a clean polish be obtained and that the inclusions not be pitted, dragged, or obscured. It is recommended that the procedures described in Practice E 3 and Guide E 768 be followed. Automated grinding and polishing procedures are recommended. Examine specimens in the as-polished condition, free of the effects of any prior etching, if used.

7.2 Measurement of Nonmetallic Inclusion Content:
7.2.1 Survey at least 350 mm² (0.54 in²) of the surface of the polished specimen at a magnification of 100× using light with a wavelength of 544 nm (green filter), an objective lens with a magnification of from 8× to 12.5×, and a numerical aperture between 0.16 and 0.20.
7.2.2 Size detected inclusions on the basis of near neighbor separation. Features within 30 µm of one another are considered to be part of the same inclusion.
7.2.3 For individual features less than 30 µm in size, three such features within 30 µm of one another are required to constitute an inclusion aggregate.
7.2.4 Add an individual feature less than 30 µm in size to an inclusion larger than 30 µm, provided both features are within 30 µm of one another. Examples are given in Figs. 3 and 4.
7.2.5 Measure and record the number of inclusion particles according to the principle of near neighbor separation and sized using the maximum Feret’s diameter that are as follows:
7.2.5.1 Greater than or equal to 30 µm but less than 100 µm in length.
7.2.5.2 Greater than or equal to 100 µm but less than 150 µm in length, and
7.2.5.3 Greater than or equal to 150 µm in length.

8. Report
8.1 Report the number of nonmetallic inclusions per 100 mm² that are as follows:
8.1.1 Greater than or equal to 30 µm but less than 100 µm in length.
8.1.2 Greater than or equal to 100 µm but less than 150 µm in length.
8.1.3 Greater than or equal to 150 µm in length.
8.2 The total area examined.

![FIG. 1 Schematic illustration of Feret’s diameter.](image)

![FIG. 2 Schematic illustration of the “near neighbor” concept and maximum Feret’s diameter.](image)

![FIG. 3 Example of a spotty oxide inclusion. The maximum Feret’s diameter is indicated.](image)
9. Precision and Bias

9.1 The precision and bias that can be expected through the use of this test method is currently under review by Subcommittee B09.11.

10. Keywords

10.1 nonmetallic inclusions; powder forged (P/F) steel parts
1. Scope

1.1 This test method covers a metallographic method for determining the maximum depth of surface finger oxide penetration and the concentration of subsurface interparticle oxide networks in critical areas of powder forged steel parts.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Document

2.1 ASTM Standards:
E 3 Guide for Preparation of Metallographic Specimens

3. Terminology

3.1 surface finger oxides—surface finger oxides are surface oxides that follow prior particle boundaries into a powder forged part from the surface and cannot be removed by physical means such as rotary tumbling. Examples of surface finger oxides are shown in Fig. 1.

3.2 interparticle oxide networks—interparticle oxide networks are continuous or discontinuous oxides that follow prior particle boundaries in powder forged parts. Examples of interparticle oxide networks are shown in Fig. 2.

4. Summary of Test Method

4.1 A section representing both surface and subsurface regions of a critical area is cut from the powder forged part and mounted for metallographic grinding and polishing.

4.2 For surface finger oxide penetration, the polished and unetched sample is examined microscopically at a magnification of 400×. The maximum depth of penetration of surface finger oxides is measured.

4.3 For interparticle oxide network concentration, the polished and unetched sample is examined microscopically at a magnification of 200 to 400× to determine the presence of interparticle oxide networks.

5. Significance and Use

5.1 The presence of surface finger oxide penetration and interparticle oxide networks are two of the properties used to evaluate powder forged steel parts for proper processing. Maximum acceptable depths of penetration of surface finger oxide penetration and acceptable concentrations of subsurface interparticle oxide networks depend on the component and its service environment.

5.2 Results of tests may be used to qualify parts for shipment.

6. Apparatus

6.1 Equipment for the metallographic preparation of test specimens.

6.2 A metallographic microscope permitting observation and measurement up to a magnification of 400×.

7. Sampling

7.1 A metallographic specimen shall be removed from the powder forged part to cover each designated critical area. Critical areas shall be defined by the applicable part drawing or the purchaser order. Specimens shall be taken from the powder forged part in the condition in which it is to be supplied. The polished surface of the specimens shall be parallel to the forging direction, that is, parallel to the direction of travel of the forging punch.

8. Procedure

8.1 Preparation of Specimens—In mounting the specimen for grinding and polishing, protection from rounding the edge of the part is essential. In polishing the specimen it is important that a clean polish be obtained and that edge detail of the part not be destroyed. Specimens shall be examined in the as-polished condition, free of the effects of any prior etching (if used). It is recommended that the procedures described in
FIG. 1 Example of Surface Finger Oxide Penetration Extending Inward from the Powder Forged Part Surface (Shown more clearly at high magnification.)
FIG. 2 Example of Interparticle Oxide Networks Within a Powder Forged Part (Shown more clearly at high magnification.)
Methods E 3 be followed. Automated grinding and polishing procedures are recommended.

8.2 Measurement of Surface Finger Oxide Penetration Depth—Scan the perimeter of the metallographic specimen, initially at a magnification of 100×, and carefully examine each designated critical area at a higher magnification, for example, 400×. Measure the maximum depth of penetration of surface finger oxides from the finished part surface in micrometers for each designated critical area.

8.3 Measurement of Interparticle Oxide Network Concentration—Scan the perimeter of the metallographic specimen at a magnification of 100×. Carefully examine each designated critical area at a higher magnification, for example, 200 to 400×. Record the presence of any interparticle oxide networks in the designated critical areas.

9. Report

9.1 The test report shall include the following information:

9.1.1 Identification of the part and location of the test specimen,

9.1.2 The maximum depth of penetration of surface finger oxides from the finished part surface in micrometers for each designated critical area, and

9.1.3 The presence or lack of interparticle oxide networks in each designated critical area. If interparticle oxide networks are present, if possible, prepare a photomicrograph for record purposes.

10. Precision and Bias

10.1 The precision and bias that can be expected through use of the test method for determining the maximum depth of surface finger oxide penetration is currently under review by Subcommittee B09.11 on Near Full Density Powder Metallurgy Parts.

10.2 The test method for interparticle oxide network concentration is a “go-no go” test designed to give a qualitative estimate. This method will be subject to sample preparation care and to interpretation.

11. Keywords

11.1 interparticle oxide networks; powder-forged (P/F) steel parts; surface finger oxides
Standard Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor ("Flowers-of-Sulfur")

This standard is issued under the fixed designation B 809; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This standard covers equipment and test methods for determining the porosity of metallic coatings, where the pores penetrate down to a silver, copper, or copper-alloy substrate.

1.2 This test method is suitable for coatings consisting of single or combined layers of any coating that does not significantly tarnish in a reduced sulfur atmosphere, such as gold, nickel, tin, tin-lead, and palladium, or their alloys.

1.3 This test method is designed to determine whether the porosity level is less than or greater than some value which by experience is considered by the user to be acceptable for the intended application.

1.4 Recent reviews of porosity testing and testing methods can be found in the literature.2,3 Guide B 765 is suitable to assist in the selection of porosity tests for electrodeposits and related metallic coatings. Other porosity test standards are Test Methods B 735, B 741, B 798, and B 799.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazards statements, see Section 8.

2. Referenced Documents

2.1 ASTM Standards:
B 374 Terminology Relating to Electroplating
B 542 Terminology Relating to Electrical Contacts and Their Use
B 735 Test Method for Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor5
B 741 Test Method for Porosity in Gold Coatings on Metal Substrates by Paper Electrography
B 765 Guide for Selection of Porosity Tests for Electrodeposits and Related Metallic Coatings4
B 798 Test Method for Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography5
B 799 Test Method for Porosity in Gold and Palladium Coatings by Sulfurous Acid/Sulfur-Dioxide Vapor5

3. Terminology

3.1 Definitions—Many terms used in this test method are defined in Terminologies B 374 and B 542.

3.2 Definitions of Terms Specific to This Standard:
3.2.1 corrosion products—reaction products of the basis metal or underplate, that protrude from, or are otherwise attached to, the coating surface after the test exposure.
3.2.2 measurement area—in this test method, that portion or portions of the surface that is examined for the presence of porosity. The measurement area shall be indicated on the drawings of the parts, or by the provision of suitably marked samples.
3.2.3 metallic coatings—in this test method, include platings, claddings, or other metallic coatings applied to the substrate. The coating can comprise a single metallic layer or a combination of metallic layers.
3.2.4 porosity—the presence of any discontinuity, crack, or hole in the coating that exposes a different underlying metal (see Guide B 765).
3.2.5 significant surface— of a coated part, is that portion (or portions) of the coating surface that is essential to the serviceability or function of the part, or which can be the source of corrosion products or tarnish films that interfere with the function of the part. For many plated products, the critical surface is identical to the measurement area.
3.2.6 tarnish—reaction products of copper or silver with oxygen or reduced sulfur (that is, hydrogen sulfide (H2S) and elemental sulfur vapor, but not sulfur dioxide (SO2) or other sulfur oxides). They consist of thin films or spots that do not protrude significantly from the surface of the metallic coating (in contrast to corrosion products).
4. Summary of Test Method

4.1 The test specimens are suspended over “flowers-of-sulfur” (powdered sulfur) in a vented container at controlled elevated relative humidity and temperature. Elemental sulfur vapor, which always exists in equilibrium with sulfur power in a closed system, attacks any exposed silver, copper, or copper alloy, such as at the bottom of pores. Brown or black tarnish spots indicate porosity.

4.2 Exposure periods may vary, depending on the extent of porosity to be revealed.

4.3 This test involves tarnish or oxidation (corrosion) reactions in which the products delineate defect sites in coatings. Since the chemistry and properties of these products may not resemble those found in natural or service environments, this test is not recommended for prediction of product performance unless correlation is first established with service experience (but see 5.3).

5. Significance and Use

5.1 A major use of this test procedure is for determining coating quality. Porosity tests are indications of the completeness of protection or coverage offered by the coatings, since the coatings described in 1.2 are intended to be protective when properly applied. The porosity test results are therefore a measure of the deposition process control.

5.2 A particular purpose of the humid sulfur vapor test is for determining the quality of underplates of nickel or nickel alloy in those finish systems that have thin, 1.2 µm or less (50 µin. or less) top layers above the nickel, since porosity in the underplate usually continues into such top layers.

5.3 The humid sulfur vapor test is often used as an environmental test to simulate many indoor humid atmosphere tarnishing and tarnish creepage effects. However, the chemistry and properties of these tarnish films may not resemble those found in other service environments. For such product performance evaluations, the test should only be used in combination with other performance evaluation tests, as specified in the referencing document for that product.

5.4 Porosity tests differ from corrosion and aging tests, since the latter are intended to measure the chemical inertness of the coating. In contrast, in a good porosity test procedure the corrosive agent should not attack the coating. It must instead, clean, depolarize, or activate the substrate metal exposed by the pore, or both, and attack it sufficiently to cause reaction products to fill the pore to the surface of the coating.

5.5 The humid sulfur test is highly sensitive, and is capable of detecting virtually all porosity that penetrates down to copper or copper alloys. Since nickel is not attacked by moist sulfur vapor at 100°C or less, this test will not detect pores or cracks in the top coating if such pores or cracks do not penetrate through the nickel underplate overlaying the copper.

5.6 The level of porosity in the coating that may be tolerable depends on the severity of the environment that the product is likely to encounter during service or storage. Also, the location of the pores on the surface is important. If the pores are few in number or away from the significant surfaces, their presence can often be tolerated.

5.7 The present test method can be used on samples of various geometries, such as curved surfaces. It can also be used for selective area coatings, if allowance is made for tarnish creepage from bare copper alloy areas.

5.8 This test method is destructive in that it reveals the presence of porosity by contaminating the surface with tarnish films. Any parts exposed to this test method should not be placed in service.

5.9 The relationship of porosity levels revealed by this test method to product performance and service life must be made by the user of the test through practical experience or by judgment. Thus, absence of porosity in the coating may be a requirement for some applications, while a few pores on the significant surfaces may be acceptable for others.

6. Apparatus

6.1 Test Vessel—May be any convenient-size vessel of glass, acrylic-resin (or of any other material that is not affected by high humidity or sulfur), such as a glass desiccator of 9 to 10 L capacity. It should have a lid or cover capable of being plugged with a stopper. The stopper shall have a 1 to 4 mm diameter hole through it to serve as a vent.

6.2 Sample Fixture or Holders—Supports or hangers shall be made from material such as glass or acrylic plastic that will not be affected by sulfur or high humidity, and shall be arranged so that the samples will be at least 75 mm away from the humidity controlling solution or sulfur powder (see 6.3). The samples shall also be at least 25 mm from the vessel walls and at least 10 mm from other samples or other surfaces. Do not use a desiccator plate. The fixture shall not cover more than 20 % of the vessel’s cross-sectional area so that air movement within the vessel will not be restricted during the test.

6.3 Glass Dish—Petri or other shallow dish approximately 15 cm in diameter to hold powdered sulfur. Dish may be supported above the constant humidity solution with plastic blocks, or floated on the liquid.

6.4 Oven, Air-circulating, capable of maintaining test vessel at a temperature of 50 ± 2°C (122 ± 4°F).

6.5 Temperature and Relative Humidity (RH) Sensor, with a remote sensor probe having a range of approximately 76 to 95 % RH at 50°C, which can be kept in the desiccator during test.

6.6 Microscope, Optical, Stereo, 10 X.—It is preferred that one eyepiece contain a graduated reticle for measuring the diameter of tarnish spots. The reticle shall be calibrated for the magnification at which the microscope is to be used.

6.7 Light Source, incandescent or circular fluorescent.

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*The Hygrodynamics Hygrometer, manufactured by Newport Scientific, Inc., has been found satisfactory for this purpose.*
7. Reagents

7.1 Potassium Nitrate (KNO₃)—American Chemical Society analyzed reagent grade, or better.
7.2 Sulfur, Sublimed (“Flowers-of-Sulfur”), N.F. or laboratory grade.⁷

8. Safety and Health Precautions

8.1 All of the normal precautions shall be observed in handling the materials required for this test. This shall also include, but not be limited to, procuring and reviewing material Safety Data Sheets (MSDS) that meet the minimum requirements of the U.S. Occupational Safety and Health Administration (OSHA) Hazard Communication Standard for all chemicals used in cleaning and testing, and observing the recommendations given.

9. Procedure

9.1 Equilibration of Test Vessel—For the initial series of tests, the test vessel shall be prepared for equilibration at least a day before the first exposure.

Note 1—For all subsequent tests, the initial 24-h equilibration procedures do not have to be repeated (see Note 2 and 9.8).

9.1.1 Place the test vessel in the oven, with sample supports in place. Make a saturated solution of potassium nitrate, prepared by adding approximately 200 g of the salt to approximately 200 mL of deionized water, with stirring, and place it in the bottom of the vessel.

Note 2—The saturated solution will contain undissolved potassium nitrate salt. This condition is necessary to achieve a constant humidity atmosphere above the solution.

9.1.2 Place lid on the vessel (do not seal it with grease), insert the temperature-humidity probe through the opening in the top of the lid (leave the stopper out), and set the oven to 55°C.

9.1.3 During equilibration, open desiccator occasionally and stir contents. As the temperature in the vessel approaches 50°C (122°F), as indicated by the temperature probe, adjust oven temperature as needed to stabilize the vessel at 50°C.

9.1.4 Fill the glass dish half-full with sulfur (break up any large lumps), and place the dish on supports above the potassium nitrate solution or float the dish directly on the solution (see Fig. 1).

9.1.5 Replace the lid and insert the vented stopper in the lid opening. Monitor the vessel temperature over several hours, and adjust oven temperature as needed to keep the vessel at 50 ± 2°C (122 ± 4°F). When stability has been attained, and the relative humidity is in the 86 to 90 % range, the apparatus is ready for insertion of test samples.

Note 3—The system described in this section may be reused for many subsequent tests without replacing the chemicals, and will remain stable for up to 6 months as long as the chemicals do not become contaminated with corrosion products or dirt. If allowed to cool, the potassium nitrate mixture will solidify, but it will liquify again when the vessel is reheated and the solution stirred. Crusts and lumps of hardened potassium nitrate should be broken up and stirred into the slurry. Add a few milliliters of deionized water if necessary to return the solution to its original condition.

9.2 Preparation of Test Samples:

9.2.1 Handle samples as little as possible, even prior to cleaning, and only with tweezers, microscope lens tissue, or clean soft cotton gloves.

9.2.2 Prior to being cleaned, the samples shall be prepared so that the measurement areas may be viewed easily through the microscope. If samples are part of assembled products, they may need to be disassembled to ensure proper access to these areas by the test environment. Since the test is specific to the planted metallic portions of the product, the latter should be separated from plastic housings, etc., whenever possible, prior to cleaning. Also, nonmetallic materials, such as paper tags, string, tape, etc., shall be removed, but take care to maintain sample identity.

9.2.3 Cleaning:

9.2.3.1 Inspect the samples under 10× magnification for evidence of particulate matter. If present, such particles should

⁷ Fisher catalogue no. S-591 or WWR Scientific catalogue no. JT4088 have been found satisfactory for this purpose.
be removed by “dusting” (that is, blowing them off the sample) with clean, oil-free air.

NOTE 4—An aerosol-can “duster” or a photographer’s brushblower are convenient tools for this purpose.

9.2.3.2 Thoroughly clean the particle-free samples with solvents or solutions that do not contain CFCs, chlorinated hydrocarbons, or other known ozone-destroying compounds. The procedure outlined in Note 5 has been found to give satisfactory results for coatings with mild to moderate surface contamination.

NOTE 5—Suggested cleaning procedure:

1. Keep individual pieces separated if there is a possibility of damage to the measurement areas during the various cleaning steps.
2. Clean samples for 5 min in an ultrasonic cleaner that contains a hot (65 to 85°C) 2 % aqueous solution of a mildly alkaline (pH 7.5 to 10) detergent.
3. After ultrasonic cleaning, rinse samples thoroughly under warm running tap water for at least 5 s.
4. Rinse samples ultrasonically for 2 min in fresh deionized water to remove the last detergent residues.
5. Immerse in fresh analytical reagent-grade methanol or isopropanol, and ultrasonically agitate for at least 30 s in order to remove the water from the samples.
6. Remove and dry samples until the alcohol has completely evaporated. If an air blast is used as an aid to drying, the air shall be oil free, clean, and dry.
7. Do not touch the measurement area of the samples with bare fingers after cleaning.

9.2.3.3 Reinspect the samples (under 10× magnification) for particulate matter on the surface. If particulates are found, repeat the cleaning step. Surface cleanliness is extremely important. Contaminants, such as plating salts and flakes of metal, may give erroneous indications of porosity.

9.3 Place the clean samples in the test vessel, in as quick a manner as possible, in order to minimize deviations from equilibrium conditions. A clean, unplated copper or copper-alloy panel should also be put into the vessel each time a test is run as an internal control, that is, to show that the test system is operative. The copper should start to darken within a few hours.

9.4 During the first 1 to 2 h of the test run, leave the desiccator slightly open, by removing the vented stopper, in order to prevent moisture condensation while the system is coming to test temperature. When the test temperature is reached and the relative humidity is in the 86 to 90 % range (which may take 1 to 2 h), replace the vented stopper.

9.5 During the first 2 to 3 h of exposure, check temperature and humidity in the desiccator and record it at suitable intervals in order to ensure the attainment of equilibrium conditions. The same shall be done towards the end of the test.

9.6 Continue the test for the required time; this shall be 24 h, unless otherwise specified. The test system may be left overnight (or over the weekend for a 3-day test), without further monitoring.

9.7 At the end of the test, remove the samples, replace the vessel lid, and allow the samples to cool to room temperature before examining them.

9.8 For all subsequent runs, eliminate the procedure described in 9.1. However, make routine checks of the actual temperature and humidity within the vessel; occasional stirring of the potassium nitrate solution may be required before a new run.

10. Examination and Evaluation

10.1 Examine the measurement areas at 10× magnification using an incandescent or circular fluorescent lamp. The presence of brown or black tarnish spots indicates that the finish is porous at these sites down to the silver or copper-alloy substrate.

10.2 If the pore sites are to be counted, the following may be useful as an aid to counting:

10.2.1 Count only tarnish and corrosion products that are brown to black.

10.2.2 Do not consider loose contamination that can easily be removed by mild air dusting as tarnish or corrosion products.

10.2.3 Move sample around under the light to vary the angle to verify pore indications. Burnished gold can give the appearance of black spots.

10.3 Measure and count a tarnish or corrosion spot when at least three-quarters of the spot falls within the measurement area. Tarnish creepage films which initiate outside the measurement area but fall within it, shall not be counted (see Fig. 2). However, the presence of significant tarnish creepage should be recorded and its location given.

10.4 Pore size shall be defined by the longest diameter of the corrosion product. Unless otherwise specified, corrosion products smaller than 0.05 mm (0.002 in.) in diameter shall not be counted. A graduated reticle in the microscope eye-piece is useful as an aid to counting and sizing.

NOTE 6—A useful sizing technique is to tabulate the pores in accordance with three size ranges: These are, approximately: (a) 0.12 mm (0.005 in.) diameter or less, (b) between 0.12 and 0.40 mm (0.005 and 0.015 in.) diameter, and (c) greater than 0.4 mm (0.015 in.) diameter.

10.5 The acceptable number, size, and location of these tarnish or corrosion spots shall be as specified in the appropriate drawing or specification.
11. Precision and Bias

11.1 Precision—The precision of this test method is being investigated using gold-plated and nickel-plated coupons.

11.2 Bias—The porosity of commercially produced metallic coatings is a property with potentially large sample-to-sample variability.\(^8\) Since there is no acceptable reference material suitable for determining the bias for porosity testing, no statement on bias is being made.

1. Scope

1.1 General—This specification covers the requirements for a coating that is a mixture of cadmium and zinc deposited on metallic products by mechanical deposition. The coating is provided in four thickness classes (see Table 1) and several finish types (see Table 2).

1.2 Toxicity—Warning: Cadmium is toxic; therefore these coatings should not be used on articles that will contact food or beverages, or for dental and other equipment that may be inserted into the mouth. Also, the coatings should not be used on articles that will be heated to high temperatures, because cadmium will form toxic fumes. Similarly, if coated articles are welded, soldered, or otherwise heated during fabrication, adequate ventilation should be provided to exhaust toxic fumes.

1.3 Similar Documents—Other kinds of mechanically deposited coatings are covered by Specifications B 635, B 695, and B 696.

1.4 The following precautionary caveat pertains only to the test method portion, Section 9, of this specification. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 117 Practice for Operating Salt Spray (Fog) Apparatus
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 320 Practice for Preparation of Iron Castings for Electroplating
B 322 Practice for Cleaning Metals Prior to Electroplating
B 374 Terminology Relating to Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section
B 571 Test Methods for Adhesion of Metallic Coatings
B 635 Specification for Coatings of Cadmium-Tin Mechanically Deposited on Iron and Steel
B 695 Specification for Coatings of Zinc Mechanically Deposited on Iron and Steel
B 696 Specification for Coatings of Cadmium Mechanically Deposited
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 762 Method of Variables Sampling of Metallic and Inorganic Coatings

### TABLE 1 Thickness Classes

<table>
<thead>
<tr>
<th>Class</th>
<th>Coating Thickness Minimum, µm</th>
<th>Typical Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>7</td>
<td>Automotive fasteners</td>
</tr>
<tr>
<td>12</td>
<td>12</td>
<td>Increased corrosion resistance (for example, bellville washers)</td>
</tr>
<tr>
<td>25</td>
<td>25</td>
<td>Exterior hardware</td>
</tr>
<tr>
<td>50</td>
<td>50</td>
<td>Pole line hardware in severe environments</td>
</tr>
</tbody>
</table>

### TABLE 2 Coating Types

<table>
<thead>
<tr>
<th>Type</th>
<th>Description</th>
<th>Typical Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>As-coated, without supplementary treatments.</td>
<td>Lowest cost where white corrosion products are acceptable. For elevated temperature application that will degrade Type II coatings (see 1.2).</td>
</tr>
<tr>
<td>Ila</td>
<td>With yellow to bronze color supplementary chromate coating.</td>
<td>Delay the appearance of white corrosion products. Increase total corrosion protection.</td>
</tr>
<tr>
<td>Iib</td>
<td>With brown to olive drab color supplementary chromate coating.</td>
<td>Greater corrosion resistances than Ila. To provide a match to military equipment.</td>
</tr>
<tr>
<td>Ilc</td>
<td>Type Ila, dyed.</td>
<td>Color coding</td>
</tr>
<tr>
<td>Id</td>
<td>Type Ila with an added lubricant or organic finish (oil, wax, lacquer, etc.)</td>
<td>Decorative purposes</td>
</tr>
</tbody>
</table>
5. Ordering Information

5.1 In order to make the application of this specification complete, the purchaser needs to supply the following information to the seller on the purchase order or other governing documents:

5.1.1 The name, designation, and year of issue of this specification,
5.1.2 Thickness class (see 4.1), including a maximum thickness if appropriate,
5.1.3 Coating type (see 4.2), including required color if Type IIc is used, and required lubricant or organic finish if Type IIId is used,
5.1.4 Nature of the substrate, for example: high-carbon steel, mild steel, copper, brass:
   5.1.4.1 State if precoating stress relief heat treatment is required and the time and temperature to be used if different from the standard values (see section 12.1),
   5.1.4.2 State if special pretreatments are required to modify the surface of the article (see Note 1),
   5.1.4.3 If special cleaning precautions are to be followed (see A1.1), and
5.1.5 Identification of significant surfaces (see 7.4.2),
5.1.6 Requirements and methods of testing one or more of the following:
   5.1.6.1 Need for and type of special test specimens (see 9.1),
   5.1.6.2 Appearance (see 7.3),
   5.1.6.3 Deposit composition (see 9.2),
   5.1.6.4 Thickness (see 9.5),
   5.1.6.5 Adhesion (see 9.6),
   5.1.6.6 Corrosion resistance (see 9.7),
   5.1.6.7 Absence of hydrogen embrittlement, waiting time prior to testing and testing loads (see 9.8), and
5.1.7 The sampling plan to be used (see 8.1) and responsibility for inspection (see section 13.1).

6. Significance and Use

6.1 Corrosion Resistance, General—This functional coating is used to provide corrosion resistance. The performance of this coating depends largely on its thickness and the kind of environment to which it is exposed. Published results of environmental corrosion studies have demonstrated that the coating provides corrosion resistance greater than equivalent thicknesses of zinc coatings in industrial environments and greater corrosion resistance than equivalent thicknesses of cadmium coatings in marine environments.

6.2 Galvanic Corrosion Resistance—The galvanic couple that results in the corrosion of steel and aluminum parts in contact with each other in corrosive environments can also be minimized by plating the steel parts with this mechanically deposited coating.

6.3 Hydrogen Embrittlement, Absence of—The mechanical coating process does not produce any permanent hydrogen embrittlement in products made from high-strength steels, for example, fasteners or lock washers.

7. Coating Requirements

7.1 Nature of Coating—The coating shall be a mechanically deposited mixture of cadmium and zinc with the composition 45 to 75 mass % zinc, remainder cadmium.

7.2 Coating Process:

7.2.1 Coating—The cadmium-zinc coating shall be produced by mechanical deposition in accordance with the process description given in Annex A1.

7.2.2 Supplementary Treatments—Type II coatings shall be produced by treatment with acidic solutions that contain hexavalent chromium compounds and anions that act as catalysts or film-forming compounds.

7.3 Appearance:

7.3.1 General—The coating on all readily visible surfaces shall be uniform in appearance, well compacted, and complete in coverage. Superficial staining from rinsing and drying and mild variations in color and luster are acceptable.

7.3.2 Surface Defects—Defects and variations in appearance in the coating that arise from surface conditions of the substrate (scratches, pores, roll marks, inclusions, etc.) and that persist in the coating despite the observance of good metal finishing practices shall not be cause for rejection.

Note: Coatings generally perform better in service when the substrate over which they are applied is smooth and free of torn metal, inclusions, pores, and other defects. The specifications covering the unfinished products should provide limits for these defects. A metal finisher can often remove defects through special treatments, such as grinding, polishing, abrasive blasting, chemical etches, and electropolishing. However, these are not normal in the treatment steps preceding the application of the coating. When they are desired, they are the subject of special agreement between the purchaser and the seller.

7.4 Thickness:

7.4.1 Conformance to Specified Class—The thickness of the coating everywhere on the significant surfaces shall conform to the requirements of the specified class as defined in 4.1.

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4 Discontinued; see 1986 Annual Book of ASTM Standards, Vol 03.06.
5 Annual Book of ASTM Standards, Vol 03.05.
6 Annual Book of ASTM Standards, Vol 03.06.
7 Annual Book of ASTM Standards, Vol 01.08.
7.4.2 Significant Surfaces—Significant surfaces are usually defined as those normally visible (directly or by reflection) that are essential to the appearance and serviceability of the article when assembled in normal position; or that can be the source of corrosion products that deface visible surfaces on the assembled article. Significant surfaces are further defined at those surfaces that are identified as such by the purchaser, for example, by indicating them on an engineering drawing of the product or by marking a sample item of product.

7.4.3 Minimum Thickness Requirement—The coating requirement of this specification is a minimum requirement; that is, the coating thickness is required to equal or exceed the specified thickness everywhere on the significant surfaces. Variation in the thickness from point to point on an article and from article to article in a production lot is inherent in mechanically deposited coatings. Therefore, if all of the articles in a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the specified minimum.

NOTE 2—The thickness of mechanically deposited coatings varies from point to point on the surface of a product, characterizedly tending to be thicker on flat surfaces, and thinner at exposed edges, sharp projections, shielded or recessed areas, and interior corners and holes, depending on the dimensions, with such thinner areas often being exempted from thickness requirements.

NOTE 3—Processes used to produce Type II finishes remove some of the coating. Because thickness requirements applied to the finished article, additional thicknesses may have to be applied to compensate for the metal removed in the Type II process.

7.5 Adhesion—The coating shall be adherent, as defined or tested in accordance with 9.6.

7.6 Corrosion Resistance:

7.6.1 Type I Coatings on Ferrous Articles—Type I coated ferrous articles shall not develop red corrosion products (“rust”) when submitted to the 5% salt spray test for the following times:

<table>
<thead>
<tr>
<th>Class</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>36</td>
</tr>
<tr>
<td>12</td>
<td>72</td>
</tr>
<tr>
<td>25</td>
<td>192</td>
</tr>
<tr>
<td>50</td>
<td>300</td>
</tr>
</tbody>
</table>

7.6.2 Type II Coatings, White Corrosion—Type II coatings shall not develop white corrosion products when submitted to the salt spray test for 72 h. The organic coating or lubricant shall be removed from Type IId coatings before the test or the test can be run on articles that are withdrawn from processing before the organic coating is applied.

7.6.3 Type II Coatings on Ferrous Articles—Type II coated ferrous articles shall not develop red corrosion products (“rust”) when submitted to the 5% salt spray test for the following times:

<table>
<thead>
<tr>
<th>Class</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>72</td>
</tr>
<tr>
<td>12</td>
<td>96</td>
</tr>
<tr>
<td>25</td>
<td>192</td>
</tr>
<tr>
<td>50</td>
<td>300</td>
</tr>
</tbody>
</table>

The organic coating or lubricant shall be removed from Type IId coatings before the salt spray test or the test can be run on articles that are withdrawn from processing before the organic coating is applied.

NOTE 4—In many instances, there is no direct relation between the results of an accelerated corrosion test and the resistance to corrosion in other tests or actual environments, because several factors that influence the progress of corrosion, such as the formation of protective film, vary greatly with the conditions encountered. The results obtained in the test should not, therefore, be regarded as a direct guide to the corrosion resistance of the tested materials in all environments where these materials may be used. Also, performance of different materials in the test cannot always be taken as a direct guide to the relative corrosion resistance of these materials in service.

8. Sampling

8.1 The purchaser and producer are urged to employ statistical process control in the coating process. Properly performed, statistical process control will assure coated products of satisfactory quality and will reduce the amount of acceptance inspection. The sampling plan used for the inspection of the quality coated article shall be agreed upon between the purchaser and producer.

8.1.1 When a collection of coated articles (inspection lot, see 8.2) is examined for compliance with the requirements placed on the articles, a relatively small number of the articles (sample) is selected at random and is inspected. The inspection lot is then classified as complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria for compliance are determined by the application of statistics. The procedure is known as sampling inspection. Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for sampling inspection of coatings.

8.1.2 Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one when they are destructive. Test Method B 602 provides a default plan if one is not specified.

8.1.3 Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. Guide B 697 provides a default plan if one is not specified.

8.1.4 Test Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numeric value and certain statistical requirements must be met. Test Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. Test Method B 762 provides a default plan if one is not specified.

8.1.5 Guide F 1470 can be used for fasteners such as internally threaded, externally threaded and nonthreaded fasteners and washers. This guide provides for two plans: one designated the “detection process” and one designated the “prevention process.” The purchaser and producer shall agree on the plan to be used.

8.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specification, that have been coated by a single supplier at one time or approximately the same time, under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

9. Test Methods

9.1 Special Test Specimens:

9.1.1 Test specimens may be used to represent the coated articles in a test if the articles are of a size, shape, or material
that is not suitable for the test, or if it is preferred not to submit articles to a destructive test because, for example, the articles are expensive or few. State the permission or the requirement to use the test specimens, their number, the material from which they shall be made, and their size and shape in the purchase order or other governing document.

9.1.2 Duplicate in the test specimen those characteristics of the article that influence the property being tested, and process it with the article through those process steps that influence the property.

9.1.3 Make the test specimen used to represent an article in an adhesion, corrosion resistance, or appearance test of the same material, in the same metallurgical condition, and have the same surface condition as the article it represents. Place it in the production lot of and process along with the article it represents.

9.1.4 Introduce a test specimen used to represent an article in a coating thickness test into the process at the point where the coating or coatings are applied and carry it through all steps that have a bearing on the coating thickness.

NOTE 5—When a test specimen is used to represent a coated article in a thickness test, the specimen will not necessarily have the same coating thickness and coating thickness distribution as the article unless the specimen and the article are of the same general size and shape. Therefore, before coated articles may be accepted on the basis of as thickness test performed on test specimens, the relationship between the thickness on the specimen and the thickness of the part needs to be established. The criterion of thickness acceptance is that coating thickness on the specimen that corresponds to the required thickness on the article.

9.2 Composition of Coating—Determine chemical composition of the coating by procedures in Method E 27, Test Methods E 396, Test Method E 536, or by other methods that can determine composition to within 0.1 mass % unit.

9.3 Process—The supplier shall certify that the cadmium-zinc coating was produced from the mechanical deposition process as required by this specification.

9.4 Appearance—Examine the coating by the unaided eye at a normal reading distance for conformance to the requirements of appearance.

9.5 Thickness:

9.5.1 Determine the thickness of the coating by Test Methods B 487 or B 499, as applicable. Other methods may be used if it can be demonstrated that the uncertainty of the measurements with these methods is less than 10%.

9.5.2 Measure the thickness of the coating at the location or locations on the significant surfaces of the article where the coating would be expected to be the thinnest, or at such locations as specified on the purchase order.

9.5.3 Make thickness measurements of Type II coatings after the application of the supplementary treatment. Chromate treatments can significantly affect the accuracy of the thickness measuring methods used. For this reason, remove Type II supplementary coatings from the test areas prior to thickness measurement. Remove the supplementary coatings by using a very mild abrasive (a paste of levigated alumina or magnesium oxide rubbed on gently with the finger). The process by which Type II supplementary coatings are produced dissolves a small amount of cadmium-zinc. For this reason the thickness requirement refers to the thickness of the coating after the application of the Type II supplementary coating.

9.6 Adhesion—Test adhesion of the cadmium-zinc deposit to the basis metal in a manner that is consistent with the service requirements of the coated article. The ability to separate the coating from the substrate by peeling, as distinct from flaking caused by rupture of the deposit or of the base metal, is evidence of failure. Use either one of the following methods for determining adhesion, or any more applicable test method as defined in Note 6:

9.6.1 Deform the part, if possible, to its rupture and examine the coating to determine evidence of failure as defined in 9.6.

9.6.2 Scrape or shear the surface of the coated article with a sharp edge, knife, or razor blade through the coating down to the basis metal and examine under 4× magnification.

NOTE 6—There is no single satisfactory test for evaluating the adhesion of mechanically deposited coatings. Those given in 9.6 are widely used; however, other tests may prove more applicable in specific cases. Various qualitative methods are discussed in Test Methods B 571. A review of other methods of measuring adhesion has been presented by Polleys. Application of pressure-sensitive tape to the coating may be misleading. Distinctions must be made between superficial surface effects which will not adversely affect other requirements and internal coating consolidation (cohesion) or adhesion to the basis metal or undercoating.

9.7 Corrosion Resistance:

9.7.1 Test the corrosion resistance of the coating by the 5% neutral salt spray (fog) test as defined in Practice B 117.

9.7.2 If samples with Type II coatings are being examined both for white corrosion products and for rust, separate sets of samples may be used to determine the 72-h end point for white corrosion and the end point for rust; this is to permit exposure for the longer period without having to wash specimens for examination of white corrosion in accordance with Practice B 117.

9.7.3 Age parts with a Type II supplementary chromate film at room temperature for 24 h before subjection to the salt spray test.

9.7.4 The presence of corrosion products, either white from the coating itself or red from an iron or steel basis metal, and visible to the unaided eye at a normal reading distance at the end to the specified test period constitute failure, except that corrosion products at the edges of specimens, or within 5 mm from edges or sharp corners, do not constitute failure. Slight “whisps” of white corrosion products, as opposed to obvious accumulations, are acceptable.

9.8 Absence of Hydrogen Embrittlement—Test coated parts being tested for the absence of embrittlement from cleaning for brittle failure in accordance with a suitable method to be specified on the purchase order (see 5.1.6.7). The description of the method shall include the means of applying a load to the part, the stress or load level to be applied, the duration of the test, and the waiting time that must elapse between deposition of the coating and testing or use of the part and the criterion of failure (see Annex A2). The stress

level induced by the test and the waiting period prior to test depend on many factors, such as shape of the part, carbon content of the steel, hardness of the part, and the stress level in use. Parts with a tensile strength of over 1000 MPa, for example, may require a 48-h waiting period; parts with a lower tensile strength may require less than a 24-h waiting period. High-carbon steel parts or those cold-worked or heat-treated to tensile strengths of 1450 MPa minimum, where these parts will be subjected to a sustained load in use, may require testing at loads specified by the purchaser.

10. Rejection and Rehearing

10.1 Coated articles that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of the test, the producer or supplier may make a claim for rehearing. Coatings that show imperfections during subsequent manufacturing operations may be rejected.

11. Certification

11.1 When specified in the purchase order or contract, the purchaser shall be furnished certification that samples representing each lot have been either tested or inspected as directed in this specification and that the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirement applies only when specified by the purchaser in the contract of order.

S1. Stress Relief—Parts that are made from steels with ultimate tensile strengths greater than 1050 MPa and that have been machined, ground, cold-formed, or cold-straightened shall be stress-relieved before processing by heat treating for 5 h at 190 ± 15°C.

SPECIAL GOVERNMENT REQUIREMENTS

The following special requirements shall apply when the purchaser is the United States Government or an agent of the United States Government.

S2. The producer or supplier shall be responsible for the performance of all inspection and test requirements specified herein (see section 6.7). Except as otherwise specified in the contract or order, the producer or supplier may use his own or any other suitable facility for the performance of the inspection and test requirements specified herein, unless disapproved by the purchaser. The purchaser shall have the right to perform any of the inspections and tests set forth in this specification where such inspections are deemed necessary to assure that material conforms to prescribed requirements.

ANNEXES

(Mandatory Information)

A1. COATING PROCESS

A1.1 The coating process is described in this annex in general terms. The producer of the coating will establish the specific operating details.

A1.1.1 Preclean as necessary to remove gross contamination (oil, scale, corrosion, preservatives, etc.). Useful cleaning guidelines are given in Practices B 183, B 242, B 320, and B 322.

A1.1.2 Prepare the surface, generally within the mechanical deposition container, by a chemical (generally a mild, inhibited acidic) procedure.

A1.1.3 Deposit, as appropriate to the basis metal, a thin metal coating by immersion, without the use of electric current, in an appropriate chemical solution. For ferrous metals, this coating is generally copper.

A1.1.4 Tumble the parts in a container with the following: cadmium and zinc powders, impact media, generally glass beads, a promoter or accelerator, and a liquid, generally water.

A1.1.5 Separate the articles from the other ingredients.

A1.1.6 Rinse.

A1.1.7 Apply any required supplementary treatment, and

A1.1.8 Dry.
A2. HYDROGEN EMBRITTLEMENT

A2.1 A major advantage of mechanical plating is that it does not produce any permanent embrittlement in hardened steel during the coating process. However, pronounced embrittlement can be produced in certain cleaning processes. The mild degree of embrittlement that might result from following proper procedures with cleaning methods permitted in this specification normally is self-relieving within a day’s time at room temperature.
1. Scope

1.1 This specification covers powder metallurgy (P/M) structural components fabricated from commercially pure (CP) titanium powder mixed with master alloy powder and elemental powders in appropriate quantity to yield combined material chemistries comparable to ingot metallurgy (I/M) alloys Titanium 6A1-4V and Titanium 6A1-6V-2Sn.

1.2 This specification covers the following materials:

1.2.1 Two types depending on alloy composition as detailed in Table 1.

1.2.1.1 Type I is comparable to I/M Ti-6A1-4V.

1.2.1.2 Type II is comparable to I/M Ti-6A1-6V-2Sn.

1.2.2 Two grades of each type that result from the specific titanium powder used are as follows:

1.2.2.1 Grade 1 is made from sponge fines with residual levels of chlorine and sodium.

1.2.2.2 Grade 2 is made from hydride/dehydride (HDH) or other process titanium with significantly lower chlorine and sodium content.

1.2.3 Two classes as a function of density (see Table 2) are as follows:

1.2.3.1 Class A density ratio is 94 % minimum.

1.2.3.2 Class B density ratio is 99 % minimum.

(Warning—CP titanium powder may be pyrophoric; its use may involve an explosion hazard.)

1.3 The values stated in inch-pound units are to be regarded as the standard. The SI units given in parentheses are for information only.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in 1.2.3.2.

### TABLE 1 Chemical Composition Requirements

<table>
<thead>
<tr>
<th>Element</th>
<th>Type I</th>
<th>Type I</th>
<th>Type II</th>
<th>Type II</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>5.50/6.75</td>
<td>5.0/6.0</td>
<td>5.50/6.75</td>
<td>5.0/6.0</td>
</tr>
<tr>
<td>Vanadium</td>
<td>3.50/4.50</td>
<td>5.0/6.0</td>
<td>3.50/4.50</td>
<td>5.0/6.0</td>
</tr>
<tr>
<td>Tin</td>
<td>N/A</td>
<td>1.5/2.5</td>
<td>N/A</td>
<td>1.5/2.5</td>
</tr>
<tr>
<td>Iron</td>
<td>0.40 max</td>
<td>0.35/1.0</td>
<td>0.40 max</td>
<td>0.35/1.0</td>
</tr>
<tr>
<td>Copper</td>
<td>N/A</td>
<td>0.35/1.0</td>
<td>N/A</td>
<td>0.35/1.0</td>
</tr>
<tr>
<td>Oxygen, max</td>
<td>0.30</td>
<td>0.30</td>
<td>0.30</td>
<td>0.30</td>
</tr>
<tr>
<td>Hydrogen, max</td>
<td>0.015</td>
<td>0.015</td>
<td>0.015</td>
<td>0.015</td>
</tr>
<tr>
<td>Nitrogen, max</td>
<td>0.04</td>
<td>0.04</td>
<td>0.04</td>
<td>0.04</td>
</tr>
<tr>
<td>Carbon, max</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
</tr>
<tr>
<td>Sodium, max</td>
<td>0.20</td>
<td>0.20</td>
<td>TBD#</td>
<td>TBD#</td>
</tr>
<tr>
<td>Chlorine, max</td>
<td>0.20</td>
<td>0.20</td>
<td>TBD#</td>
<td>TBD#</td>
</tr>
<tr>
<td>Silicon, max</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
</tr>
<tr>
<td>Residual elements each, max</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
</tr>
<tr>
<td>Residual elements total, max</td>
<td>0.40</td>
<td>0.40</td>
<td>0.40</td>
<td>0.40</td>
</tr>
</tbody>
</table>

*Not applicable.*

# Various chloride levels may be available between the standard 0.20 max and the wrought equivalent of 0.001 max. The acceptable level for specific product applications shall be agreed upon between the purchaser and supplier and specified on the purchase order.

2. Referenced Documents

2.1 ASTM Standards:

B 243 Terminology of Powder Metallurgy

B 311 Test Method for Density Determination for Powder Metallurgy (P/M) Materials Containing Less Than Two Percent Porosity

B 328 Test Methods for Density, Oil Content, and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings

3. Terminology

3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243.

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2 Annual Book of ASTM Standards, Vol 02.05.
3.2 Descriptions of Terms Specific to This Standard—
Additional descriptive information is available in the Related Material section of Volume 02.05 of the Annual Book of ASTM Standards.

4. Ordering Information
4.1 Orders for components under this specification shall include the following information:
4.1.1 Dimensions (see Section 9),
4.1.2 Chemical composition (see Section 6 and Table 1),
4.1.3 Density (see 7.1 and Table 2),
4.1.4 Mechanical properties (see Section 8 and Table X1.1), and
4.1.5 Certification (see Section 13).

5. Materials and Manufacture
5.1 Structural components shall be fabricated by cold compacting a mixture of CP titanium, master alloy, and other elemental powders into suitable shapes. The compacts shall be vacuum sintered and hot isostatically pressed, if necessary, to provide parts conforming to the requirements of this specification.

6. Chemical Composition
6.1 Chemical composition shall conform to the requirements of Table 1.
6.2 Chemical analysis shall be made in accordance with methods prescribed in Volume 03.05 of the Annual Book of ASTM Standards, or any other standard method mutually agreed upon between the manufacturer and the purchaser.

7. Physical Properties
7.1 Density:
7.1.1 Density ratio shall exceed minimum limits prescribed in Table 2. The purchaser and the producer shall mutually agree on pore free density values.
7.1.2 Density shall be determined in accordance with Test Method B 328 for Class A materials.
7.1.3 Density shall be determined in accordance with Test Method B 311 for Class B materials.

8. Mechanical Properties
8.1 Whenever feasible tests shall be performed on material removed from actual components. The test requirements shall be determined after consideration of actual component function.
8.2 The required mechanical properties and a sampling plan shall be agreed upon between the manufacturer and the purchaser. All shipments of components subsequent to the establishment of testing conditions shall conform to the agreed limits.

9. Dimensions and Tolerances
9.1 Dimensions and tolerances of the structural components shall be indicated on drawings accompanying the purchase order or contract.

10. Sampling
10.1 Lot—Unless otherwise specified, a lot shall consist of components fabricated from powder of the same mix lot; compacted, sintered (and hot isostatically pressed, if required) under the same conditions.
10.2 Chemical Analysis—If required by purchase agreement, at least one sample for chemical analysis shall be taken from each lot. A representative sample of chips may be obtained by dry-milling, drilling or crushing at least two pieces without lubrication using clean, dry tools.
10.3 Mechanical Testing—If required by purchase agreement, the manufacturer and purchaser shall mutually agree on the representative number of specimens for mechanical testing, from each lot.

11. Inspection
11.1 Unless otherwise specified, inspection of components supplied on contract shall be made by the purchaser.

12. Rejection and Rehearing
12.1 Components that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with test results, the producer or supplier may make a claim for a rehearing.

13. Certification
13.1 When specified in the purchase order or contract, a producer’s certification of compliance document shall be furnished to the purchaser, verifying that the components manufactured were sampled, tested and inspected in accordance with this specification and have met the requirements. When specified in the purchase order or contract, a report of test results shall be furnished.
13.2 Certification by an independent, third party indicating conformance to the requirements of this specification may be considered upon the request of the purchaser.
13.3 The purchase order or contract must specify whether or not the certification includes a report of chemical analysis.

14. Keywords
14.1 compaction; cold isostatic pressing (CIP); ELCL titanium; HDH titanium powder; hot isostatic pressing (HIP); powder metallurgy; “sponge fines” titanium powder; structural components; Ti-6Al-4V; Ti-6Al-6V-2Sn; titanium; titanium alloys; vacuum sinter
APPENDIX

(Nonmandatory Information)

X1. MECHANICAL PROPERTY DATA

X1.1 Typical data for the mechanical properties of elementally mixed titanium alloy specimens are given in Table X1.1. These data do not constitute a part of this specification. They indicate, to the purchaser, the mechanical properties that may be expected from tension specimens conforming to the specified density and chemical requirements.

Note X1.1—Refer to Refs. (1-10) for supplemental material property information.

The boldface numbers in parentheses refer to a list of references at the end of the text.

REFERENCES


TABLE X1.1 Typical Properties

<table>
<thead>
<tr>
<th>Type</th>
<th>Grade</th>
<th>Class</th>
<th>Tensile Strength</th>
<th>Yield Strength</th>
<th>Elongation in 1 in. (25 mm)</th>
<th>Reduction in Area, %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>10^3 psi (MPa)</td>
<td>10^3 psi (MPa)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I</td>
<td>1</td>
<td>A</td>
<td>111 (765)</td>
<td>101 (690)</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>I</td>
<td>1</td>
<td>B</td>
<td>123 (848)</td>
<td>108 (745)</td>
<td>8</td>
<td>14</td>
</tr>
<tr>
<td>II</td>
<td>1</td>
<td>A</td>
<td>131 (903)</td>
<td>113 (779)</td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td>II</td>
<td>1</td>
<td>B</td>
<td>139 (958)</td>
<td>131 (903)</td>
<td>6</td>
<td>8</td>
</tr>
<tr>
<td>I</td>
<td>2</td>
<td>A</td>
<td>131 (903)</td>
<td>118 (814)</td>
<td>8</td>
<td>18</td>
</tr>
<tr>
<td>I</td>
<td>2</td>
<td>B</td>
<td>139 (958)</td>
<td>125 (862)</td>
<td>13</td>
<td>29</td>
</tr>
<tr>
<td>II</td>
<td>2</td>
<td>A</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>II</td>
<td>2</td>
<td>B</td>
<td>139 (958)</td>
<td>128 (883)</td>
<td>13</td>
<td>13</td>
</tr>
</tbody>
</table>

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Standard Guide for
Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis¹

This standard is issued under the fixed designation B 821; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This guide covers the dispersion in liquids of metal powders and related compounds for subsequent use in particle size analysis instruments. This guide describes a general procedure for achieving and determining dispersion; it also lists procedures that are currently in general use for certain materials.

1.2 This guide is limited to metal powders and related metal compounds. However, the general procedure described herein may be used, with caution as to its significance, for other particulate materials, such as ceramics, pigments, minerals, etc.

1.3 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 243 Terminology of Powder Metallurgy²
B 430 Test Method for Particle Size Distribution of Refractory Metal Powders and Related Compounds by Turbidimetry²
B 761 Test Method for Particle Size Distribution of Refractory Metals and Their Compounds by X-Ray Monitoring of Gravity Sedimentation²
B 822 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering²

3. Terminology

3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243.

4. Significance and Use

4.1 The method of powder dispersion in a liquid has a significant effect on the results of a particle size distribution analysis. The analysis will show a too-coarse, unstable, or nonrepeatable distribution if the powder has not been dispersed adequately. It is therefore important that parties wishing to compare their analyses use the same dispersion technique.

4.2 This guide provides established powder dispersion techniques for certain materials and the means of deriving techniques for materials not listed. It should be used by all parties performing liquid-dispersed particle size analysis of all of the materials covered by this guide (see 1.1, 1.2, and 4.1).

4.3 This guide should be used in the preparation of powders for use in Test Methods B 430, B 761, and B 822 and other procedures that analyze metal powder particle size distributions in liquid-dispersed systems.

5. Apparatus

5.1 Microscope, suitable for observation of particles in the size range of 5 to 1000 µm.
5.2 Ultrasonic Probe, ½ -in. (25.4-mm) tip, with the power level to be determined by this guide.
5.3 Ultrasonic Bath—Power level to be determined by this guide.

6. Reagents

6.1 Purity of Reagents—Reagent grade chemicals should be used in all tests. Unless otherwise indicated, it is intended that all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 Surfactants—Suggested surfactants are listed in Table 1 and footnotes 4 through 6.⁴⁻⁵⁻⁶

* A Summary of Changes section appears at the end of this standard.

¹ This guide is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.02 on Base Metal Powders.
³ Annual Book of ASTM Standards, Vol 02.05.

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7. General Dispersion Procedure

7.1 The general procedure for determining and achieving proper dispersion is outlined in Fig. 1 and described in detail below:

7.1.1 Place a test portion of the powder to be analyzed in a beaker containing the carrier liquid, selected according to 7.1.2.

7.1.2 Selection of Carrier Liquid:

**NOTE 1**—The selected carrier liquid must be compatible with the components of the instrument used for the particle size analysis.

7.1.2.1 If the powder reacts with, or is soluble in, water and organic liquids, it must be analyzed in the dry state, and the remainder of this guide is then not applicable.

---

**TABLE 1 Recommended Dispersion Procedures**

<table>
<thead>
<tr>
<th>Material</th>
<th>Carrier Liquid</th>
<th>Surfactant</th>
<th>Surfactant Concentration</th>
<th>Ultrasonic Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Type</td>
</tr>
<tr>
<td>Chromium carbide</td>
<td>water</td>
<td>none</td>
<td>...</td>
<td>none</td>
</tr>
<tr>
<td>Copper</td>
<td>water</td>
<td>Tween 21(^a)</td>
<td>3–5 drops(^c)</td>
<td>bath</td>
</tr>
<tr>
<td>Ferroalloys</td>
<td>isopropyl alcohol</td>
<td>Tween 21(^a)</td>
<td>10 %</td>
<td>bath</td>
</tr>
<tr>
<td>Iron/steel</td>
<td>water</td>
<td>Tween 21(^a)</td>
<td>3–5 drops(^c)</td>
<td>bath</td>
</tr>
<tr>
<td>Manganese sulfide</td>
<td>water</td>
<td>Tween 21(^a)</td>
<td>3–5 drops(^c)</td>
<td>bath</td>
</tr>
<tr>
<td>Molybdenum</td>
<td>water</td>
<td>sodium hexametaphosphate</td>
<td>0.01 %</td>
<td>probe</td>
</tr>
<tr>
<td>Nickel</td>
<td>water</td>
<td>Tween 21(^a)</td>
<td>3–5 drops(^c)</td>
<td>bath</td>
</tr>
<tr>
<td>Tantalum</td>
<td>water</td>
<td>sodium hexametaphosphate</td>
<td>0.01 %</td>
<td>probe</td>
</tr>
<tr>
<td>Tantalum carbide</td>
<td>water</td>
<td>sodium hexametaphosphate</td>
<td>0.01 %</td>
<td>or</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>or</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>or</td>
</tr>
<tr>
<td>Tungsten</td>
<td>water</td>
<td>sodium hexametaphosphate</td>
<td>0.01 %</td>
<td>probe</td>
</tr>
<tr>
<td>Tungsten carbide</td>
<td>water</td>
<td>sodium hexametaphosphate</td>
<td>0.01 %</td>
<td>or</td>
</tr>
<tr>
<td></td>
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<td></td>
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<td></td>
<td></td>
<td>or</td>
</tr>
</tbody>
</table>

\(^a\) As described in Test Method B 430.
\(^b\) Tween 21, chemically known as polyoxyethylene sorbitan monolaurate, is available from ICI Americas, Inc., Specialty Chemicals Division, Murphy and Concord Pike, Wilmington, DE 19897.
\(^c\) Three to five drops Tween 21 in 30 to 50 mL water.

7.2.1 If the powder reacts with, or is soluble in, water and organic liquids, it must be analyzed in the dry state, and the remainder of this guide is then not applicable.

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**FIG. 1 General Dispersion Procedure**

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\(^1\) Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.


7.1.2.2 If the powder reacts with, or is soluble in, water, but not organic liquids, select an appropriate organic liquid.

7.1.2.3 If the powder is neither reactive nor soluble in water, select distilled or deionized water as the carrier liquid.

7.1.3 Selection of Surfactant—If the powder is not wettable by the chosen carrier liquid, select a suitable surfactant (dispersing agent).

**Note 2**—Ultrasonic energy treatment may be necessary to separate particles so that the individual particles may be wetted by the carrier liquid or liquid/surfactant solution.

**Note 3**—Suggested surfactants are listed in Table 1 and footnotes 4 through 6.

7.1.3.1 The appropriate surfactant and its concentration are determined by trial and error; a series of concentrations of different candidate surfactants must be tried on separate samples and the resultant particle size distribution analyses compared. The optimum surfactant and concentration are usually those that produce the finest particle size distribution results.

**Note 4**—Excess surfactant may cause a coarser particle size distribution in the subsequent particle size analysis.

7.1.4 Dispersion Check:

7.1.4.1 Determine whether the powder is dispersed in the liquid by examining it carefully in a beaker during and after stirring. If the powder appears to be distributed uniformly throughout the liquid, and does not flocculate within a few seconds after the discontinuation of stirring, particle size analysis can then be performed (9.1) and the results evaluated.

7.1.4.2 Ultrasonic Energy Treatment—Even if the powder appears to be uniformly dispersed, ultrasonic energy treatment may be necessary.

**Note 5**—Ultrasonic treatment may also be necessary to break up agglomerates in powders that appear to be dispersed, unless the agglomerate distribution is desired from the subsequent analysis.

7.1.4.3 Disperse the sample by placing the carrier liquid/sample beaker in an ultrasonic bath or by inserting an ultrasonic probe into the liquid/sample mixture. Continuous stirring of the liquid/sample mixture may be necessary through part or all of the ultrasonic treatment. As with surfactant selection (7.1.3.1), the appropriate time and power level for ultrasonic treatment must be determined by trial and error. Select the time and power level by using the minimums necessary to ensure precision and adequate dispersion, as determined in 7.1.4.1. The optimum ultrasonic treatment is usually that which produces the finest particle size distribution results without fracturing the individual particles.

**Note 6**—Particle fracture can be evaluated by examining the treated powder in a suitable microscope and noting whether the particle shape or distribution has changed significantly as the power level or treatment time has been increased. Fracture of particles is also often indicated by a shift from a unimodal to bimodal particle size distribution as the ultrasonic power level or treatment time is increased.

**Note 7**—Some indication of the type of equipment, starting times, and power levels for ultrasonic energy treatment may be obtained from Table 1.

7.1.4.4 Check for dispersion, as in 7.1.4.1. If the powder is now well-dispersed, continue with the particle size analysis (9.1).

7.1.4.5 If the powder is still not well-dispersed after ultrasonic energy treatment, select a different surfactant and repeat the steps given in 7.1.3 and 7.1.4 (and their relevant subparagraphs). Continue with this repetitive process until dispersion is attained.

8. Recommended Dispersion Procedures

8.1 Table 1 lists the dispersion procedures currently in general use for several metals and metal compounds. These procedures have been shown by experience to produce consistent, reproducible particle size analysis results for the materials listed.

9. Particle Size Distribution Analysis

9.1 After dispersion has been achieved by one of the above techniques, immediately perform the required particle size analysis by whatever method is applicable (for example, Test Method B 430, B 761, or B 822).

10. Keywords

10.1 liquid dispersion; metal powders; particle size analysis; powder metallurgy

**SUMMARY OF CHANGES**

Committee B09 has identified the location of selected changes to this standard since the last issue (B 821 – 92(1997)) that may impact the use of this standard.

(1) Paragraphs 4.2, 4.3, and 6.1 were modified to replace the word “shall” with the word “should” in all occurrences. Rationale: This standard is a guide and not a practice. Since a guide is used to recommend a course of action rather than to specify one, each occurrence of the word “shall” was replaced with the word “should.”

(2) Footnote 6 was modified to bring it up to date with the current version of the referenced document. Rationale: The referenced document is no longer available, but a new version contains the pertinent information.
Standard Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering

This standard is issued under the fixed designation B 822; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the particle size distribution by light scattering, reported as volume percent, of particulate materials including metals and compounds.

1.2 This test method applies to analyses with both aqueous and nonaqueous dispersions. In addition, analysis can be performed with a gaseous dispersion for materials that are hygroscopic or react with a liquid carrier.

1.3 This test method is applicable to the measurement of particulate materials in the range of 0.4 to 2000 µm, or a subset of that range, as applicable to the particle size distribution being measured.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 215 Practices for Sampling Finished Lots of Metal Powders

B 243 Terminology of Powder Metallurgy

B 821 Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis

E 1617 Practice for Reporting Particle Size Characterization Data

2.2 ISO Standard:

ISO13320-1 Particle Size Analysis—Laser Diffraction Methods—Part 1: General Principles

3. Terminology

3.1 Definitions—Definitions of powder metallurgy terms

3.2 Definitions of Terms Specific to This Standard:

3.2.1 background—extraneous scattering of light by elements other than the particles to be measured; includes scattering by contamination in the measurement path.

3.2.2 Fraunhofer Diffraction—the optical theory that describes the low-angle scattering of light by particles that are large compared to the wavelength of the incident light.

3.2.3 Mie Scattering—the complex electromagnetic theory that describes the scattering of light by spherical particles. It is usually applied to particles with diameters that are close to the wavelength of the incident light. The real and imaginary indices of light refraction of the particles are needed.

3.2.4 multiple scattering—the rescattering of light by a particle in the path of light scattered by another particle. This usually occurs in heavy concentrations of a particle dispersion.

4. Summary of Test Method

4.1 A prepared sample of particulate material is dispersed in water, or a compatible organic liquid, and circulated through the path of a light beam or some other suitable light source. A dry sample may be aspirated through the light in a carrier gas. The particles pass through the light beam and scatter it. Photodetector arrays collect the scattered light that is converted to electrical signals, which are then analyzed in a microprocessor. The signal is converted to a size distribution using Fraunhofer Diffraction or Mie Scattering, or a combination of both. Scattering information is analyzed assuming a spherical model. Calculated particle sizes are therefore presented as equivalent spherical diameters. Additional information pertaining to the general principles of particle size distribution analysis by light scattering can be found in ISO Standard 13320-1.

5. Significance and Use

5.1 Reported particle size measurement is a function of both the actual particle dimension and shape factor as well as the particular physical or chemical properties being measured. Caution is required when comparing data from instruments operating on different physical or chemical parameters or with...
different particle size measurement ranges. Sample acquisition, handling, and preparation can also affect reported particle size results.

5.1.1 It is important to recognize that the results obtained by this test method, or any other method for particle size determination using different physical principles, may disagree. The results are strongly influenced by the physical principles employed by each method of particle size analysis. The results of any particle sizing method should be used only in a relative sense; they should not be regarded as absolute when comparing results obtained by other methods.

5.2 Light scattering theory has been available for many years for use in the determination of particle size. Several manufacturers of testing equipment now have units based on these principles. Although each type of testing equipment uses the same basic principles for light scattering as a function of particle size, different assumptions pertinent to application of the theory, and different models for converting light measurements to particle size, may lead to different results for each instrument. Therefore, the use of this test method cannot guarantee directly comparable results from different types of instruments.

5.3 Knowledge of the particle size distribution of metal powders is useful in predicting the powder-processing behavior and ultimate performance of powder metallurgy parts. Particle size distribution is related closely to the flowability, moldability, compressibility, and die-filling characteristics of a powder, as well as to the final structure and properties of finished powder metallurgy (P/M) parts.

5.4 This test method is useful to both suppliers and users of powders in determining the particle size distributions for product specifications, manufacturing control, development, and research.

5.5 This test method may be used to obtain data for comparison between lots of the same material or for establishing conformance, as in acceptance testing.

6. Interferences

6.1 Air bubbles entrained in the circulating fluid will scatter light and then be reported as particles. Circulating fluids may not require degassing, but they should be bubble-free upon visual inspection.

6.2 Contaminants, such as nonaqueous solvents, oil, or other organic coatings on the sample, may emulsify in an aqueous carrier, scatter light, and then be reported as part of the particle size distribution. Samples containing such contaminants may be analyzed in a nonaqueous carrier solvent to dissolve the contaminant, or they may be washed free of the contaminant with a compatible aqueous solvent.

6.3 The presence of oil, water, or foreign substances in a gaseous dispersion will cause clogging or agglomeration or will bias the particle size results. The gas supplied should be free of these substances.

6.4 Reagglomeration or settling of particulates during analysis will cause erroneous results. Dispersions shall be prepared in accordance with Guide B 821, and a stable dispersion shall be maintained throughout the analysis.

6.5 Insufficient sample loading may cause electrical noise interference and poor data repeatability. Excessive sample loading may cause excessive light attenuation and multiple scattering, resulting in erroneous particle size distributions.

7. Apparatus

7.1 Particle Size Analyzer, based on Fraunhofer Diffraction or Mie Scattering, or a combination of both light scattering analysis techniques. Care must be taken to ensure that the analyzer system or subsystem is optimum for the size range of the powder being tested.

7.2 Liquid or Gaseous Sample Handling System.

8. Reagents and Materials

8.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society. ⑥ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 Appropriate Application-Specific Carrier, as determined by Guide B 821. The carrier shall meet the following conditions:

8.2.1 It shall be chemically compatible with the construction material of the sample delivery system.

8.2.2 It shall not cause dissolution of the particles, and

8.2.3 It shall be sufficiently clean and non-absorbing to achieve acceptable background levels.

8.3 Antifoaming Agent, or equivalent.

8.4 Dry, Clean Gas, for gaseous dispersions.

8.5 Appropriate Surfactant, as determined by Guide B 821, subject to the conditions listed in 8.2.

9. Sampling and Sample Size

9.1 Obtain a test sample according to Practices B 215. The test portion shall be extracted from the test sample using a microsample splitter; quartering shall not be used.

9.2 The maximum test sample for liquid dispersion will be no more than 25 g. No more than 500 g will be needed for a gaseous dispersion.

10. Calibration and Standardization

10.1 Performance of the instrument is defined by the spacing and position of the optical components (refer to the instruction manual provided by the manufacturer).

10.2 No absolute standards are available for particle size analysis. Diagnostic powders should be available from the equipment manufacturer to ensure consistent instrument functioning.

11. Procedure

11.1 Allow the instrument to warm up for a minimum of 20 min.

⑥ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.
11.2 Install the desired sample delivery system and select the applicable instrument range, as indicated by the instructions provided by the instrument manufacturer.

11.3 Establish correct optical alignment, if necessary, according to the requirements of the manufacturer.

NOTE 1—Optical alignment should be checked upon startup, whenever the sample delivery system is changed, or at least once a day.

11.4 Measure the background in the mode in which the analysis will be conducted. Be sure that the carrier is flowing through the light path while measuring background. Background values shall not exceed the specifications of the manufacturer. If background values exceed the recommendations of the manufacturer, perform the necessary procedures as specified by the manufacturer to bring the background values to within acceptable limits.

11.5 Obtain a representative test sample according to Practices B 215. Extract a test portion from the test sample using a microsample splitter. Refer to the recommendations of the equipment manufacturer to ensure that the amount of the test portion is acceptable to achieve optimum light scattering conditions. A wide range of sample sizes is acceptable, depending on median particle size (50%), particle density (mass/volume), and sample delivery system.

11.6 Select the appropriate run time for the sample. This procedure is very specific to the application and is generally gaged by the run-to-run repeatability.

11.7 Select the desired data output parameters, according to the requirements set forth by the instrument manufacturer.

11.8 For liquid dispersions, disperse the test portion according to the procedure outlined in Guide B 821. Gaseous dispersions require no additional sample preparation.

11.9 Transfer the prepared sample directly to the sample delivery system. In the liquid system, allow circulation for 20 s before measuring. In the dry sampling system, engage the sample switch to allow the sample to begin to flow into the light path while measuring background. Backwash through the light path while measuring background. Backwash with air to remove particles remaining in the sample delivery system.

11.10 Perform the sample analysis according to the instructions of the manufacturer.

11.11 In the liquid system, drain and fill the sample dispersion system in preparation for the next sample analysis. Drain and rinse as necessary, to achieve background values within acceptable operating limits, as specified by the manufacturer.

NOTE 2—When changing from either polar to a nonpolar organic liquid or nonpolar to polar liquid, it will be necessary to rinse the sample delivery system several times with a compatible solvent, such as alcohol, to eliminate the formation of an interference emulsion due to cross contamination of the two carriers.

11.12 In the dry gaseous system, brush or vacuum out all particles throughout the sample system. Purge with air to remove particles remaining in the sample delivery system.

11.13 Repeat Steps 11.5 through 11.11 for additional test portion analyses.

12. Report

12.1 Practice E 1617 specifies three detail levels for reporting particle size characterization data. It is up to the supplier and the user of the data to determine which level of reporting is needed. As a minimum, report the following information:

12.1.1 The instrument name and model number used and the range selected,
12.1.2 The method of dispersing the test portion,
12.1.3 The instrument analysis run time,
12.1.4 Any curve fit models used (where applicable),
12.1.5 The real and imaginary refractive index of the sample material (where applicable),
12.1.6 The real refractive index of the dispersing liquid,
12.1.7 The volume mean diameter,
12.1.8 The differential volume percent versus diameter, and
12.1.9 The cumulative volume percent versus diameter.

13. Precision and Bias

13.1 No statement can be made concerning the precision and bias of this test method at this time. An interlaboratory study is underway.

14. Keywords

14.1 laser diffraction; light scattering; metal powders; particle size distribution; powder metallurgy
1. Scope

1.1 This specification covers a variety of nonferrous powder metallurgy (P/M) structural materials and includes a classification system, or material designation code. With the classification system, this specification includes chemical composition and minimum tensile yield strength.

1.2 The property values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

Note 1—Paragraphs 6.1 and 8.1 govern material classification by the designation code. The classification system is explained in the Appendix.

2. Referenced Documents

2.1 ASTM Standards:
   B 243 Terminology of Powder Metallurgy
   B 328 Test Method for Density, Oil Content, and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings

2.2 Other Standard:
   MPIF Standard 35, Materials Standard for P/M Structural Parts

3. Terminology

3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243. Additional descriptive information is available in the Related Materials section of Vol 02.05 of the Annual Book of ASTM Standards.

4. Ordering Information

4.1 Materials for parts covered by this specification shall be ordered by materials designation code.

4.2 Orders for parts under this specification may include the following information:
   4.2.1 Certification, if required (see Section 10),
   4.2.2 Test methods and mechanical properties other than strength (see 8.2 and 8.3),
   4.2.3 Density (see 7.1),
   4.2.4 Porosity and oil content (see 7.2), and
   4.2.5 Special packaging, if required.

5. Materials and Manufacture

5.1 Structural parts shall be made by pressing and sintering metal powders. Parts may also be made by repressing and resintering sintered parts, if necessary, to produce finished parts in conformance with the requirements of this specification.

6. Chemical Composition

6.1 The material shall conform to the requirements provided in Table 1.

6.2 Chemical analysis, if required, shall be performed by any method agreed upon by the manufacturer and the purchaser.

7. Physical Properties

7.1 Density:
   7.1.1 The buyer and the seller may agree upon a minimum average density for the part and minimum densities for specific regions of the part.
   7.1.2 Density shall be determined in accordance with Test Method B 328.

7.2 Porosity:
   7.2.1 The buyer and the seller may agree upon a minimum volume oil content for parts that are to be self-lubricating.
   7.2.2 The buyer and the seller may agree upon a functional test for porosity in parts that are to be self-lubricating, or for permeability where fluid flow must be restricted.

8. Mechanical Properties

8.1 The minimum guaranteed tensile yield strength, as shown in Table 2, is a numerical suffix to the material designation code and is read as 10^3 psi. The code is adopted from MPIF Standard 35. All tensile yield strengths are defined as the 0.2 % offset yield strengths.

8.2 The purchaser and manufacturer should agree upon the method to be used to verify the minimum strength characteristics of the finished parts. Since it is usually impossible to machine tensile test specimens from these parts, alternative strength tests are advisable. An example would be measuring...
the force needed to break teeth off a gear with the gear properly fixtured.

8.3 The tensile yield strength of the part may be measured indirectly by testing flat tensile bars (See Fig. 1) molded from the same mixed powder lot at the density of the critical region of the parts and then processed along with the parts.

8.4 Transverse rupture strength values can also be related to tensile yield strengths by correlation. While nonferrous P/M materials are technically too ductile for this simple beam test, the test values are reproducible and useful.

9. Sampling

9.1 Lot—Unless otherwise specified, a lot shall consist of parts of the same form and dimensions made from powders of the same composition, molded and processed under the same conditions, and submitted for inspection at one time.

9.2 Chemical Analysis—When requested on the purchase order, at least one sample for chemical analysis shall be taken from each lot. The analysis shall be performed by a mutually agreed upon method.

9.3 Mechanical Tests—The manufacturer and the purchaser shall agree upon a representative number of specimens for mechanical tests.

10. Rejection and Rehearing

10.1 Parts failing to conform to the requirements of this specification may be rejected. Rejection should be reported to the producer or supplier promptly and in writing.

11. Certification

11.1 When specified in the purchase order or contract, a certification from the producer shall be furnished to the purchaser which states that the parts were manufactured, sampled, tested, and inspected in accordance with this specification and have been found to meet its requirements. When specified in the purchase order or contract, a report of the test results shall be furnished.

12. Keywords

12.1 brass; bronze; nickel silver; nonferrous powder metallurgy; nonferrous structural parts; powder metallurgy (P/M); structural parts
X1.1 P/M Material Code Designation:

X1.1.1 The P/M material code designation, or identifying code for structural P/M parts, defines a specific material as to chemistry and minimum strength, expressed in $10^3$ psi (6.895 MPa (6.895 N/mm$^2$)). For example, CZ-1000-9 is a P/M copper zinc material containing nominal 90% copper and 10% zinc. It has a minimum yield strength of $9 	imes 10^3$ psi (9000 psi) in the as-sintered condition.

X1.1.2 The system offers a convenient means of designating both the chemistry and minimum strength value of any standard P/M material. For each standard material, the density is given as one of the typical values and is no longer a requirement of the specification.

X1.1.3 Code designations in this specification and revisions thereof apply only to P/M materials for which specifications have been adopted. In order to avoid confusion, the P/M material designation coding system is intended for use only with such materials, and it should not be used to create nonstandard materials. The explanatory notes, property values, and other contents of this specification have no application to any other materials.

X1.1.4 In the coding system, the prefix letters denote the general type of material. For example, the prefix CZ represents copper (C) and zinc (Z), which is known as brass. The prefix letter codes are as follows:

- X1.1.4.1 C = copper (Cu),
- X1.1.4.2 CT = bronze (Cu-Sn),
- X1.1.4.3 CNZ = nickel silver (Cu-Ni-Zn),
- X1.1.4.4 CZ = brass (Cu-Zn),
- X1.1.4.5 N = nickel (Ni),
- X1.1.4.6 P = lead (Pb), and
- X1.1.4.7 T = tin (Sn).

X1.2 Prefix and Four-Digit Code—The four digits following the prefix letter code refer to the composition of the material. In nonferrous materials, the first two numbers in the four-digit series designate the percent of the major alloying constituent; the last two numbers of the four digit series designate the percent of the minor alloying constituent. For improved machinability, lead is sometimes the third alloying element in a nonferrous alloy system. Lead will then be indicated only by the letter “P” in the prefix. The percent of lead or any other minor alloying element that happens to be excluded from the four-digit nomenclature is represented in the “chemical composition” information that appears with each standard material. For an illustration of P/M nonferrous material designation coding, see Fig. X1.1.

X1.3 Suffix Digit Code—The two-digit suffix represents the minimum strength value, expressed in $10^3$ psi (6.895 MPa (6.895 N/mm$^2$)), that the user can expect from the P/M material possessing that chemistry. The minimum tensile yield strength for these materials in the as-sintered condition is given in Table X1.1.

X1.4 Data Source—Information used in compiling this specification was contributed by the membership of ASTM Committee B09 on Metal Powders and Metal Powder Products and the Standards Committee of the Powder Metallurgy Parts Association of MPIF. These technical data are on file at MPIF Headquarters, Princeton, NJ and are reproduced in this specification with the permission of the Metal Powder Industries Federation.

![Fig. X1.1 Illustration of Powder Metallurgy Nonferrous Material Designation Coding](image)
### TABLE X1.1 Nonferrous Alloys: Brass, Bronze, and Nickel Silver

This table provides powder metallurgy material properties for various nonferrous alloys. The data includes minimum and typical values for tensile properties, transverse rupture strength, unnotched Charpy impact energy, density, and compressive yield strength. The hardness is also listed for each material.

#### Minimum Values

<table>
<thead>
<tr>
<th>Material Designation Code</th>
<th>Minimum Strength, Yield, $10^3$ psi</th>
<th>Ultimate Strength, $10^3$ psi</th>
<th>Yield Strength, 0.2%, $10^3$ psi</th>
<th>Elongation, in 1 in., %</th>
<th>Young's Modulus, $10^6$ psi</th>
<th>Poisson's Ratio</th>
<th>Transverse Rupture Strength, $10^3$ psi</th>
<th>Unnotched Charpy Impact Energy, ft-lb</th>
<th>Density, g/cm$^3$</th>
<th>Compresive Yield Strength, 0.1%, $10^3$ psi</th>
<th>Hardness</th>
</tr>
</thead>
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<tr>
<td>CZ-1000-9</td>
<td>9</td>
<td>18.0</td>
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<td>9.0</td>
<td>11.5</td>
<td>0.31</td>
<td>39</td>
<td>15.0</td>
<td>7.60</td>
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<td>65HRH</td>
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<td>11.0</td>
<td>10.5</td>
<td>13.0</td>
<td>0.31</td>
<td>46</td>
<td>24.0</td>
<td>7.90</td>
<td>12</td>
<td>72</td>
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<tr>
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<td>23.0</td>
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<td>31.0</td>
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<td>80</td>
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<td>CZP-1002-7</td>
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<td>10.0</td>
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<td>10</td>
<td>66HRH</td>
</tr>
<tr>
<td>CZ-2000-11</td>
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<td>23.0</td>
<td>13.5</td>
<td>9.0</td>
<td>13.0</td>
<td>0.31</td>
<td>52</td>
<td>27.0</td>
<td>7.60</td>
<td>12</td>
<td>73</td>
</tr>
<tr>
<td>CZ-2000-12</td>
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<td>13.5</td>
<td>9.0</td>
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<td>52</td>
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<td>12</td>
<td>73</td>
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<tr>
<td>CZP-2002-11</td>
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<tr>
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<td>12</td>
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<tr>
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<td>14</td>
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<td>16.0</td>
<td>13.0</td>
<td>0.31</td>
<td>71</td>
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<td>8.00</td>
<td>15</td>
<td>88</td>
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<tr>
<td>CNZ-1818-17</td>
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<td>34.0</td>
<td>20.0</td>
<td>11.0</td>
<td>13.5</td>
<td>0.31</td>
<td>73</td>
<td>24.0</td>
<td>7.90</td>
<td>24</td>
<td>90HRH</td>
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<tr>
<td>CNZP-1816-13</td>
<td>13</td>
<td>26.0</td>
<td>15.0</td>
<td>10.0</td>
<td>13.5</td>
<td>0.31</td>
<td>50</td>
<td>22.0</td>
<td>7.90</td>
<td>18</td>
<td>86HRH</td>
</tr>
<tr>
<td>CT-1000-13 (repressed)</td>
<td>13</td>
<td>22.0</td>
<td>16.0</td>
<td>4.0</td>
<td>8.5</td>
<td>0.31</td>
<td>45</td>
<td>4.0</td>
<td>7.20</td>
<td>20</td>
<td>82HRH</td>
</tr>
</tbody>
</table>

### Notes

- **Note 1:** $10^3$ psi = 6.895 MPa (6.895 N/mm$^2$).
- **Note 2:** 1 in. = 25.4 mm.
- **Note 3:** 1 ft-lb = 1.356 J.

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Standard Guide for
Electroforming with Nickel and Copper\textsuperscript{1}

This standard is issued under the fixed designation B 832; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (\(\epsilon\)) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers electroforming practice and describes the processing of mandrels, the design of electroformed articles, and the use of copper and nickel electroplating solutions for electroforming.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- B 183 Practice for Preparation of Low-Carbon Steel for Electroplating\textsuperscript{2}
- B 242 Practice for Preparation of High-Carbon Steel for Electroplating\textsuperscript{2}
- B 252 Practice for Preparation of Zinc Alloy Die Castings for Electroplating and Conversion Coatings\textsuperscript{2}
- B 253 Guide for Preparation of Aluminum Alloys for Electroplating\textsuperscript{2}
- B 254 Practice for Preparation of and Electroplating on Stainless Steel\textsuperscript{2}
- B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings\textsuperscript{2}
- B 311 Test Method for Density Determination for Powder Metallurgy (P/M) Materials Containing Less Than Two Percent Porosity\textsuperscript{2}
- B 343 Practice for Preparation of Nickel for Electroplating with Nickel\textsuperscript{2}
- B 374 Terminology Relating to Electroplating\textsuperscript{2}
- B 489 Practice for Bend Test for Ductility of Electrodeposited and Autocatalytically Deposited Metal Coatings on Metals\textsuperscript{2}
- B 490 Practice for Micrometer Bend Test for Ductility of Electrodeposits\textsuperscript{2}


\textsuperscript{2} Annual Book of ASTM Standards, Vol 02.05.

3. Summary of Electroforming Practice

3.1 Electroforming is defined (see Terminology B 374) as the production or reproduction of articles by electrodeposition upon a mandrel or mold that is subsequently separated from the deposit.

3.2 The basic fabrication steps are as follows: a suitable mandrel is fabricated and prepared for electroplating; the mandrel is placed in an appropriate electroplating solution and metal is deposited upon the mandrel by electrolysis; when the required thickness of metal has been applied, the metal-covered mandrel is removed from the solution; and the mandrel is separated from the electrodeposited metal. The electroform is a separate, free-standing entity composed entirely of electrodeposited metal. Electroforming is concerned with the fabrication of articles of various kinds.

4. Significance and Use

4.1 The specialized use of the electroplating process for electroforming results in the manufacture of tools and products that are unique and often impossible to make economically by traditional methods of fabrication. Current applications of nickel electroforming include: textile printing screens; components of rocket thrust chambers, nozzles, and motor cases; molds and dies for making automotive arm-rests and instrument panels; stampers for making phonograph records, videodiscs, and audio compact discs; mesh products for making...
porous battery electrodes, filters, and razor screens; and optical parts, bellows, and radar wave guides (1-3).  

4.2 Copper is extensively used for electroforming thin foil for the printed circuit industry. Copper foil is formed continuously by electrodeposition onto rotating drums. Copper is often used as a backing material for electroformed nickel shells and in other applications where its high thermal and electrical conductivities are required. Other metals including gold are electroformed on a smaller scale.

4.3 Electroforming is used whenever the difficulty and cost of producing the object by mechanical means is unusually high; unusual mechanical and physical properties are required in the finished piece; extremely close dimensional tolerances must be held on internal dimensions and on surfaces of irregular contour; very fine reproduction of detail and complex combinations of surface finish are required; and the part cannot be made by other available methods.

5. Processing of Mandrels for Electroforming

5.1 General Considerations:

5.1.1 Mandrels may be classified as conductors or nonconductors of electricity, and each of these may be permanent, semipermanent, or expendable (Table 1).

<table>
<thead>
<tr>
<th>TABLE 1 Types of Mandrel Materials</th>
</tr>
</thead>
<tbody>
<tr>
<td>Types</td>
</tr>
<tr>
<td>Conductors</td>
</tr>
<tr>
<td>Expendable</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Permanent</td>
</tr>
<tr>
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<tr>
<td></td>
</tr>
<tr>
<td>Nonconductors</td>
</tr>
<tr>
<td>Expendable</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>Permanent (or Semi-Permanent)</td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

5.1.2 Whether or not a mandrel is a conductor will determine the procedures required to prepare it for electroforming. Conductive mandrels are usually pure metals or alloys of metals and are prepared by standard procedures but may require an additional thin parting film to facilitate separation of the electroform from the mandrel (unless the mandrel is removed by melting or chemical dissolution).

5.1.3 Whether or not a permanent or expendable mandrel should be used is largely dependent on the particular article that is to be electroformed. If no reentrant shapes or angles are involved, it is possible to use permanent, rigid mandrels that can be separated from the finished electroform mechanically and reused. If reentrant angles and shapes are involved, it is necessary to use mandrel materials that can be removed by melting or by chemical dissolution, or materials that are collapsible, such as polyvinyl chloride and other plastics. In some cases, multiple piece mandrels are used that can be removed even with reentrant features.

5.1.4 Many solid materials can be used to fabricate mandrels for electroforming, but the following generalizations may help in selecting a suitable material: permanent mandrels are preferred for accuracy and for large production runs; expendable mandrels must be used whenever the part is so designed that a permanent mandrel cannot be withdrawn; and it is important that the mandrel retain its dimensional stability in warm plating baths. Wax and most plastics expand when exposed to electroplating solutions operated at elevated temperatures. In such cases, it may be necessary to use acid copper, nickel sulfamate, and other electroplating solutions that function at room temperature.

5.2 Mandrel Design:

5.2.1 The electroforming operation can often be simplified by design changes that do not impair the functioning of the piece. Some of the design considerations are summarized in 5.2.2, 5.2.3, 5.2.4, 5.2.5, and 5.2.6. Examples of mandrel shapes that may present problems during electroforming are illustrated in Fig. 1.

5.2.2 Exterior (convex) angles should be provided with as generous a radius as possible to avoid excessive build up and treeing of the deposit during electroforming. Interior (concave) angles on the mandrel should be provided with a fillet radius of at least 0.05 cm per 5 cm (0.02 in. per 2 in.) of length of a side of the angle.

5.2.3 Whenever possible, permanent mandrels should be tapered at least 0.08 mm per m (0.001 in. per ft) to facilitate removal from the mandrel. (Where this is not permissible, the mandrel may be made of a material with a high or low coefficient of thermal expansion so that separation can be effected by heating or cooling).

5.2.4 A fine surface finish on the mandrel, achieved by lapping or by electropolishing, will generally facilitate separation of mandrel and electroform. A finish of 0.05 µm (2 µin.) rms is frequently specified.

5.2.5 Flat bottom grooves, sharp angle indentations, blind holes, fins, v-shaped projections, v-bottom grooves, deep scoops, slots, concave recesses, and rings and ribs can cause problems with metal distribution during electroforming unless inside and outside angles and corners are rounded.

5.2.6 An engineering drawing of the mandrel, the electroformed article, and auxiliary equipment or fixture for separating the electroform from the mandrel should be prepared. The drawing of the mandrel should provide for electrical connections to be made in nonfunctional areas of the electroform. It should provide reference points for and mechanical means of holding if finish machining is necessary before removal of the mandrel.

5.3 Mandrel Fabrication:

5.3.1 The method of fabrication of the mandrel will depend on the type selected, the material chosen, and the object to be
5.4 Preparing Non-Conducting Mandrels:
5.4.1 Nonconducting mandrels must be made impervious to water and other processing solutions and then rendered conductive. Porous materials, for example, leather and plastic, may be impregnated with wax, shellac, lacquer, or a synthetic resin formulation. It is often preferable to use thin films of lacquer to seal porous, nonmetallic mandrels.
5.4.2 Nonconducting materials may be rendered conductive by applying a chemically reduced film of silver, copper, or nickel to the surface. In general, these processes are carried out by spraying the reagent containing the metal ions of choice simultaneously with a specific reducing agent onto the surface of the mandrel using a double-nozzle spray gun. The chemicals react at the surface; the metal is reduced and is deposited on the mandrel surface. Chemical reduction processes are preferred because dimensional accuracy is not affected, the film has little adhesion, and parting is not difficult. If necessary, a silver film can be stripped from a nickel electroform with either nitric acid, warm sulfuric acid, or a cyanide solution.
5.4.3 Other ways of making non-conducting materials conductive include: using finely divided metal powders dispersed in binders ("bronzing"), applying finely divided graphite to wax, and to natural or synthetic rubbers that have an affinity for graphite, and applying graphite with a binder.
5.4.4 Vapor deposition of silver and other metals is preferred for nonconducting mandrels used in the semiconductor industry, the optical disc industry, and the manufacture of holograms. In these cases the mandrel must be made of a material that does not outgas in the vacuum chamber. Glass is the preferred substrate for making masters and stampers for optical read-out discs of all kinds.
5.5 Preparing Metallic Mandrels:
5.5.1 Standard procedures should be used whenever adherent electrodeposits are applied to metallic mandrels prior to and in preparation for electroforming. See Practices B 183, B 242, B 254, B 281, and B 558, for example.
5.5.2 With most metallic mandrels an additional chemical treatment that forms a parting film on the surface is required to separate the electroform from the mandrel. After removing all traces of grease and oil by means of solvents, various metallic mandrels are given different treatments for this purpose (see 5.5.3, 5.5.4, 5.5.5, 5.5.6, and 5.5.7).

Note—Examples of deposit distribution on contours that require special consideration are shown in an exaggerated fashion. The designer should confer with the electroformer before designing an electroform having any of these contours. An experienced electroformer can minimize some of the exaggeration shown.

FIG. 1 Examples of Deposit Distribution on Electroforms
5.5.3 Stainless steel, nickel, and nickel- or chromium-plated steel are cleaned using standard procedures, rinsed, and passivated by immersion in a 2 % solution of sodium dichromate for 30 to 60 s at room temperature. The mandrel must then be rinsed to remove all traces of the dichromate solution.

5.5.4 Copper and brass mandrels that have been nickel and/or chromium-plated may be treated as described in 5.5.3. If not electroplated, the surface can be made passive by immersion in a solution containing 8 g/L sodium sulfide.

5.5.5 Aluminum alloys may require special treatments even when they are used as expendable mandrels to be separated by chemical dissolution. If the deposits are highly stressed, it may be necessary to use the zincate or stannate treatments included in Guide B 253 to achieve a degree of adhesion that will prevent lifting of the deposit from the mandrel. When low-stressed deposits (near zero) are being produced, treatment of the aluminum by degreasing, cathodic alkaline cleaning, and immersion in a 50 % solution of nitric acid may be sufficient.

5.5.6 Zinc and its alloys may require no other preparation than conventional cleaning if used for expendable mandrels and are to be parted by chemical dissolution. In the case of nickel electroforming, it is necessary to electroplate the zinc alloy with copper and treat it accordingly to prevent attack of the mandrel. See Practice B 252.

5.5.7 The low-melting point alloys included in Table 1 employed to make expendable mandrels that can be melted away have a tendency to leave a residue of tin on the surface of the electroform. The mandrel can be plated with copper prior to electroforming to prevent this.

6. Nickel and Copper Electroforming Solutions

6.1 The choice of metal selected for the electroform will depend on the mechanical and physical properties required in the finished article as related to function. The two metals selected most frequently are nickel and copper. The operation and control of nickel and copper electroforming solutions are described in this section.

6.2 The nickel electroplating solutions commonly used for electroforming are Watts and nickel sulfamate with and without addition agents. The advantages of nickel electroforming from sulfamate solutions are the low internal stress of the deposits and the high rates of deposition that are possible. The important copper electroforming solutions are copper sulfate and copper fluoborate. The formulations of nickel electroforming solutions, typical operating conditions, and typical mechanical properties of the deposits are given in Table 2. Similar information for copper electroforming is given in Table 3.

6.3 Watts Solutions—The Watts bath contains nickel sulfate, nickel chloride, and boric acid and yields nickel deposits that are matte in appearance and that are tensively stressed. The solution is relatively inexpensive and is successfully used for electroforming. Nickel sulfate is the main source of nickel ions. Nickel chloride increases solution conductivity and has a beneficial effect on the uniformity of metal distribution at the cathode. Boric acid acts as a buffer to control pH at the cathode-solution interface. Antipitting agents (wetting agents) are essential for avoiding pitting due to the clinging of air and hydrogen bubbles. With care, the internal stress of the electroformed nickel can be controlled by means of organic addition agents. See 8.3.

6.4 Nickel Sulfamate Solutions—A formulation for nickel sulfamate solutions is included in Table 2. These are analogous to Watts solutions in which the nickel sulfate is replaced with nickel sulfamate. The internal stress is lower than in the Watts solutions.
solution, as indicated by the information on mechanical properties in Table 2. The zero stress level may be obtained by maintaining the solution in a high state of purity and by eliminating the nickel chloride. To minimize hydrolysis of nickel sulfamate and the formation of sulfate and ammonium ions, these solutions may be operated above pH 3.5 and below 50°C. To ensure efficient dissolution of nickel anodes in the absence of chlorides, it is essential to use sulfur-activated nickel anode materials. A stable tensile stress can be maintained in nickel sulfamate solutions by including nickel chloride in the formulation, by using an adequate anode area (1.5 to 2 times the area of the cathode), and by using a fully-active nickel anode material to maintain the potential on the anode basket as low as possible, thus avoiding oxidation of the sulfamate anion (see 8.3.5). Under these conditions the stress normally is about 35 MPa (5000 psi) tensile for a well-worked solution.

6.5 Copper Electroforming Solutions—Copper sulfate solutions are used more often than copper fluoroborate solutions. The internal stress of copper deposits is generally lower than that of electrodeposited nickel. One of the features of the fluoroborate solution is the ability to electrodeposit copper at high current densities.

7. Properties of Electrodeposited Nickel and Copper

7.1 The mechanical properties—tensile strength, elongation, hardness, and internal stress—are influenced by the operational variables including temperature, pH, and current density, and by solution composition (4). The qualitative effects of these variables on the mechanical properties of nickel and copper electrodeposits are summarized in Table 4, Table 5, Table 6, and Table 7. Relatively small amounts of metallic impurities in solution can also affect mechanical properties. The properties are interrelated, and steps taken to increase the hardness of the deposit usually increase its strength and lower its ductility. The refinement of crystal structure, for example by the use of organic addition agents, is accompanied by increased hardness and tensile strength and reduced ductility. Typical properties of deposits from various additive-free baths are included in Table 2 and Table 3. See Refs. 5-7.

7.2 The mechanical properties, especially the percent elongation or ductility, may be affected by the thickness of the electrodeposited metal used in determining the properties. For example, the ductility of nickel increases with increasing thickness up to about 250 µm after which it becomes relatively constant (8). Mechanical testing should be done at the thickness of interest even though it may be more convenient to test thick deposits.

8. Control of Electroforming Processes

8.1 Successful electroforming requires careful control of the purity of the electrolyte and of the operating variables, such as pH, current density, temperature, and agitation. The common problems encountered in electroforming include controlling metal distribution, internal stress, roughness, and nodule formation. Addition agents may help overcome some of these problems, but their concentrations must be closely controlled.

8.2 Metal Distribution:

8.2.1 The variation of the thickness of the metal deposited at various points on the surface of a mandrel is related to current distribution. Recessed areas will receive less current; areas that project from the surface will receive higher current. The current density and the rate of metal deposition will be lower in recessed areas than at areas which project from the surface. The result is that metal distribution will be nonuniform in many cases. The deposit will be relatively thin in recessed areas and relatively thick on projections.

8.2.2 Metal distribution is improved by proper racking and by the use of thieves, shields, and/or conforming or auxiliary anodes. The use of these processing aids makes it possible to control metal distribution and obtain relatively uniform deposits.

8.3 Internal Stress:

8.3.1 The control of internal stress is extremely important in electroforming because of the deliberately low adhesion between the electroform and the mandrel. Internal stress refers to forces created within an electrodeposits as a result of the electrocrystallization process and/or the codeposition of impurities such as hydrogen, sulfur, and other elements. The forces are either tensile (contractile) or compressive (expansive) in nature; rarely are electrodeposits free of some degree of internal stress. Internal stress may be measured in accordance with Test Method B 636.

8.3.2 Excessive tensile or compressive stress can cause the following problems: distortion of the electroform when it is separated from the mandrel; difficulty of separating the electroform from the mandrel; curling, peeling, or separation of the electroform prematurely from the mandrel; and buckling and blistering of the deposit.

8.3.3 Internal stress is influenced by the nature and composition of the electroplating solution. Typical values of internal stress for electroforming solutions are given in Table 8.

8.3.4 Typical stress reducers for nickel electroforming are saccharin, para-toluene sulfonamide, meta-benzene disulfonate, and 1-3-6 sodium naphthalene trisulfonate. All of these
organic stress-reducing agents introduce sulfur into nickel deposits, and this limits the temperature at which the electroform can be used in service. Nickel electrodeposits with small amounts of sulfur may become embrittled when heated to temperatures above 200°C. The exact temperature of embrittlement depends on the sulfur content, the time at the elevated temperature, and other factors. Control of internal stress by means of organic addition agents requires an optimum level of the additive, regular replenishment as it is consumed, and frequent (or continuous) carbon treatment to control the concentration of decomposition products that form as a result of reduction of the additive at the cathode.

8.3.6 The use of levelling agents, such as 2 butyne 1:4 diol, for nickel electroforming can improve metal distribution on the mandrel by suppressing the growth of nodules and by preventing the formation of a plane of weakness when electroforming into a corner. In general, levelling agents increase internal stress in the tensile direction. Although the breakdown products formed by organic addition agents generally increase internal stress, continuous filtration through carbon removes only the breakdown products in the case of butyne diol, and the stress can be closely controlled with this additive.

8.4 Roughness:

8.4.1 Any condition which would tend to cause roughness in decorative plating will have a much more serious effect on electroforming operations. Nodules, nuggets, and trees will form. These become high current density areas, and the larger
they get, the faster they grow, and the more they rob surrounding areas of deposit. As a consequence, the filtration rates used in electroforming are very high in an effort to prevent roughness; the rates may amount to passing the entire solution through a filter several times an hour.

8.4.2 The sources of roughness include airborne dirt, anode particles, crystallized salts that fall into the electroplating solution, and particles which precipitate from hard water constituents. Good housekeeping can eliminate most sources of roughness.

8.5 **Treeing**:

8.5.1 Treeing at edges and corners may be troublesome and is minimized by the use of shields. Certain addition agents, such as the levelling agents discussed in 8.3.6, suppress the treeing tendency. Another approach applicable in many cases is to extend the mandrel beyond the dimensions actually desired so that the treeing occurs on a part of the electroform that can be machined away. If nickel electroforming is interrupted to remove trees and nodules by machining, the machined nickel surface must be activated to insure good nickel-to-nickel adhesion. Methods of preparing nickel surfaces for deposition with nickel have been standardized (see Practice B 343).

8.6 **Other Control Techniques**:

8.6.1 Agitation of every kind, singly or in combination, should be employed whenever possible to control burning and pitting at high current density sites. Solution agitation, either air or mechanical, may induce roughness, however, unless the solution is kept clean by using a high filtration rate. Cathode rotation, when applicable, is an effective means of solution agitation.

9. **Post-Electroforming Operations**

9.1 The operations that are performed after electroforming is completed are: machining and final finishing of the electroform, parting or separation from the mandrel, and backing the electroform.

9.2 **Machining and Finishing**:

<table>
<thead>
<tr>
<th>Table 6</th>
<th>Variables that Affect Mechanical Properties of the Deposit—Watts Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Property</td>
<td>Operational Solution Composition</td>
</tr>
</tbody>
</table>
| Tensile Strength | Relatively independent of plating solution temperature within range suggested.  
Relatively independent of changes in cathode current density.  
Relatively independent of pH variation within range suggested. | Increases with increasing nickel content.  
Increases with increasing chloride content. |
| Elongation | Increases with temperature to 55°C followed by slight decrease at higher temperature.  
Relatively independent of pH variation within range suggested. | Decreases with increasing nickel content. |
| Hardness | Decreases with temperature rise to 55°C but increases with higher temperature.  
Decreases significantly with increasing cathode current density to 5.4 A/dm². At higher current densities the hardness increases with increasing current density. | Increases with increasing nickel content.  
Increases with increasing chloride content. |
| Internal Stress | Relatively independent of plating solution temperature.  
Decreases slightly, then increases with increasing cathode current density.  
Relatively independent of pH variation within range suggested. | Increases slightly with increasing nickel content.  
Increases markedly with increasing chloride content. |

<table>
<thead>
<tr>
<th>Table 7</th>
<th>Variables that Affect Mechanical Properties of the Deposit—Nickel Sulfamate Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Property</td>
<td>Operational Solution Composition</td>
</tr>
</tbody>
</table>
| Tensile Strength | Decreases with increasing temperature to 49°C, then increases slowly with further temperature increase.  
Increases with increasing pH.  
Decreases with increasing current density. | Decreases slightly with increasing nickel content. |
| Elongation | Decreases as the temperature varies in either direction from 43°C.  
Decreases with increasing pH.  
Increases moderately with increasing current density. | Increases slightly with increasing nickel content.  
Increases slightly with increasing chloride content. |
| Hardness | Increases with increasing temperature within operating range suggested.  
Increases with increasing solution pH.  
Reaches a min at about 13 A/dm². | Decreases slightly with increasing concentration of nickel ion.  
Decreases slightly with increasing chloride content. |
| Internal Stress | Decreases with increasing solution temperature.  
Reaches a min at pH 4.0–4.2 with a nickel metal concentration of 76.5 g/L. Reaches a minimum at 3.0–3.2 with a nickel metal concentration of 107 g/L.  
Increases with increasing current density. | Depends on nickel metal content in the solution (see effect of pH).  
Increases significantly with increasing chloride content. |

<table>
<thead>
<tr>
<th>Table 8</th>
<th>Typical Values of Internal Stress for Electroforming Solutions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electroforming Solution</td>
<td>Internal Stress, MPa^A</td>
</tr>
<tr>
<td>Watts nickel</td>
<td>110–210</td>
</tr>
<tr>
<td>Nickel sulfamate, no chloride</td>
<td>0–55</td>
</tr>
<tr>
<td>Nickel sulfamate, with chloride (0–10 g/L)</td>
<td>20–70</td>
</tr>
<tr>
<td>Copper sulfate</td>
<td>0–14</td>
</tr>
<tr>
<td>Copper fluoborate</td>
<td>0–20</td>
</tr>
</tbody>
</table>

^A Positive values are tensile.
9.2.1 Necessary machining or other mechanical finishing operations are usually performed before the electroform is separated from the mandrel to avoid deformation. The machining and grinding of electrodeposited nickel may be difficult. Directions for machining and grinding of nickel and other electrodeposits have been published (9).

9.3 Parting:

9.3.1 Electroforms are removed from permanent mandrels mechanically by the use of one or a combination of several of the following techniques:

9.3.1.1 Impact, by a sudden pull or hammer blow.
9.3.1.2 Gradual Force, applied by a hydraulic ram to push, or a jack-screw or wheel-puller to pull the pieces apart.
9.3.1.3 Cooling, for example with a mixture of dry ice and naphtha. This works best if the mandrel has a lower coefficient of expansion than the electrodeposited. On withdrawal from the cold bath, the electroform will expand faster than the mandrel, permitting separation.
9.3.1.4 Heating, with a torch or hot water or oil bath, either to melt or soften a parting compound or to take advantage of a difference in coefficients of expansion between mandrel and electroform.
9.3.1.5 Prying, with a sharp tool may be used with care to separate relatively flat pieces, such as phonograph record stampers or engraving plates.

9.3.2 Expendable mandrels are melted or dissolved out as follows:

9.3.2.1 Zinc alloys are dissolved with hydrochloric acid.
9.3.2.2 Aluminum alloys are dissolved in strong, hot sodium hydroxide solutions.
9.3.2.3 Low-melting alloys are melted and shaken out. The alloy may be collected and used over. If “tinning” occurs, a nickel electroform may be cleaned with strong nitric acid.
9.3.2.4 Thermoplastics may be softened by heat so that the bulk of the mandrel may be withdrawn, after which the electroform is cleaned with a suitable solvent. An alternative is to dissolve the entire mandrel with a solvent.

9.3.3 The separation of mandrel and electroform should be considered at an early stage since the separation can be simplified by certain design changes. A fine surface finish facilitates parting. Gripping devices may be incorporated on the mandrel, and a knock-off block may be provided so force can be applied for separating the mandrel and the electroform. A taper can be specified when feasible.

9.4 Backing the Electroform:

9.4.1 It is often necessary to back the electroform with some other material, which is then finished to specified dimensions to fit into a bolster or onto a printing press. This is true, for example, in the case of molds, dies, printing plates, and tools in general.

9.4.2 The most important backing methods include the following: casting with low-melting temperature alloys, spraying with various materials, electroplating with other metals, use of thermosetting resins, and spark-eroded steel back-ups and electrochemical machining techniques that sink conforming cavities in the back-up material.

10. Product Requirements and Test Procedures

10.1 No single statement of requirements can be written that applies to all electroformed articles. Each electroform is unique and has its own particular set of functional requirements. The following should be considered in developing detailed requirements and test procedures.

10.2 The electrodeposited metal or alloy should be specified including any known detrimental effects of impurities. Chemical composition can be determined by common analytical procedures.

10.3 The density of electroformed materials is often an indication of its porosity compared to wrought materials of the same composition. Values of density for metals are listed in many handbooks. The density may be measured by the method described in Test Method B 311 and should be at least 99 % of the value of the wrought material.

10.4 The mechanical properties, tensile strength, yield strength, and elongation should be specified if applicable. Special test specimens prepared before, during, and after electroforming may be tested by standard uniaxial tension testing, in accordance with Test Methods E 8, to certify properties. Other test methods are included in Section 2 and may be applicable. For example, qualitative methods for measuring ductility (elongation) are given in Practices B 489 and B 490. In critical cases electroformed prototypes should be tested. The mechanical properties should be determined at the specified thickness even though it might be more convenient to measure thinner electrodeposits.

10.5 Hardness may be specified in certain applications and can be measured by the test methods given in Test Methods B 578 and E 384.

10.6 Thickness is often an important dimension of the electroform. It should be specified and measured using standard inspection tools of appropriate accuracy. Guide B 659 is a guide to coating thickness measuring methods.

10.7 The appearance of the electroform, including surface finish, should be specified. The initial layers of electrodeposited metal will generally reproduce the finish on the mandrel with great fidelity and hence, the appearance and finish on the mandrel must also be specified. The appearance and finish of the surface farthest from the mandrel (the back of the electroform) may be important in some applications and should be specified when appropriate. Roughness on the back of the electroform may be controlled by the use of leveling agents, but in other cases the methods discussed in 8.4 may be applied. Cracks, pits, voids, and inclusions are often detrimental and must be controlled by visual inspection, fluoroscopic testing, dye penetrant inspection, and other techniques.

10.8 Adhesion may be specified in those cases where the electroform is comprised of two or more layers of electrodeposited metals. Test methods for determining adhesion qualitatively are given in Test Methods B 571.

10.9 Electrical conductivity should be specified when it is a requirement.

10.10 Requirements for high- or low-temperature performance should be known and specified. The properties of electrodeposited metals are influenced by variations in temperature (7, 8).
11. Keywords

11.1 copper; copper electroforming; electroforming; electroplating; mandrels; nickel; nickel electroforming

REFERENCES


INTRODUCTION

When atomic hydrogen enters steels and certain other alloys, it can cause loss of ductility or load carrying ability or cracking (usually as submicroscopic cracks), or catastrophic brittle failures at applied stresses well below the yield strength or even the normal design strength for the alloys. This phenomenon often occurs in alloys that show no significant loss in ductility, when measured by conventional tensile tests, and is frequently referred to as hydrogen-induced delayed brittle failure, hydrogen stress cracking, or hydrogen embrittlement. The hydrogen can be introduced during cleaning, pickling, phosphating, electroplating, autocatalytic processes, and in the service environment as a result of cathodic protection reactions or corrosion reactions. Hydrogen can also be introduced during fabrication, for example, during roll forming, machining, and drilling due to lubricant breakdown as well as during welding or brazing operations.

1. Scope

1.1 This test method covers the determination of, on a statistical basis, the probability of the existence of hydrogen embrittlement or degradation in:

1.1.1 A batch of barrel electroplated, autocatalytic plated, phosphated, or chemically processed threaded articles or fasteners and

1.1.2 A batch of rack plated threaded articles, fasteners, or rod.

1.2 Industrial practice for threaded articles, fasteners, and rod has evolved three graduated levels of test exposure to ensure reduced risk of hydrogen embrittlement (see Section 3). These levels have evolved from commercial applications having varying levels of criticality. In essence, they represent the confidence level that is required. They also represent the time that finished goods are held before they can be shipped and used. This time equates to additional cost to the manufacturer that may of necessity be added to the cost of the finished goods.

1.3 This test method is applicable to threaded articles, fasteners, and rod made from steel with ≥1000 MPa (with corresponding hardness values of 300 HV₁₀㎏/f, 303 HB, or 31 HRc) or surface hardened threaded articles, fasteners, or rod.

1.4 This test method shall be carried out after hydrogen embrittlement relief heat treatment in accordance with the requirements of Guide B 850. It may also be used for assessing differences in processing solutions, conditions, and techniques. This test method has two main functions: first, when used with a statistical sampling plan it can be used for lot acceptance or rejection, and second, it can be used as a control test to determine the effectiveness of the various processing steps including pre- and post-baking treatments to reduce the mobile hydrogen in the articles, fasteners, or rod. While this test method is capable of indicating those items that are embrittled to the extent defined in Section 3, it does not guarantee complete freedom from embrittlement.

1.5 This test method does not relieve the processor from imposing and monitoring suitable process control.

1.6 This test method has been coordinated with ISO/DIS 10587 and is technically equivalent. (Warning—Great care should be taken when applying this test method. The heads of embrittled articles, fasteners, or rod may suddenly break off and become flying projectiles capable of causing blindness or other serious injury. This hazard can occur as long as 200 h...
after the test has started. Hence, shields or other apparatus should be provided to avoid such injury.)

Note 1—Test Method F 1940 can be used as a process control and verification to prevent hydrogen embrittlement in fasteners covered by this test method.

Note 2—The use of inhibitors in acid pickling baths does not necessarily guarantee avoidance of hydrogen embrittlement.

1.7 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards: 2
   B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
   B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
   F 436 Specification for Hardened Steel Washers
   B 850 Guide for Post-Coating Treatments of Steel for Reducing Risk of Hydrogen Embrittlement
   F 1940 Test Method for Process Control Verification to Prevent Hydrogen Embrittlement in Plated or Coated Fasteners

2.2 ISO Standards:
   ISO/DIS 10587 Residual Embrittlement in Metallic Coated, Externally Threaded Articles, Fasteners and Rod—Inclined Wedge Method
   ISO 4519 Electrodeposited Metallic Coatings and Related Finishes—Sampling Procedures for Inspection by Attributes

2.3 Military Standard:
   MIL-STD-1312 Fastener Test Methods 4

3. Terminology

3.1 Definitions—For the purposes of this test method the following definitions apply:

3.1.1 batch—a distinct portion of items processed collectively as a single group through the same identical treatment steps at the same time on the same rack or in the same barrel.

3.1.2 embrittled—where parts fail immediately or up to 48 h in test.

3.1.2.1 Discussion—The degree to which parts within a single plated batch or a given lot can be embrittled can vary over a wide range. The degree of embrittlement is a function of the concentration of atomic hydrogen in the individual parts in the batch or lot, measured in parts per million, and in particular that portion of the hydrogen that is mobile or free to migrate to areas of high stress concentration.

3.1.3 Grade 48 proof—where there are no failures after 48 h of test.

3.1.4 Grade 96 proof—where there are no failures after 96 h of test.

3.1.5 Grade 200 proof—where there are no failures after 200 h of test.

3.1.6 lot—a group of items processed through the same or similar steps at the same time or over a contiguous time period and from the same heat of material. The lot may be broken down into a number of batches for processing purposes and then reassembled into the same lot.

4. Summary of Test Method

4.1 The threaded articles, fasteners, or rod are subjected to stress by tensioning with a mating nut after insertion through a clearance hole in a hardened rectangular wedge of steel; see Fig. 1. Additional hardened rectangular pieces of steel with parallel faces are provided as filler plates and are inserted so that the required length of the threaded article is placed under test. Other loading systems and fixtures are permissible as long as the same load, angle, and exposure are created for the test. The upper surface of the wedge is ground at an angle to the lower surface. The mating nut is tensioned by any means.

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2 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard’s Document Summary page on the ASTM website.


4 Available from Standardization Documents Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.
TABLE 1 Wedge Angle Selection (in degrees)

<table>
<thead>
<tr>
<th>Nominal Size of Threaded Article</th>
<th>Articles with Unthreaded Lengths Less than 2 Diameters</th>
<th>Articles with Unthreaded Lengths 2 Diameters and Longer</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 to 6 mm</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td>½ to ¾ in.</td>
<td>6</td>
<td>6</td>
</tr>
<tr>
<td>6 to 18 mm</td>
<td>4</td>
<td>6</td>
</tr>
<tr>
<td>¾ to 1½ in.</td>
<td>4</td>
<td>6</td>
</tr>
<tr>
<td>over 18 to 38 mm</td>
<td>0</td>
<td>4</td>
</tr>
<tr>
<td>over ¾ to 1½ in.</td>
<td>0</td>
<td>4</td>
</tr>
</tbody>
</table>

NOTE 3—Increasing the applied torque by a small percentage as a safety factor is not recommended.

5. Significance and Use

5.1 The use of this test method can significantly reduce the risk of sudden catastrophic failure of threaded articles and fasteners, below their design strength, due to hydrogen embrittlement.

6. Apparatus

6.1 Test Fixture comprising a hardened wedge (see Fig. 1), one or more filler plates, and a hardened washer. The hole in each shall be as close to the major diameter of the threaded article, fastener, or rod being tested as practical. Excess clearance space may cause the fastener to tilt in the hole and can result in a failure at a lower torque value.

6.2 Fixture With Multiple Holes has been found useful for multiple or repetitive testing. The fixture can be readily made from a rectangular piece of an air hardening grade of steel with one face ground to the appropriate wedge angle and hardened to HRc 60.

6.3 Wedge—Shall have an angle as specified in Table 1.

6.4 Filler Plate(s)—Shall be of the same steel grade and hardness as the wedge fixture and have a thickness such that, after installation and tightening, a minimum of three full threads of the test fastener will be engaged and no more than five full threads will extend beyond the nut.

6.5 Washer—Shall be HRc 38 to 45 and shall conform to the requirements of Specification F 436.

6.6 Torque Application Device—If the torque method of tightening is used, the tightening torque shall be determined using a load measuring device capable of measuring the actual tension induced in the article, fastener, or rod as the item is tightened.

6.7 Torque Determination—Five items from the test lot shall be selected at random. Each shall be assembled into the load measuring device, mated with a nut, and the nut tightened until a load equal to 75 % of the ultimate tensile strength of the item is induced. The torque required to induce this load shall be measured and the arithmetic average of the five measured torques shall be the tightening torque. Calculated torque versus tension methods of testing such as the \( T = KD_L \) formula used in MIL-STD-1313 are not sufficiently accurate for use in this test and shall not be used.

7. Sampling

7.1 The document specifying this test method shall specify an AQI level and sampling plan to be used. Guidance in the selection of sampling plans is provided in Guide B 697. Widely used sampling plans are provided in Test Method B 602 and its equivalent ISO 4519.

7.2 A minimum sample size of 30 pieces is necessary from each embrittlement relief treated batch that exceeds 500 pieces plated as a single group.

8. Procedure

8.1 Test Item Placement—Place the test items in the clearance holes with the heads positioned against the angle of the wedge. In the case of items with square, hexagonal, or similar straight side heads, a straight side shall be placed against the angle of the wedge. In the case of elliptical or other shaped heads, the side with the minor radius of the ellipse shall be placed against the angle of the wedge. In the case of items without heads, studs, or threaded rod, one end shall be nutted and tested as the head. When the items are threaded with different pitch threads, the finer thread shall be treated as the head. Nut the free end of the items and run them up finger-tight. No significance has been found between the start of the thread on an article in relation to the angle of the wedge.

8.2 Torque Application—Clamp the wedge device with the nutted ends facing in a convenient position in a securely attached vice. Using a calibrated torque tool tighten the nuts to the desired torque and record the values. The wedge should be removed from the vice and left undisturbed for the test period. See Section 3.

9. Evaluation

9.1 Cracks, Separated Heads, and Breakage—After the specified holding period is complete, examine each item for failures such as cracks, separated heads, and breakage. Use finger pressure to check each head for breakage. Cracks can be identified by examination at 10× magnification, magnetic particle inspection, or the use of a liquid dye penetrant.

9.2 Relaxed Torque—Following the examination of the specimens in 9.1, place the wedge in a vice and carefully turn each mating nut, with the torque tool, in the on direction until a forward angular motion, after break loose, is noticeable. Record the torque value at break loose and compare it with the initially recorded torque. Torque relaxation greater than 10 % shall be recorded as failure. Remove the nuts and examine the items for transverse cracks, which shall also be recorded as failure.

10. Report

10.1 Report the following information:

10.1.1 ASTM designation number of this test method,

10.1.2 Batch identification number and total number of parts in the batch,

10.1.3 Number of parts tested,
10.1.4 Number of broken parts, parts with visible cracks or other observed failures, and parts that exhibited relaxed torque, and

10.1.5 Duration of the test method.

11. Precision and Bias

11.1 Precision—The precision of this test method has not been determined.

11.2 Bias—The bias has not been determined.

12. Keywords

12.1 hydrogen embrittlement test; metallic coated; residual embrittlement test; testing threaded articles; threaded fasteners; threaded rod

APPENDIX

(Nonmandatory Information)

X1. SOURCES OF INTRODUCTION OF HYDROGEN INTO THREADED ARTICLES

X1.1 The preparation and metallic coating of threaded articles, fasteners, and rod are usually accomplished by the barrel-plating process. In this process, quantities of an item are placed within a containment vessel, called a barrel. The barrel is designed to move the group of items, together, through each of the process steps, allowing ready ingress and egress of processing solutions and rinses. As the barrel is moved through the process steps, it is also rotated such that the individual items are constantly cascading over one another. In some of the process steps, notably the electrocleaning and electroplating steps, an electric current is applied to the group of items. The cascading action randomly exposes the surfaces of each individual piece to the process electrodes while also maintaining electrical continuity among all the parts.

X1.2 During both the electrolytic and non-electrolytic steps hydrogen is generated and exposed to the individual items in the same random manner. Experience and experimentation have shown that despite the best practice, some individual items of the group will receive more hydrogen exposure than others of the group due to the randomness of the barrel-plating process.

X1.3 Examination and analysis of barrel-plated items have shown that when hydrogen charging of such items does occur, it follows the normal distribution or bell-shaped curve. A very few of the items absorb no hydrogen, the vast majority absorb a small amount of hydrogen, and a very few items absorb more hydrogen. Baking treatment, which can vary in time and temperature, can render the normally mobile hydrogen immobile, thus rendering the individual items free of hydrogen embrittlement. However, a number of variables exist within processes that, despite the best practice, increase hydrogen charging on the parts. Platers cannot eliminate or easily control such random hydrogen charging. Therefore, testing representative quantities of the finished items, selected using a statistical sampling plan, is necessary. Thus, it is not always possible to guarantee that lots of threaded articles produced by such processes are completely free of hydrogen embrittlement. Rather they can only guarantee that representative quantities of the lot have been tested and have shown no hydrogen embrittlement failures for the specified period of test.
1. Scope

1.1 This specification covers the requirements for electrodeposited zinc cobalt alloy coatings on metals.

1.2 The following precautionary caveat pertains to the test method portion only, Section 8, of this specification: This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 117 Practice for Operating Salt Spray (Fog) Apparatus
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 320 Practice for Preparation of Iron Castings for Electroplating
B 322 Practice for Cleaning Metals Prior to Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section
B 499 Test Method for Measurement of Coating Thicknesses By the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals
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B 507 Practice for Design of Articles to Be Electroplated on Racks
B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
B 571 Test Methods for Adhesion of Metallic Coatings
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 762 Method of Variables Sampling of Metallic and Inorganic Coatings
B 849 Specification for Pretreatments of Iron or Steel for Reducing Risk of Hydrogen Embrittlement
B 850 Specification for Post-Coating Treatments of Iron or Steel for Reducing Risk of Hydrogen Embrittlement
D 3951 Practice for Commercial Packaging

3. Terminology

3.1 Definitions—Many terms used in this specification are defined in Terminology B 374.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 significant surface, n—that portion of a coated article’s surface where the coating is required to meet all the requirements of the coating specification for that article. Significant surfaces are usually those that are essential to the serviceability or function of the article or can be a source of corrosion products or tarnish films that interfere with the function or desirable appearance of the article. Significant surfaces are those surfaces that are identified by the purchaser by, for example, indicating them on an engineering drawing of the product or marking a sample item of the product.

4. Classification

4.1 There is one coating class, and it is defined as Class 1—deposits having 99 mass % zinc, the balance being cobalt.

4.2 There are five coating types and they are defined as follows:

4.2.1 Type a—With colorless (blue bright) chromate conversion coatings.
4.2.2 Type b—With yellow chromate conversion coating.
4.2.3 Type c—With bronze chromate conversion coating.
4.2.4 Type d—With black chromate conversion coating.
4.2.5 Type e—Any of the above types plus organic topcoat.

Note 1—Whereas colored chromate conversion coatings are usually meant to include various shades of yellow to bronze when used with non-alloyed zinc, yellow and bronze chromate conversion coatings are considered distinctly different when applied to alloyed zinc coatings and are formulated specifically to produce the desired coating.
4.3 There are three grades according to thickness and are defined as follows:

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<td>3</td>
</tr>
</tbody>
</table>

5. Ordering Information

5.1 In order to make the application of this specification complete, the purchaser needs to supply the following information to the seller in the purchase order and drawings.

5.1.1 Title, ASTM designation number, and date of issue of this specification.

5.1.2 Deposit by classification including class, type, and grade (see 4.1, 4.2, 4.3).

5.1.3 Composition and metallurgical condition of the substrate to be coated.

5.1.4 Location of significant surfaces (3.2.1).

5.1.5 Heat treatment for stress relief, whether it has been performed by purchaser or is required (6.7).

5.1.6 Heat treatment after electroplating, if required (6.8).

5.1.7 Whether or not location of rack marks is to be defined (6.3.1).

5.1.8 Any requirement for special test specimens (8.1.1).

5.1.9 Acceptance inspection procedure to be used (Section 7).

5.1.10 Any requirement for certification (Section 10).

5.1.11 Any other items needing agreement. For the purposes of this specification, prior agreement on the nature of the finish is required as items plated in bulk may differ in appearance from those that are rack plated.

6. Coating Requirements

6.1 Substrate—The metal to be plated shall be free of flaws and defects that will be detrimental to the zinc alloy coating. It shall be subjected to such cleaning, pickling, and electroplating procedures as are necessary to yield deposits with the desired quality.

Note 2—Proper preparatory procedures and thorough cleaning are essential to ensure satisfactory adhesion and corrosion resistance performance of the coating. Materials used for cleaning should not damage the basis metal, for example, by causing defects such as pits, intergranular attack, or stress corrosion cracking. It is recommended that the following ASTM practices for cleaning, where appropriate, be used: Practices B 183, B 242, B 320 and B 322.

6.1.1 The electroplating shall be applied after all basis metal heat treatments have been completed.

6.2 Nature of Coating:

6.2.1 The coating shall consist of a zinc cobalt alloy that is approximately 99 mass % zinc and the balance cobalt.

6.2.2 The coating shall be produced from an aqueous electroplating system, either alkaline or acid, and may be specified at the discretion of the purchaser.

6.2.3 The coating shall have such supplementary conversion coatings as defined in 4.2 and specified in the purchase order.

6.3 Appearance:

6.3.1 The coating on all readily visible surfaces shall have an acceptable and characteristic appearance as agreed upon by the purchaser and seller. The coating shall be uniform insofar as the basis metal will permit. When the article is to be plated on a rack, contact marks may be unavoidable. Location of such mark(s) shall be indicated on the article or its drawing.

6.3.2 Defects and variations in appearance that arise from surface conditions of the substrate (scratches, pores, roll marks, inclusions, and the like) and that persist in the coating despite the observance of good metal finishing practices, shall not be cause for rejection. The coating shall be adherent, free from blisters, pits, or discontinuities, and shall be free of cracks in the as plated state. Flaking shall be cause for rejection in either the as plated state or after subsequent operations.

Note 3—These coatings are commonly used in automotive applications where subsequent forming, bending, and crimping operations are commonly performed. These operations will necessarily detract from the performance of the coatings. While some cracking of coatings will be unavoidable, flaking of the coatings after these subsequent operations shall be cause for rejection.

Note 4—Coatings generally perform better in service when the substrate over which they are applied is smooth and free of torn metal, inclusions, pores, and other defects. The specifications covering the unfinished product should provide limits for these defects. A metal finisher can often remove defects through special treatments, such as grinding, polishing, abrasive blasting, chemical etches, and electropolishing. However, these are not normal in the treatment steps preceding the application of the coating. When they are desired, they are the subject of special agreement between the purchaser and the seller.

6.4 Thickness—The thickness of the coating everywhere on the significant surfaces as defined in 3.2.1 and shall conform to the requirements of the specified grade as defined in 4.3.

Note 5—The thickness of electrodeposited coatings varies from point to point on the surfaces of a product (see Practice B 507). The thickness is less in interior corners and in holes. Such surfaces are often exempt from thickness requirements. If the full thickness is required in those locations, the electroplater will have to use special techniques that will probably raise the cost of the process.

Note 6—The coating requirement of this specification is a minimum. Variation in the thickness from point to point on an article and from article to article in a production lot is inherent in electroplating. Therefore, if all of the articles in a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the specified minimum.

6.5 Adhesion—The coating shall withstand normal handling and storage conditions without chipping, flaking, or other coating damage and shall conform to the minimum requirements set forth in Section 8.

6.6 Corrosion Resistance—The corrosion resistance of the coating may be evaluated using the method in Appendix X1.

6.7 Pretreatment of Iron and Steel for Reducing the Risk of Hydrogen Embrittlement:

6.7.1 Parts that are made of steels with ultimate tensile strengths of 1000 MPa (hardness of 31 HRC or greater), that have been machined, ground, cold formed, or cold straightened subsequent to heat treatment shall require stress relief heat treatment when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specification B 849 may be consulted for a list of pretreatments that are widely used.

6.8 Post-Coating Treatments of Iron and Steel for Reducing the Risk of Hydrogen Embrittlement:

6.8.1 Parts that are made of steels with ultimate tensile strengths of 1000 MPa (hardness of 31 HRC or greater), as well as surface hardened parts, may require post-coating...
hydrogen embrittlement relief baking when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specification B 850 may be consulted for a list of post-treatments that are widely used.

7. Sampling

7.1 The sampling plan used for the inspection of a quantity of the coated articles shall be as agreed upon between the purchaser and the seller.

**Note 7**—Usually when a collection of coated articles, the inspection lot (7.2), is examined for compliance with the requirements placed on the articles, a relatively small number of the articles, the sample, is selected at random and is inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Three standards, Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for the sampling inspection of coatings. Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one when they are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 identifies the plan to be used. Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and seller need to agree on the plan to be used. Method B 762 can be used only for coating requirements that have a numerical limit, such as a coating thickness. The test must yield a numerical value and certain statistical requirements must be met. Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Method B 762 identifies the plan to be used.

**Note 8**—When both destructive and nondestructive tests exist for the measurement of a characteristic, the purchaser needs to state which is to be used so that the proper sampling plan is selected. A test may destroy the coating but in a noncritical area; or, although it may destroy the coating, a tested part can be reclaimed by stripping and recoating. The purchaser needs to state whether the test is to be considered destructive or nondestructive.

7.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, have been produced to the same specification, have been coated by a single supplier at one time or at approximately the same time under essentially identical conditions, and are submitted for acceptance or rejection as a group.

7.3 If special test specimens are used to represent the coated articles in a test, the number used shall be that required in 8.1.1.

8. Test Methods

8.1 Special Test Specimens:

8.1.1 The permission or the requirement to use special test specimens, the number to be used, the material from which they are to be made, and their shape and size shall be stated by the purchaser.

**Note 9**—Test specimens often are used to represent the coated articles in a test if the articles are of a size, shape, or material that is not suitable for the test or if it is preferred not to submit articles to a destructive test because, for example, the articles are expensive or few in number. The specimen should duplicate the characteristics of the article that influences the property being tested.

8.1.2 Special test specimens used to represent articles in an adhesion, porosity, corrosion resistance, or appearance test shall be made of the same material, shall be in the same metallurgical condition, and shall have the same surface condition as the articles they represent. They shall be placed in the production lot of and be processed along with the articles they represent.

8.1.3 Special test specimens used to represent articles in a coating thickness test may be made of a material that is suitable for the test method even if the represented article is not of the same material. For example, a low-carbon steel specimen may represent a brass article when the magnetic thickness test is used (Test Method B 499). The thickness specimen need not be carried through the complete process with the represented article. If not, it shall be introduced into the process at the point where the coating is applied and it shall be carried through all steps that have a bearing on the coating thickness. In rack plating, the specimen shall be racked in the same way with the same distance from and orientation with the anodes and other items in the process as the article it represents.

**Note 10**—When special test specimens are used to represent coated articles in a thickness test, the specimens will not necessarily have the same thickness and thickness distribution as the articles unless the specimens and the articles are of the same general size and shape. Therefore, before finished articles can be accepted on the basis of a thickness test performed on special test specimens, the relationship between the thickness on the specimen and the thickness on the part needs to be established. The criterion of acceptance is that thickness on the specimen corresponds to the required thickness on the article.

8.2 Composition of Coating—The composition of the coating shall be verified by atomic absorption spectrophotometry (AA), inductively coupled plasma (ICP), or directly coupled plasma (DCP) or other methods if found to be capable of results within 10 % of known standards.

8.3 Appearance—The coating shall be examined at up to 10X magnification for conformance to the requirements of appearance.

8.4 Thickness—The coating thickness shall be measured at locations on the significant surface designated by the purchaser, and the measurement shall be made with an accuracy of 10 % or better by use of one of the following standards, Test Methods B 487, B 499, B 504, and B 568.

8.5 Adhesion—The adhesion of the coating shall be tested in accordance with Test Methods B 571. The choice of test method shall be one of those appropriate for zinc coatings.

8.6 Corrosion Resistance—See Appendix X1.

9. Rejection and Rehearing

9.1 Articles that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of a test, the producer or supplier may make a claim for a rehearing. Coatings that show imperfections during subsequent manufacturing operations may be rejected.

10. Certification

10.1 When specified in the purchase order or contract, the purchaser shall be furnished certification that samples representing each lot have been either tested or inspected as directed in this specification, and the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.
11. Packaging

11.1 The packaging and packing requirements shall be in accordance with Practice D 3951 or as specified in the contract or order.

12. Keywords

12.1 coatings electrodeposited; corrosion resistant coatings; electrodeposited zinc cobalt alloy plating; zinc cobalt

APPENDIX

(Nonmandatory Information)

X1. Durations of Continuous Testing

X1.1 The corrosion resistance of the coating may be evaluated using Practice B 117. Table X1.1 suggests durations of continuous testing that properly applied coatings should withstand but are to be used as a guide only.

<table>
<thead>
<tr>
<th>Class</th>
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<th>Zinc Alloy Corrosion Product</th>
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<td>A</td>
<td>240</td>
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<tr>
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<td>340</td>
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<tr>
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<td>B</td>
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</tr>
<tr>
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<td>196</td>
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<tr>
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<td>600</td>
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</tr>
<tr>
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<td>6</td>
<td>D</td>
<td>240</td>
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</tr>
<tr>
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<td>D/E</td>
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<td>200</td>
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</tr>
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<td>400</td>
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Standard Specification for Electrodeposited Coatings of Zinc Nickel Alloy Deposits

This standard is issued under the fixed designation B 841; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers the requirements for electrodeposited zinc nickel alloy coatings on metals.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 The following precautionary statement pertains to the test method portion only, Section 8, of this specification: This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

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3.2.2 Discussion—Significant surfaces are those surfaces that are identified by the purchaser by, for example, indicating them on an engineering drawing of the product or marking a sample item of the product.

4. Classification

4.1 There is one class of zinc nickel coating defined as follows:

4.1.1 Class I—Deposits having a minimum of 5 and maximum 12 mass % nickel, the balance being zinc.

4.2 There are five chromate conversion coating types that are defined as follows:

4.2.1 Type A—with colorless (blue bright) conversion coatings,
4.2.2 Type B—with yellow iridescent conversion coatings,
4.2.3 Type C—with bronze conversion coatings,
4.2.4 Type D—with black chromate conversion coatings, and
4.2.5 Type E—Any of the above plus organic topcoat.

NOTE 1—Whereas colored chromate conversion coatings are usually

meant to include various shades of yellow to bronze when used with nonalloyed zinc, yellow and bronze chromate conversion coatings are considered distinctly different in appearance and performance when applied to alloyed zinc coatings and are formulated specifically to produce the desired coating and are formulated specifically to produce the desired coating.

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5.1.2 Deposit by classification including Class, Type, and Grade (see 4.1, 4.2, 4.3),

5.1.3 Composition and metallurgical condition of the substrate to be coated,

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5.1.5 Heat treatment for stress relief, whether it has been performed by purchaser or is required (see 6.7),

5.1.6 Heat treatment after electroplating, if required (see 6.8),

5.1.7 Whether or not location of rack marks is to be defined (see 6.3.1),

5.1.8 Any requirement for special test specimens (see 8.1.1),

5.1.9 Acceptance inspection procedure to be used (see Section 8), and

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5.1.11 Any other items needing agreement. For the purposes of this specification, prior agreement on the nature of the finish is required as items plated in bulk may differ in appearance from those that are rack plated.

6. Coating Requirements

6.1 Substrate—The metal to be plated shall be free of flaws and defects that will be detrimental to the zinc alloy coating. It shall be subjected to such cleaning, pickling, and electroplating procedures as are necessary to yield deposits with the desired quality.

NOTE 2—Proper preparatory procedures and thorough cleaning are essential to ensure satisfactory adhesion and corrosion resistance performance of the coating. Materials used for cleaning should not damage the basis metal, for example by causing defects such as pits, intergranular attack or stress corrosion cracking. It is recommended that the following practices for cleaning, where appropriate, be used: Practices B 183, B 242, B 320 and B 322.

6.1.1 The electroplating shall be applied after all basis metal heat treatments have been completed.

6.2 Nature of Coating:

6.2.1 The coating shall consist of a zinc nickel alloy that has a minimum of 5 and maximum 12 mass percent nickel, the balance being zinc.

6.2.2 The coating shall be produced from an aqueous electroplating system that may be either an alkaline or acid formulation as specified by the purchaser.

6.2.3 The coating shall have such supplementary conversion coatings as defined in 4.2 and specified in the purchase order.

6.3 Appearance:

6.3.1 The coating on all ready visible surfaces shall have an acceptable and characteristic appearance as agreed upon by the purchaser and seller. The coating shall be uniform insofar as the basis metal will permit. When the article is to be plated on a rack, contact marks may be unavoidable. Location of such marks(s) shall be indicated on the article or its drawing.

6.3.2 Defects and variations in appearance that arise from surface conditions of the substrate (scratches, pores, roll marks, inclusions, and the like) and that persist in the coating despite the observance of good metal finishing practices shall not be cause for rejection. The coating shall be adherent, free from blisters, pits, or discontinuities, and shall be free of cracks in the as plated state. Flaking shall be cause for rejection in either the as plated state or after subsequent operations.

NOTE 3—These coatings are commonly used in automotive applications where subsequent forming, bending, and crimping operations are commonly performed. These operations will necessarily detract from the performance of the coatings. While some cracking of coatings will be unavoidable, flaking of the coating after these subsequent operations shall be cause for rejection.

NOTE 4—Coatings generally perform better in service when the substrate over which they are applied is smooth and free of torn metal, inclusions, pores, and other defects. The specifications covering the unfinished product should provide limits for these defects. A metal finisher can often remove defects through special treatments, such as grinding, polishing, abrasive blasting, chemical etches, and electropolishing. However, these are not normal in the treatment steps preceding the application of the coating. When they are desired, they are the subject of special agreement between the purchaser and the seller.

6.4 Thickness:

6.4.1 The thickness of the coating everywhere on the significant surfaces as defined in 3.2.1 and shall conform to the requirements of the specified grade as defined in 4.3.

NOTE 5—The thickness of electrodeposited coatings varies from point to point on the surfaces of a product (see Practice B 507). The thickness is less in interior corners and in holes. Such surfaces are often exempt from thickness requirements. If the full thickness is required in those locations, the electroplater will have to use special techniques that will probably raise the cost of the process.

NOTE 6—The coating requirement of this specification is a minimum. Variation in the thickness from point to point on an article and from article to article in a production lot is inherent in electroplating. Therefore, if all of the articles in a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the specified minimum.

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6.7 Pretreatment of Iron and Steel for Reducing the Risk of Hydrogen Embrittlement:

6.7.1 Parts that are made of steels with ultimate tensile strengths of 1000 MPa (hardness of 31 HRC or greater) that...
have been machined, ground, cold formed, or cold straightened subsequent to heat treatment shall require stress relief heat treatment when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specification B 849 may be consulted for a list of pretreatments that are widely used.

6.8 Post-Coating Treatments of Iron and Steel for Reducing the Risk of Hydrogen Embrittlement:

6.8.1 Parts that are made of steels with ultimate tensile strengths of 1000 MPa (hardness of 31 HRc or greater), as well as surface hardened parts, may require post-coating hydrogen embrittlement relief baking when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specification B 850 may be consulted for a list of post-treatments that are widely used.

7. Sampling

7.1 The sampling plan used for the inspection of a quantity of the coated articles shall be as agreed upon between the purchaser and the seller.

Note 7—Usually, when a collection of coated articles, the inspection lot (see 7.2), is examined for compliance with the requirements placed on the articles, a relatively small number of the articles, the sample, is selected at random and is inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Three standards, Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for the sampling inspection of coatings. Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one when they are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 identifies the plan to be used. Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and seller need to agree on the plan to be used. Method B 762 can be used only for coating requirements that have a numerical limit, such as a coating thickness. The test must yield a numerical value and certain statistical requirements must be met. Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Method B 762 identifies the plan to be used.

Note 8—When both destructive and nondestructive tests exist for the measurement of a characteristic, the purchaser needs to state which is to be used so that the proper sampling plan is selected. A test may destroy the coating but in a noncritical area; or, although it may destroy the coating, a tested part can be reclaimed by stripping and recoating. The purchaser needs to state whether the test is to be considered destructive or nondestructive.

7.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specification that have been coated by a single supplier at one time or at approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

7.3 If special test specimens are used to represent the coated articles in a test, the number used shall be that required in 8.1.1.

8. Test Methods

8.1 Special Test Specimens:

8.1.1 The permission or the requirement to use special test specimens, the number to be used, the material from which they are to be made, and their shape and size shall be stated by the purchaser.

Note 9—Test specimens often are used to represent the coated articles in a test if the articles are of a size, shape, or material that is not suitable for the test or if it is preferred not to submit articles to a destructive test because, for example, the articles are expensive or few in number. The specimen should duplicate the characteristics of the article that influence the property being tested.

8.1.2 Special test specimens used to represent articles in an adhesion, porosity, corrosion resistance, or appearance test shall be made of the same material, in the same metallurgical condition, and have the same surface condition as the articles they represent, and be placed in the production lot of and be processed along with the articles they represent.

8.1.3 Special test specimens used to represent articles in a coating thickness test may be made of a material that is suitable for the test method even if the represented article is not of the same material. For example, a low-carbon steel specimen may represent a brass article when the magnetic thickness test is used (see Test Method B 499). The thickness specimen need not be carried through the complete process with the represented article. If not, introduce it into the process at the point where the coating is applied and carry it through all steps that have a bearing on the coating thickness. In rack plating, rack the specimen in the same way with the same distance from and orientation with the anodes and other items in the process as the article it represents.

Note 10—When special test specimens are used to represent coated articles in a thickness test, the specimens will not necessarily have the same thickness and thickness distribution as the articles unless the specimens and the articles are of the same general size and shape. Therefore, before finished articles can be accepted on the basis of a thickness test performed on special test specimens, the relationship between the thickness on the specimen and the thickness on the part needs to be established. The criterion of acceptance is that thickness on the specimen that corresponds to the required thickness on the article.

8.2 Composition of Coating—Verify the composition of the coating by atomic absorption spectrophotometry (AA), inductively coupled plasma (ICP), or directly coupled plasma (DCP) or other methods if found to be capable of results within 10 % of known standards.

8.3 Appearance—Examine the coating at up to 10× magnification for conformance to the requirements of appearance.

8.4 Thickness—Measure the coating thickness at locations on the significant surface designated by the purchaser, and make the measurement with an accuracy of 10 % or better by use of one of the following test methods: Test Methods B 487, B 499, B 504, B 568.

8.5 Adhesion—Test the adhesion of the coating in accordance with Test Method B 571. The choice of test method shall be one of those appropriate for zinc coatings.

8.6 Corrosion Resistance—See Appendix X1.

9. Rejection and Rehearing

9.1 Articles that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of a test, the producer or supplier may make a claim for a rehearing. Coatings that show
imperfections during subsequent manufacturing operations may be rejected.

10. Certification

10.1 When specified in the purchase order or contract, the purchaser shall be furnished certification that samples representing each lot have been either tested or inspected as directed in this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

11. Packaging

11.1 The packaging and packing requirements shall be in accordance with Practice D 3951 or as specified in the contract or order.

12. Keywords

12.1 coatings electrodeposited; corrosion resistant coatings; electrodeposited zinc nickel alloy plating; electroplating; zinc alloy; zinc nickel

APPENDIX

(Nonmandatory Information)

X1. Corrosion Resistance of Coating

X1.1 The corrosion resistance of the coating may be evaluated using Practice B 117. Table X1.1 suggests durations of continuous testing that properly applied coatings should withstand but are to be used as a guide only.

<table>
<thead>
<tr>
<th>Class</th>
<th>Grade</th>
<th>Type</th>
<th>Base Corrosion</th>
<th>Alloy Corrosion</th>
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</thead>
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<td>A</td>
<td>150</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>A/E</td>
<td>300</td>
<td>150</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>A</td>
<td>240</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>A/E</td>
<td>400</td>
<td>150</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>A</td>
<td>500</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>A/E</td>
<td>620</td>
<td>150</td>
</tr>
<tr>
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<td>B</td>
<td>500</td>
<td>120</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>B/E</td>
<td>620</td>
<td>240</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>B</td>
<td>720</td>
<td>120</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>B/E</td>
<td>840</td>
<td>240</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>B</td>
<td>960</td>
<td>120</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>B/E</td>
<td>1080</td>
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</tr>
<tr>
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<td>5</td>
<td>C</td>
<td>750</td>
<td>180</td>
</tr>
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<td></td>
<td>5</td>
<td>C/E</td>
<td>1000</td>
<td>300</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>C</td>
<td>960</td>
<td>180</td>
</tr>
<tr>
<td></td>
<td>8</td>
<td>C/E</td>
<td>1200</td>
<td>300</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>C</td>
<td>1000</td>
<td>180</td>
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<td>10</td>
<td>C/E</td>
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<td>5</td>
<td>D</td>
<td>500</td>
<td>100</td>
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<td></td>
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<td>D/E</td>
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<td></td>
<td>10</td>
<td>D/E</td>
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</table>
Standard Specification for Electrodeposited Coatings of Zinc Iron Alloy Deposits

This standard is issued under the fixed designation B 842; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers the requirements for electrodeposited zinc iron alloy coatings on metals.

1.2 The values stated in either inch-pound or SI units are to be regarded as standard. The units given in parentheses are for information only.

1.3 The following precautionary caveat pertains to the test method portion only, Section 8, of this specification: This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 117 Practice for Operating Salt Spray (Fog) Apparatus
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 320 Practice for Preparation of Iron Castings for Electroplating
B 322 Practice for Cleaning Metals Prior to Electroplating
B 374 Terminology Relating to Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section
B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
B 507 Practice for Design of Articles to Be Electroplated on racks
B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
B 571 Test Method for Adhesion of Metallic Coatings
B 602 Test Method of Attribute Sampling of Metallic and Inorganic Coatings
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 762 Test Method of Variables Sampling of Metallic and Inorganic Coatings
B 849 Specification for Pretreatments of Iron or Steel for Reducing Hydrogen Embrittlement
B 850 Specification for Post-Coating Treatments of Iron or Steel for Reducing Risk of Hydrogen Embrittlement
D 3951 Practice for Commercial Packaging

3. Terminology

3.1 Definitions—Many terms used in this specification are defined in Terminology B 374.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 significant surface, n—that portion of a coated article’s surface where the coating is required to meet all the requirements of the coating specification for that article.

3.2.1.1 Discussion—Significant surfaces are usually those that are essential to the serviceability or function of the article, or that can be a source of corrosion products or tarnish films that interfere with the function or desirable appearance of the article. Significant surfaces are those surfaces that are identified by the purchaser, for example, indicating them on an engineering drawing of the product or marking a sample item of the product.

4. Classification

4.1 There is one class of zinc iron alloy that is defined as follows:

4.1.1 Class I—Deposits having approximately 99 mass % zinc, balance iron.

4.2 There are two coating types that are defined as follows:

4.2.1 Type A—Zinc iron with black chromate conversion coating, and

4.2.2 Type B—Zinc iron with iridescent yellow chromate conversion coating.

---


2 Annual Book of ASTM Standards, Vol 02.05.

4.3 There are three coating grades according to thickness that are defined as follows:

<table>
<thead>
<tr>
<th>Minimum Thickness, µm</th>
<th>New ASTM Grade</th>
<th>Old ASTM Grade</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>6</td>
<td>1</td>
</tr>
<tr>
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<td>12</td>
<td>2</td>
</tr>
<tr>
<td>18</td>
<td>18</td>
<td>3</td>
</tr>
</tbody>
</table>

5. Ordering Information

5.1 In order to make the application of this specification complete, the purchaser needs to supply the following information to the seller in the purchase order and drawings:

5.1.1 Title, ASTM designation number, and date of issue of this specification,

5.1.2 Deposit by classification including class (see 4.1), type (see 4.2), and grade (see 4.3),

5.1.3 Composition and metallurgical condition of the substrate to be coated,

5.1.4 Location of significant surfaces (see 3.2),

5.1.5 Heat treatment for stress relief, whether it has been performed by purchaser or is required (see 6.7),

5.1.6 Heat treatment after electroplating, if required (see 6.8),

5.1.7 Any requirement for submission of sample coated articles,

5.1.8 Whether or not location of rack marks is to be defined (see 6.3.1),

5.1.9 Any requirement for special test specimens (see 8.1),

5.1.10 Acceptance inspection procedure to be used (see Section 8),

5.1.11 Any requirement for certification (see Section 10), and

5.1.12 Any other items needing agreement. For the purposes of this specification, prior agreement on the nature of the finish is required as items plated in bulk may differ slightly in appearance from those that are rack plated.

6. Coating Requirements

6.1 Substrate—The metal to be plated shall be free of flaws and defects that will be detrimental to the zinc alloy coating. It shall be subjected to such cleaning, pickling, and electroplating procedures as are necessary to yield deposits with the desired quality.

NOTE 1—Proper preparatory procedures and thorough cleaning are essential to ensure satisfactory adhesion and corrosion resistance performance of the coating. Materials used for cleaning should not damage the basis metal. It is recommended that the following practices for cleaning be used, where appropriate: Practices B 183, B 242, B 320 and B 322.

6.1.1 The electroplating shall be applied after all basis metal heat treatments have been completed.

6.2 Nature of Coating—The coating shall consist of a zinc iron alloy that is approximately 99 mass % zinc and the balance iron.

6.3 Appearance:

6.3.1 The coating on all readily visible surfaces shall have an acceptable and characteristic appearance as agreed upon by the purchaser and seller. The coating shall be uniform insofar as the basis metal will permit. When the article is to be plated on a rack, contact marks may be unavoidable. Location of such marks(s) shall be indicated on the article or its drawing.

6.3.2 Defects and variations in appearance that arise from surface conditions of the substrate (scratches, pores, roll marks, inclusions, and the like) and that persist in the coating despite the observance of good metal finishing practices shall not be cause for rejection. The coating shall be adherent, free from blisters, pits, or discontinuities, and shall be free of cracks in the as-plated state. Flaking shall be cause for rejection in either the as-plated state or after subsequent operations.

NOTE 2—These coatings are commonly used in automotive applications where subsequent forming, bending, and crimping operations are commonly performed. These operations will necessarily detract from the performance of the coatings. While some cracking of coatings will be unavoidable, flaking of the coating after these subsequent operations shall be cause for rejection.

NOTE 3—Coatings generally perform better in service when the substrate over which they are applied is smooth and free of torn metal, inclusions, pores, and other defects. The specifications covering the unfinished product should provide limits for these defects. A metal finisher can often remove defects through special treatments, such as grinding, polishing, abrasive blasting, chemical etches, and electropolishing. However, these are not normal in the treatment steps preceding the application of the coating. When they are desired, they are the subject of special agreement between the purchaser and the seller.

6.4 Thickness—The thickness of the coating everywhere on the significant surfaces shall conform to the requirements as specified in 4.2 and defined in 3.2.1.

NOTE 4—The coating requirement of this specification is a minimum. Variation in the thickness from point to point on an article and from article to article in a production lot is inherent in electroplating. Therefore, if all of the articles in a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the specified minimum.

6.5 Adhesion—The coating shall withstand normal handling and storage conditions without chipping, flaking, or other coating damage and shall conform to the minimum requirements set forth in Section 8.

6.6 Corrosion Resistance—The corrosion resistance of the coating may be evaluated using the method in Appendix X1.

6.7 Pretreatment of Iron and Steel to Reduce the Risk of Hydrogen Embrittlement:

6.7.1 Parts that are made of steels with ultimate tensile strengths of 1000 MPa (hardness of 31 HRC or greater) that have been machined, ground, cold formed, or cold straightened subsequent to heat treatment shall require stress relief heat treatment when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specification B 849 may be consulted for a list of pretreatments that are widely used.

6.8 Coating Treatments of Iron and Steel to Reduce the Risk of Hydrogen Embrittlement:

6.8.1 Parts that are made of steels with ultimate tensile strengths of 1000 MPa (hardness of 31 HRC or greater) as well as surface hardened parts, may require post-coating hydrogen embrittlement relief baking when specified by the purchaser, the tensile strength to be supplied by the purchaser. Specification B 850 may be consulted for a list of post-treatments that are widely used.

7. Sampling

7.1 The sampling plan used for the inspection of a quantity of the coated articles shall be as agreed upon between the
purchaser and the seller.

Note 5—Usually, when a collection of coated articles, the inspection lot (see 7.2), is examined for compliance with the requirements placed on the articles, a relatively small number of the articles, the sample, is selected at random and is inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria for compliance are determined by the application of statistics. This procedure is known as sampling inspection. Three standards, Test Method B 602, Guide B 697, and Test Method B 762, contain sampling plans that are designed for the sampling inspection of coatings. Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one for destructive tests. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 identifies the plan to be used. Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and seller need to agree on the plan to be used. Test Method B 762 can be used only for coating requirements that have a numerical limit, such as a coating thickness. The test must yield a numerical value and certain statistical requirements must be met. Test Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Test Method B 762 identifies the plan to be used.

Note 6—When both destructive and nondestructive tests exist for the measurement of a characteristic, the purchaser needs to state which is to be used so that the proper sampling plan is selected. A test may destroy the coating but in a noncritical area, or, although it may destroy the coating, a tested part can be reclaimed by stripping and recoating. The purchaser needs to state whether the test is to be considered destructive or nondestructive.

7.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specification, that have been coated by a single supplier at one time or at approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

7.3 If special test specimens are used to represent the coated articles in a test, the number used shall be that required in 8.1.1.

8. Test Methods

8.1 Special Test Specimens:

8.1.1 The permission or the requirement to use special test specimens, the number to be used, the material from which they are to be made, and the shape and size of the specimens, shall be stated by the purchaser.

Note 7—Test specimens often are used to represent the coated articles in a test if the articles are of a size, shape, or material that is not suitable for the test, or if it is preferred not to submit articles to a destructive test because, for example, the articles are expensive or few in number. The specimen should duplicate the characteristics of the article that influence the property being tested.

8.1.2 Special test specimens used to represent articles in an adhesion, porosity, corrosion resistance, or appearance test shall be made of the same material, in the same metallurgical condition, and have the same surface condition as the articles they represent, and be placed in the production lot of and be processed along with the articles they represent.

8.1.3 Special test specimens used to represent articles in a coating thickness test may be made of a material that is suitable for the test method even if the represented article is not of the same material. For example, a low-carbon steel specimen may represent a brass article when the magnetic thickness test is used (see Test Method B 499). The thickness specimen need not be carried through the complete process with the represented article. If not, introduce it into the process at the point where the coating is applied and carry it through all steps that have a bearing on the coating thickness. In rack plating, rack the specimen in the same way with the same distance from and orientation with the anodes and other items in the process as the article it represents.

Note 8—When special test specimens are used to represent coated articles in a thickness test, the specimens will not necessarily have the same thickness and thickness distribution as the articles unless the specimens and the articles are of the same general size and shape. Therefore, before finished articles can be accepted on the basis of a thickness test performed on special test specimens, the relationship between the thickness on the specimen and the thickness on the part needs to be established. The criterion of acceptance is that thickness on the specimen that corresponds to the required thickness on the article.

8.2 Composition of Coating—Verify the composition of the coating by atomic absorption spectrophotometry (AA), inductively coupled plasma (ICP), or directly coupled plasma (DCP) or other methods if found to be capable of results within 10% of known standards.

8.3 Appearance—Examine the coating at up to 10 × magnification for conformance to the requirements of appearance.

8.4 Thickness—Measure the coating thickness at locations on the significant surface designated by the purchaser, and make the measurement with an accuracy of 10% or better by use of one of the following test methods: Test Methods B 487, B 499, B 504, or B 568.

8.5 Adhesion—Test the adhesion of the coating in accordance with Test Method B 571. The choice of test method shall be one of those appropriate for zinc coatings.

8.6 Corrosion Resistance—See Appendix X1.

9. Rejection and Rehearing

9.1 Articles that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of a test, the producer or supplier may make a claim for a rehearing. Coatings that show imperfections during subsequent manufacturing operations may be rejected.

10. Certification

10.1 When specified in the purchase order or contract, the purchaser shall be furnished certification that samples representing each lot have been either tested or inspected as directed in this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

11. Packaging

11.1 The packaging and packing requirements shall be in accordance with Practice D 3951 or as specified in the contract or order.
12. Keywords
12.1 alloy zinc; coatings—corrosion resistant; electro-deposited zinc iron; zinc alloy; zinc iron

APPENDIX
(Nonmandatory Information)

X1. CORROSION RESISTANCE

X1.1 The corrosion resistance of the coating may be evaluated using Practice B 117. Table X1.1 suggests durations of continuous testing that properly applied coatings should withstand, but are to be used as a guide only.

<table>
<thead>
<tr>
<th>Class</th>
<th>Type</th>
<th>Grade</th>
<th>Typical Appearance</th>
<th>Base Metal Corrosion</th>
<th>Zinc Corrosion Product</th>
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<td>3</td>
<td>yellow</td>
<td>700</td>
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1. Scope

1.1 This specification covers powder forged ferrous materials fabricated by hot densification of atomized prealloyed or iron powders and intended for use as structural parts.

1.2 This specification covers powder forged parts made from the following materials:

1.2.1 Compositions:

1.2.1.1 P/F-10XX Carbon Steel (produced from atomized iron powder and graphite powder),

1.2.1.2 P/F-10CXX Copper-Carbon Steel (produced from atomized iron powder, copper and graphite powders),

1.2.1.3 P/F-11XX Carbon Steel with manganese sulfide for enhanced machinability (produced from atomized iron powder, manganese sulfide, and graphite powders),

1.2.1.4 P/F-11CXX Copper-Carbon Steel with manganese sulfide for enhanced machinability (produced from atomized iron powder, copper, manganese sulfide, and graphite powders),

1.2.1.5 P/F-42XX Nickel-Molybdenum Steel (produced from prealloyed atomized iron-nickel-molybdenum powder and graphite powder),

1.2.1.6 P/F-46XX Nickel-Molybdenum Steel (produced from prealloyed atomized iron-nickel-molybdenum powder and graphite powder),

1.2.1.7 P/F-44XX Molybdenum Steel (produced from prealloyed atomized iron-molybdenum powder and graphite powder),

1.2.1.8 P/F-49XX Molybdenum Steel (produced from prealloyed atomized iron-molybdenum powder and graphite powder).

Note 1—Alloy composition designations are modifications of the AISI-SAE nomenclature. For example: 10CXX designates a plain carbon steel containing copper and XX amount of carbon. Compositional limits of alloy and impurity elements may be different from the AISI-SAE limits. Chemical composition limits are specified in Section 6.

Note 2—XX designates the forged carbon content, in hundredths of a percent, that is specified by the purchaser for the application. For a given specified carbon content, the permissible limits shall be as specified in 6.2.

1.2.2 Grades:

1.2.2.1 Grade A—Density equivalent to a maximum of 0.5% porosity. The minimum density of those sections of the powder forged part so designated by the applicable part drawing shall not be less than the value specified in Table 1.

1.2.2.2 Grade B—Density equivalent to a maximum of 1.5% porosity. The minimum density of those sections of the powder forged part so designated by the applicable part drawing shall not be less than the value specified in Table 1.

1.3 Property values stated in inch-pound units are the standard. Conversion factors to SI units may be approximate.

2. Referenced Documents

2.1 ASTM Standards:

A 255 Test Methods for Determining Hardenability of Steel

B 243 Terminology of Powder Metallurgy

B 311 Test Method for Density Determination for Powder Metallurgy (P/M) Materials Containing Less Than Two Percent Porosity

B 795 Test Method for Determining the Percentage of Alloyed or Unalloyed Iron Contamination Present in Low-Alloy Powder Forged (P/F) Steel Parts

B 796 Test Method for Nonmetallic Inclusion Level of Powder Forged (P/F) Steel Parts

B 797 Test Method for Surface Finger Oxide Penetration Depth and Presence of Interparticle Oxide Networks in Powder Forged (P/F) Steel Parts

E 3 Practice for Preparation of Metallographic Specimens

E 8 Test Methods for Tension Testing of Metallic Materials

E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials

E 23 Test Methods for Notched Bar Impact Testing of Metallic Materials


E 415 Test Method for Optical Emission Vacuum Spectrographic Analysis of Carbon and Low-Alloy Steel

E 562 Test Method for Determining Volume Fraction by

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Footnotes:


2 Annual Book of ASTM Standards, Vol 01.05.

3 Annual Book of ASTM Standards, Vol 02.05.

4 Annual Book of ASTM Standards, Vol 03.01.

5 Annual Book of ASTM Standards, Vol 03.05.

6 Annual Book of ASTM Standards, Vol 03.06.
Systematic Manual Point Count5
E 1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel and in Iron, Nickel, and Cobalt Alloys5
E 1077 Test Methods for Estimating the Depth of Decarburization of Steel Specimens4
2.2 Other Standards:
J 423 SAE Recommended Practice, Methods of Measuring Case Depth7
MPIF 35 Materials Standards for P/F Steel Parts8

3. Terminology

3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243. Additional descriptive information is available in the Related Material Section of Vol. 02.05 of the Annual Book of ASTM Standards.

3.2 Descriptions of Terms Specific to This Standard:

3.2.1 core region—a core region is one where there is either no decarburization as determined by the procedure in 9.3.4 or there is no hardened surface as determined by the procedure in S2.2.

3.2.2 critical region—a critical region of a part is one that requires a density level or a microstructural characteristic to be separately specified.

### TABLE 1 Minimum Density for Selected Powder Forged Steel Compositions (Fully Annealed Heat Treatment Condition—Ferrite/Pearlite Microstructure)\(^{A}\)

<table>
<thead>
<tr>
<th>Chemical Composition</th>
<th>Density (g/cm(^3))</th>
</tr>
</thead>
<tbody>
<tr>
<td>P/F-1040</td>
<td>7.81</td>
</tr>
<tr>
<td>P/F-1060</td>
<td>7.81</td>
</tr>
<tr>
<td>P/F-10C40</td>
<td>7.81 (^{C})</td>
</tr>
<tr>
<td>P/F-10C60</td>
<td>7.81 (^{C})</td>
</tr>
<tr>
<td>P/F-1140</td>
<td>7.78</td>
</tr>
<tr>
<td>P/F-1160</td>
<td>7.78</td>
</tr>
<tr>
<td>P/F-11C40</td>
<td>7.79 (^{C})</td>
</tr>
<tr>
<td>P/F-11C60</td>
<td>7.79 (^{C})</td>
</tr>
<tr>
<td>P/F-4220</td>
<td>7.82</td>
</tr>
<tr>
<td>P/F-4240</td>
<td>7.82</td>
</tr>
<tr>
<td>P/F-4260</td>
<td>7.80</td>
</tr>
<tr>
<td>P/F-4420</td>
<td>7.82</td>
</tr>
<tr>
<td>P/F-4440</td>
<td>7.81</td>
</tr>
<tr>
<td>P/F-4460</td>
<td>7.81</td>
</tr>
<tr>
<td>P/F-4620</td>
<td>7.82</td>
</tr>
<tr>
<td>P/F-4640</td>
<td>7.81</td>
</tr>
<tr>
<td>P/F-4660</td>
<td>7.81</td>
</tr>
<tr>
<td>P/F-4680</td>
<td>7.80</td>
</tr>
<tr>
<td>P/F-4920</td>
<td>7.83</td>
</tr>
<tr>
<td>P/F-4940</td>
<td>7.82</td>
</tr>
<tr>
<td>P/F-4960</td>
<td>7.82</td>
</tr>
</tbody>
</table>

\(^{A}\) Quench-hardening and tempering will reduce the density values. Normalized samples may have lower density values than fully annealed materials.

### TABLE 2 Chemical Composition Requirements for Powder Forged Parts Chemical Composition (Weight %)

<table>
<thead>
<tr>
<th>Element</th>
<th>P/F-10XX</th>
<th>P/F-10CXX</th>
<th>P/F-11XX</th>
<th>P/F-11CXX</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel, max</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
</tr>
<tr>
<td>Molybdenum, max</td>
<td>0.05</td>
<td>0.05</td>
<td>0.05</td>
<td>0.05</td>
</tr>
<tr>
<td>Manganese</td>
<td>0.10–0.25</td>
<td>0.10–0.25</td>
<td>0.30–0.60 (^{A})</td>
<td>0.30–0.60 (^{A})</td>
</tr>
<tr>
<td>Copper, max</td>
<td>0.30 max</td>
<td>1.8–2.2</td>
<td>0.30 max</td>
<td>1.8–2.2</td>
</tr>
<tr>
<td>Chromium, max</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
<td>0.10</td>
</tr>
<tr>
<td>Sulfur, max</td>
<td>0.025</td>
<td>0.025</td>
<td>0.23(^{A})</td>
<td>0.23(^{A})</td>
</tr>
<tr>
<td>Silicon, max</td>
<td>0.03</td>
<td>0.03</td>
<td>0.03</td>
<td>0.03</td>
</tr>
<tr>
<td>Phosphorus, max</td>
<td>0.03</td>
<td>0.03</td>
<td>0.03</td>
<td>0.03</td>
</tr>
<tr>
<td>Carbon, (^{b})</td>
<td>(____)</td>
<td>(____)</td>
<td>(____)</td>
<td>(____)</td>
</tr>
<tr>
<td>Oxygen, (^{c})</td>
<td>(____)</td>
<td>(____)</td>
<td>(____)</td>
<td>(____)</td>
</tr>
<tr>
<td>Total Iron Balance(^{d})</td>
<td>Balance(^{d})</td>
<td>Balance(^{d})</td>
<td>Balance(^{d})</td>
<td>Balance(^{d})</td>
</tr>
</tbody>
</table>

\(^{A}\) Covers manganese sulfide (MnS) additions of from 0.3 to 0.5%. The manganese content in solution is similar to P/F-10XX or P/F-10CXX, that is, 0.10 to 0.25%.

\(^{b}\) Carbon content shall be as specified by the purchaser.

\(^{c}\) When required, maximum oxygen content shall conform to the amount specified by the purchaser. See S1.

\(^{d}\) For information only. Quantitative determination of this element is not required.
5. Materials and Manufacture

5.1 Make the structural parts by hot forging of powder metallurgy (P/M) preforms in confined dies with or without subsequent heat treatment. Prepare P/M preforms by pressing or by pressing and sintering material conforming to the designations in 1.2.1 and meeting the chemical compositions specified in Section 6 and Table 2.

6. Chemical Composition

6.1 The hot forged material shall conform to the requirements prescribed in Table 2.

6.2 Unless otherwise specified, the hot forged carbon content shall not deviate from that specified by the purchaser by more than ±0.05 weight percent.

6.3 Determine chemical analysis for the elements copper, chromium, manganese, molybdenum, nickel, phosphorus, and silicon in accordance with Method E 415 (preferred method) or Test Method E 350. Determine analysis for the elements carbon and sulfur in accordance with Test Method E 1019.

7. Density Requirement

7.1 The minimum density of those sections of powder forged parts so designated by the applicable part drawing shall not be less than the values specified in Table 1.

7.2 Determine the density of complete parts or sections of parts in accordance with Test Method B 311.

8. Mechanical Property Requirements

8.1 Mechanical Properties:

8.1.1 The preferred method for verifying the acceptable performance of a finished part is for the manufacturer and the purchaser to agree upon a qualification test to be performed on an actual part. The specific test should be determined following consideration of the function of the part. An example would be measuring the force needed to break teeth off a gear, using a prescribed test fixture.

8.1.2 Where the part configuration permits, standard mechanical property test specimens may be machined from the part in the condition in which it is to be used. (Remove test specimens from parts to be used in the quenched and tempered condition after heat treatment of the part to ensure the microstructure is representative of the actual part.) The applicable part drawing or purchase order shall designate the location from which the mechanical property test specimens are to be removed and the type of specimen to be tested.

8.1.3 The core hardness range of parts shall be in accordance with the applicable part drawing or purchase order.

8.1.4 The surface hardness range of parts shall be in accordance with the applicable part drawing or purchase order.

8.1.5 Typical mechanical properties of Grade A materials covered by this specification are shown in Appendix X1.

8.2 Mechanical Property Test Methods:

8.2.1 Tensile Test Method—When requested, take tensile test specimens from parts in accordance with the applicable part drawing or purchase order. Test tensile specimens in accordance with Test Method E 8. Determine yield strength by the 0.2 % offset method.

8.2.2 Impact Energy Test Method—When requested, take Charpy V-notch impact test bars from parts in accordance with the applicable part drawing or purchase order. Test impact bars in accordance with Test Methods E 23; at room temperature, or, at a temperature agreed between the manufacturer and purchaser.

8.2.3 Hardness Test Method—Determine hardness measurements in accordance with Test Methods E 18. Make core hardness measurements on sectioned parts within the core region of the part. Determine surface hardness measurements in accordance with the applicable part drawing on the original forged surface, or, if machined, on the machined part surface.

9. Microstructure Requirements

9.1 Surface Finger Oxide Penetration:

9.1.1 The maximum depth of penetration of surface finger oxides from the finished part surface, for each designated critical region of a powder forged part, shall not exceed that agreed upon between the manufacturer and purchaser. Designate critical regions on the applicable part drawing or purchase order.

9.1.2 Determine the surface finger oxide penetration in accordance with Test Method B 797.

9.2 Interparticle Oxide Networks:

9.2.1 The extent of any interparticle oxide networks in each designated critical region of a powder forged part shall not exceed that agreed upon between the manufacturer and purchaser. Designate critical regions on the applicable part drawing or purchase order.

9.2.2 Determine the interparticle oxide networks in accordance with Test Method B 797.

9.3 Decarburization Depth:

9.3.1 The maximum depth of complete decarburization (only ferrite present) of surfaces of powder forged parts shall not exceed that agreed between the manufacturer and purchaser.

9.3.2 The depth of total decarburization (total decarburization = complete decarburization + partial decarburization), the depth at which core carbon content is reached, shall not exceed that agreed between the manufacturer and purchaser. Alternatively, for quenched and tempered parts, an effective decarburization depth (depth to a specified hardness) may be specified.

9.3.3 Determine the depth of complete decarburization by the microscopical method in accordance with Test Method E 1077.

9.3.4 Depth of total or effective decarburization.

9.3.4.1 Slow-Cooled or Normalized Parts—Estimate the depth of total decarburization of slow-cooled or normalized parts microscopically from the sum of the depths of complete and partial decarburization in accordance with Test Method E 1077.

9.3.4.2 Quenched and Tempered Parts—Determine the depth of effective decarburization by the microhardness method in accordance with Test Method E 1077.

9.4 Nonmetallic Inclusion Level:

9.4.1 The nonmetallic inclusion level of Grade A powder forged parts shall not exceed that specified by the applicable part drawing or purchase order.

9.4.2 Determine the nonmetallic inclusion level in accordance with Test Method B 796. For materials that contain
manganese sulfide additions, modify the inclusion assessment to count only those discrete inclusions greater than or equal to 100 μm maximum caliper (Feret’s) diameter. 

**NOTE 3—**Porosity dominates the mechanical properties of Grade B parts. Inclusion assessment of Grade B parts is therefore not necessary.

9.5 Cross Product Contamination:
9.5.1 The amount of unalloyed iron contamination of P/F – 42XX, P/F – 46XX, P/F – 44XX, or P/F – 49XX, or alloyed iron contamination of P/F – 10XX, P/F – 10CXX, P/F – 11XX, or P/F – 11CXX parts shall not exceed that agreed between the manufacturer and purchaser.
9.5.2 Determine alloyed or unalloyed iron contamination in accordance with Test Method B 795.

10. Sampling
10.1 Lot—Unless otherwise specified, a lot shall consist of parts of the same form and dimensions, produced from a single mixed powder batch, compacted, and processed under the same conditions and submitted for inspection at one time.

10.2 Chemical Analysis—When a full chemical analysis is requested on the purchase order, either the chemical analysis provided by the powder supplier for the specific lot of powder used to make the parts, or, testing of the forged parts, may be used to meet this requirement. However, the forged carbon content, and oxygen content when specified, shall be measured on the forged parts. Take samples from the core area of the part. For spectrometric analysis, the sample shall consist of a single solid piece carefully cut using a cutting fluid to prevent overheating. After cutting, wash the piece with low residue acetone to remove the cutting fluid and dry with compressed air. For carbon and wet chemical analysis, remove drillings, chips, or solid pieces without the use of water, oil, or other lubricant, and with care to prevent overheating. Take care to keep dirt and foreign substances out of the sample.

10.3 Mechanical Tests—The manufacturer and purchaser shall mutually agree on a representative number of specimens for qualification testing or mechanical property testing.

11. Inspection
11.1 Inspection of the parts supplied under this specification shall be the responsibility of the manufacturer or a mutually agreed upon third party.
11.2 If the purchaser desires that his representative inspect or witness the inspection and testing of the material prior to shipment, such a requirement shall be part of the purchase order.

12. Rejection and Rehearing
12.1 Parts that fail to conform to the requirements of this specification may be rejected. Rejection should be reported to the manufacturer or supplier promptly and in writing. In case of dissatisfaction with the results of the test, the manufacturer or supplier may make claim for a rehearing.

13. Certification
13.1 When specified in the purchase order, furnish a manufacturer’s certification to the purchaser that the parts were manufactured, sampled, tested, and inspected in accordance with this specification and have been found to meet the requirements. When specified in the purchase order, furnish a report of the test results.

14. Keywords
14.1 powder forged (P/F) parts; powder forged (P/F) steels; powder forging (P/F)

**SUPPLEMENTARY REQUIREMENTS**
The following supplementary requirements shall apply only when specified by the purchaser in the purchase order.

**S1. Forged Oxygen Content**

S1.1 The maximum permissible oxygen content of forged parts shall be as specified by the applicable part drawing or the purchase order.

S1.2 Take at least one sample for analysis from each lot. Take samples from the core region of the part. The sample shall consist of a single solid piece carefully cut with a low speed precision cut-off wheel using cutting fluid to prevent overheating. After cutting, wash the sample with low residue acetone to remove the cutting fluid and dry with compressed air. For carbon and wet chemical analysis, remove drillings, chips, or solid pieces without the use of water, oil, or other lubricant, and with care to prevent overheating. Take care to keep dirt and foreign substances out of the sample.

**S2. Case Hardening**

S2.1 The effective case depth of surface hardened (for example, carburized or carbonitrided) powder forged steel parts shall meet the range specified by the applicable part drawing or as agreed upon by the manufacturer and purchaser.

S2.2 Determine the effective case depth of surface hardened parts in accordance with SAE Recommended Practice, J 423, using the hardness traverse procedure.

**S3. Critical Region Porosity**

S3.1 The maximum area percent porosity plus oxide inclusions of each designated critical region of a powder forged part shall not exceed that agreed upon between the manufacturer and purchaser. Designate critical regions on the applicable part drawing or purchase order.

S3.2 Determine the critical region percentage porosity plus oxide inclusions in accordance with Practice E 562 or by an agreed upon automated image analysis method.

**S4. Microstructural Uniformity**

S4.1 Microstructural uniformity requirements for powder
forged parts shall be agreed upon between the manufacturer and purchaser.

S4.2 Remove a metallographic specimen representing the specified region or regions of the part for examination. Prepare the specimen following the procedures described in Methods E3.

APPENDIX

(Nonmandatory Information)

X1. MECHANICAL PROPERTIES OF POWDER FORGED (P/F) FERROUS STRUCTURAL PARTS

X1.1 Data for typical mechanical properties of Grade A powder forged ferrous structural parts are given in Table X1.1. The data do not constitute a part of the specification. They are merely intended to indicate to the purchaser the typical mechanical properties that may be expected from specimens machined from sample blanks of the chemical composition and heat treatment condition specified.

TABLE X1.1 Carbon Steel

<table>
<thead>
<tr>
<th>Material Code Designation</th>
<th>Heat Treat Condition</th>
<th>Ultimate Strength (10^3 psi)</th>
<th>Yield Strength (10^3 psi)</th>
<th>Elongation (in 1 in.)</th>
<th>Reduction of Area (%)</th>
<th>Hardness (Rockwell)</th>
<th>Impact Energy (ft·lb)</th>
<th>Mean Fatigue Limit (psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P/F-1020 N</td>
<td></td>
<td>64</td>
<td>50</td>
<td>30</td>
<td>55</td>
<td>70 HRB</td>
<td>20</td>
<td>55</td>
</tr>
<tr>
<td>P/F-1040 N</td>
<td></td>
<td>75</td>
<td>45</td>
<td>27</td>
<td>50</td>
<td>75 HRB</td>
<td>3</td>
<td>60</td>
</tr>
<tr>
<td>P/F-1060 N</td>
<td></td>
<td>140</td>
<td>120</td>
<td>12</td>
<td>42</td>
<td>30 HRC</td>
<td>15</td>
<td>110D</td>
</tr>
<tr>
<td>Q</td>
<td></td>
<td>125</td>
<td>100</td>
<td>12</td>
<td>42</td>
<td>30 HRC</td>
<td>15</td>
<td>110D</td>
</tr>
<tr>
<td>Q</td>
<td></td>
<td>195</td>
<td>175</td>
<td>8</td>
<td>25</td>
<td>40 HRC</td>
<td>10</td>
<td>130D</td>
</tr>
</tbody>
</table>

Typical Values

<table>
<thead>
<tr>
<th>Material Code Designation</th>
<th>Heat Treat Condition</th>
<th>Ultimate Strength (MPa)</th>
<th>Yield Strength (MPa)</th>
<th>Elongation (25.4 mm)</th>
<th>Reduction of Area (%)</th>
<th>Hardness (Rockwell)</th>
<th>Impact Energy (J)</th>
<th>Mean Fatigue Limit (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P/F-1020 N</td>
<td></td>
<td>440</td>
<td>340</td>
<td>30</td>
<td>55</td>
<td>70 HRB</td>
<td>27</td>
<td>380</td>
</tr>
<tr>
<td>P/F-1040 N</td>
<td></td>
<td>250</td>
<td>310</td>
<td>27</td>
<td>50</td>
<td>75 HRB</td>
<td>4</td>
<td>410</td>
</tr>
<tr>
<td>P/F-1060 N</td>
<td></td>
<td>970</td>
<td>830</td>
<td>12</td>
<td>42</td>
<td>30 HRC</td>
<td>20</td>
<td>760P</td>
</tr>
<tr>
<td>Q</td>
<td></td>
<td>860</td>
<td>690</td>
<td>12</td>
<td>37</td>
<td>90 HRC</td>
<td>3</td>
<td>450D</td>
</tr>
<tr>
<td>Q</td>
<td></td>
<td>1340</td>
<td>1210</td>
<td>8</td>
<td>25</td>
<td>40 HRC</td>
<td>14</td>
<td>900P</td>
</tr>
</tbody>
</table>

Data from MPIF Standard 35.

Mechanical property data derived from laboratory-prepared test specimens sintered and forged under commercial manufacturing conditions.

N: Normalized condition (austenitize and cool in still air)

Q: Quenched and tempered condition (austenitized, oil quenched and tempered 1 h at temperature to Rockwell C hardness level indicated)

D: For these heat-treated steels, the hardenability of the alloy is not sufficient to completely through harden the 0.375-in. (9.53-mm) diameter test specimen. Typically, smaller cross sections have higher compressive yield strengths and larger sections somewhat lower strengths due to the hardenability response of the materials.
### TABLE X1.2 Copper Steel

<table>
<thead>
<tr>
<th>Material Code Designation</th>
<th>Heat Treat Condition</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>Yield Strength (MPa)</th>
<th>Elongation (in 1 in.) (%)</th>
<th>Reduction of Area (%)</th>
<th>Hardness (Rockwell)</th>
<th>Impact Energy (ft·lbf)</th>
<th>Mean Fatigue Limit (10⁸ psi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P/F-10C40 N</td>
<td>N</td>
<td>100</td>
<td>70</td>
<td>18</td>
<td>38</td>
<td>97 HRB</td>
<td>3</td>
<td>90</td>
</tr>
<tr>
<td>P/F-10C50 N</td>
<td>N</td>
<td>120</td>
<td>80</td>
<td>16</td>
<td>30</td>
<td>22 HRC</td>
<td>2</td>
<td>95</td>
</tr>
<tr>
<td>P/F-10C60 N</td>
<td>N</td>
<td>125</td>
<td>85</td>
<td>11</td>
<td>27</td>
<td>24 HRC</td>
<td>2</td>
<td>100</td>
</tr>
<tr>
<td>P/F-11C40 N</td>
<td>N</td>
<td>95</td>
<td>70</td>
<td>14</td>
<td>36</td>
<td>98 HRB</td>
<td>4</td>
<td>85</td>
</tr>
<tr>
<td>Q</td>
<td>130</td>
<td>120</td>
<td>10</td>
<td>10</td>
<td>30</td>
<td>26 HRC</td>
<td>5</td>
<td>115</td>
</tr>
<tr>
<td>Q</td>
<td>190</td>
<td>160</td>
<td>7</td>
<td>7</td>
<td>25</td>
<td>38 HRC</td>
<td>7</td>
<td>125</td>
</tr>
<tr>
<td>P/F-11C50 N</td>
<td>N</td>
<td>125</td>
<td>85</td>
<td>15</td>
<td>30</td>
<td>24 HRC</td>
<td>4</td>
<td>90</td>
</tr>
<tr>
<td>P/F-11C60 N</td>
<td>N</td>
<td>130</td>
<td>90</td>
<td>11</td>
<td>23</td>
<td>28 HRC</td>
<td>3</td>
<td>90</td>
</tr>
</tbody>
</table>

**B 848**

### TABLE X1.3 Low Alloy P/F-42XX Steel

<table>
<thead>
<tr>
<th>Material Code Designation</th>
<th>Heat Treat Condition</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>Yield Strength (MPa)</th>
<th>Elongation (in 25.4 mm) (%)</th>
<th>Reduction of Area (%)</th>
<th>Hardness (Rockwell)</th>
<th>Impact Energy (J)</th>
<th>Mean Fatigue Limit (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>P/F-4220 N</td>
<td>N</td>
<td>75</td>
<td>55</td>
<td>25</td>
<td>55</td>
<td>84 HRB</td>
<td>25</td>
<td>60</td>
</tr>
<tr>
<td>Q</td>
<td>120</td>
<td>100</td>
<td>23</td>
<td>23</td>
<td>55</td>
<td>26 HRC</td>
<td>30</td>
<td>95</td>
</tr>
<tr>
<td>Q</td>
<td>175</td>
<td>140</td>
<td>9</td>
<td>9</td>
<td>35</td>
<td>38 HRC</td>
<td>25</td>
<td>105</td>
</tr>
<tr>
<td>P/F-4240 N</td>
<td>N</td>
<td>100</td>
<td>70</td>
<td>16</td>
<td>40</td>
<td>97 HRB</td>
<td>8</td>
<td>80</td>
</tr>
<tr>
<td>Q</td>
<td>130</td>
<td>120</td>
<td>15</td>
<td>15</td>
<td>40</td>
<td>28 HRC</td>
<td>20</td>
<td>130</td>
</tr>
<tr>
<td>Q</td>
<td>190</td>
<td>170</td>
<td>9</td>
<td>9</td>
<td>35</td>
<td>38 HRC</td>
<td>8</td>
<td>180</td>
</tr>
<tr>
<td>P/F-4260 N</td>
<td>N</td>
<td>110</td>
<td>75</td>
<td>15</td>
<td>30</td>
<td>22 HRC</td>
<td>5</td>
<td>80</td>
</tr>
<tr>
<td>Q</td>
<td>130</td>
<td>120</td>
<td>15</td>
<td>15</td>
<td>35</td>
<td>30 HRC</td>
<td>18</td>
<td>130</td>
</tr>
<tr>
<td>Q</td>
<td>190</td>
<td>170</td>
<td>9</td>
<td>9</td>
<td>32</td>
<td>38 HRC</td>
<td>14</td>
<td>180</td>
</tr>
<tr>
<td>Q</td>
<td>235</td>
<td>210</td>
<td>5</td>
<td>5</td>
<td>25</td>
<td>45 HRC</td>
<td>8</td>
<td>205</td>
</tr>
<tr>
<td>Q</td>
<td>280</td>
<td>255</td>
<td>&lt;1</td>
<td>25</td>
<td>25</td>
<td>54 HRC</td>
<td>3</td>
<td>255</td>
</tr>
</tbody>
</table>

### Typical Values

- **A** Mechanical property data derived from laboratory-prepared test specimens sintered and forged under commercial manufacturing conditions.
- **B** N: Normalized condition (austenitize and cool in still air)
- **Q**: Quenched and tempered condition (austenitized, oil quenched and tempered to Rockwell C hardness level indicated)
- **C** For these heat-treated steels, the hardenability of the alloy is not sufficient to completely through harden the 0.375-in. (9.53-mm) diameter test specimen. Typically, smaller cross sections have higher compressive yield strengths and larger sections somewhat lower strengths due to the hardenability response of the materials.
### TABLE X1.4 Low Alloy P/F-46XX Steel

<table>
<thead>
<tr>
<th>Material Code</th>
<th>Heat Treat Condition</th>
<th>Tensile Properties</th>
<th>Compressive Properties</th>
<th>Mean Fatigue Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Ultimate Strength</td>
<td>Yield Strength</td>
<td>Hardness</td>
</tr>
<tr>
<td></td>
<td></td>
<td>(10^3 psi)</td>
<td>(10^3 psi)</td>
<td>(Rockwell)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.2 % Offset</td>
<td>(in 1 in.)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Elongation (%)</td>
<td>Reduction of Area (%)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>(10^3 psi)</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Typical Values**

- **A** Mechanical property data derived from laboratory-prepared test specimens sintered and forged under commercial manufacturing conditions.
- **B** N: Normalized condition (austenitize and cool in still air)
- **Q**: Quenched and tempered condition (austenitized, oil quenched, and tempered to Rockwell C hardness level indicated.

### X1.2 Hardenability and Jominy Curves

#### X1.2.1 Hardenability

Hardenability is a measure of the depth of hardening that can be achieved; the higher the value, the more hardenable the steel. The hardenability depth was determined from a standard Jominy test (Test Method A 255) and the hardness versus depth curve produced using the Rockwell A (HRA) hardness scale. Jominy curves are provided. The depth, in sixteenths of an inch (mm), where the hardness value falls below 50 HRC (75.9 HRA) is listed as the J depth. If a P/F steel did not reach 50 HRC at the surface, the J depth is listed as <1.

### Material Designation J Depth in 1/16-in. Units

<table>
<thead>
<tr>
<th>Material Designation</th>
<th>J Depth in 1/16-in. Units</th>
<th>J Depth in mm Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>P/F-1020</td>
<td>&lt;1</td>
<td>&lt;1.5</td>
</tr>
<tr>
<td>P/F-1060</td>
<td>1.5</td>
<td>2</td>
</tr>
<tr>
<td>P/F-11C40</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td>P/F-4220</td>
<td>&lt;1</td>
<td>&lt;1.5</td>
</tr>
<tr>
<td>P/F-4240</td>
<td>1</td>
<td>5</td>
</tr>
<tr>
<td>P/F-4260</td>
<td>11</td>
<td>3</td>
</tr>
<tr>
<td>P/F-4620</td>
<td>&lt;1</td>
<td>&lt;1.5</td>
</tr>
<tr>
<td>P/F-4640</td>
<td>1</td>
<td>5</td>
</tr>
<tr>
<td>P/F-4660</td>
<td>13</td>
<td>21</td>
</tr>
<tr>
<td>P/F-4680</td>
<td>18</td>
<td>29</td>
</tr>
</tbody>
</table>

---

*Data from MPIF Standard 35.*
To select a material optimum in both properties and cost effectiveness, it is essential that the part application be discussed with the P/F parts manufacturer. Both the purchaser and manufacturer should, in order to avoid possible misconceptions or misunderstandings, agree on the following conditions prior to the manufacture of a P/F component: material selection, chemistry, proof testing, and typical property values and process, which may affect the part application.
Standard Specification for Pre-Treatments of Iron or Steel for Reducing Risk of Hydrogen Embrittlement

This standard is issued under the fixed designation B 849; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

When atomic hydrogen enters steels and certain other metals, for example, aluminum and titanium alloys, it can cause a loss of ductility, load carrying ability, or cracking (usually as submicroscopic cracks) as well as catastrophic brittle failures at applied stresses well below the yield strength or even the normal design strength for the alloys. This phenomenon often occurs in alloys that show no significant loss in ductility, when measured by conventional tensile tests, and is referred to frequently as hydrogen-induced delayed brittle failure, hydrogen stress cracking, or hydrogen embrittlement. The hydrogen can be introduced during cleaning, pickling, phosphating, electroplating, autocatalytic processes, porcelain enameling, and in the service environment as a result of cathodic protection reactions or corrosion reactions. Hydrogen can also be introduced during fabrication, for example, during roll forming, machining, and drilling, due to the breakdown of unsuitable lubricants as well as during welding or brazing operations. Parts that have been machined, ground, cold-formed, or cold-straightened subsequent to hardening heat treatment are especially susceptible to hydrogen embrittlement damage.

The results of research work indicate that the susceptibility of any material to hydrogen embrittlement in a given test is related directly to its trap population. The time-temperature relationship of the heat treatment is therefore dependent on the composition and structure of steels as well as plating metals and plating procedures. Additionally, for most high-strength steels, the effectiveness of the heat treatment falls off rapidly with a reduction of time and temperature.

1. Scope

1.1 This specification covers procedures for reducing the susceptibility or degree of susceptibility to hydrogen embrittlement or degradation that may arise in electroplating, autocatalytic plating, porcelain enameling, chemical conversion coating, and phosphating and the associated pretreatment processes. This specification is applicable to those steels whose properties are not affected adversely by baking at 190 to 230°C or higher (see 6.1.1).

1.2 The heat treatment procedures established herein have been shown to be effective for reducing the susceptibility of steel parts of tensile strength 1000 MPa or greater that have been machined, ground, cold-formed, or cold-straightened subsequent to heat treatment. This heat-treatment procedure is used prior to any operation capable of hydrogen charging the parts, such as the cleaning procedures prior to electroplating, autocatalytic plating, porcelain enameling, and other chemical coating operations.

NOTE 1—1 MPa = 145.1 psi.

1.3 This specification has been coordinated with ISO/DIS 9587 and is technically equivalent.

1.4 The values stated in SI units are to be regarded as the standard.

2. Referenced Documents

2.1 ASTM Standards:
A 919 Terminology Relating to Heat Treatment of Metals
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 322 Practice for Cleaning Metals Prior to Electroplating
B 374 Terminology Relating to Electroplating

3 Annual Book of ASTM Standards, Vol 02.05.
TABLE 1 Classes of Stress Relief Requirements for High-Strength Steels (See Sections 4 through 6 for Details on the Use of Table 1)

<table>
<thead>
<tr>
<th>Class</th>
<th>Stress-Relief Heat-Treatment Classes for High-Strength Steels</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Steel of Tensile Strength, (Rm), MPa</td>
</tr>
<tr>
<td>SR-0</td>
<td>not applicable</td>
</tr>
<tr>
<td>SR-1</td>
<td>over 1800</td>
</tr>
<tr>
<td>SR-2*</td>
<td>over 1800</td>
</tr>
<tr>
<td>SR-3</td>
<td>1401 to 1800</td>
</tr>
<tr>
<td>SR-4*</td>
<td>1450 to 1800</td>
</tr>
<tr>
<td>SR-5*</td>
<td>1034 or greater</td>
</tr>
<tr>
<td>SR-6</td>
<td>1000 to 1400</td>
</tr>
<tr>
<td>SR-7*</td>
<td>1050 to 1450</td>
</tr>
<tr>
<td>SR-8</td>
<td>surface-hardened parts ≤ 1400</td>
</tr>
</tbody>
</table>

* Classes SR-2, SR-4, SR-5, and SR-7 are traditional treatments used in Federal Standard QQ-C-320. They do not apply to any other standard.

3. Terminology

3.1 Definitions—Many of the terms used in this specification can be found in Terminology B 374, A 919, or ISO 2080.

4. Requirements

4.1 Heat treatment shall be performed on basis metals to reduce the risk of hydrogen embrittlement in accordance with Table 1. The duration of heat treatment shall commence in all cases from the time at which the whole of each part attains the specified temperature.

4.2 Parts made from steel with actual tensile strengths ≥ 1000 MPa (with corresponding hardness values of 300 HV10kgf, 303 HB, or 31 HRc) and surface-hardened parts shall require heat treatment unless Class SR-0 is specified. Preparation involving cathodic treatments in alkaline or acid solutions shall be avoided.

4.3 Table 1 lists the stress-relief heat-treatment classes to be specified by the purchaser to the electroplater, supplier, or processor on the part drawing or purchase order. When no stress relief treatment class is specified by the purchaser, Class SR-1 shall be applied (see Note 4).

Note: 2—The treatment class selected is based on experience with the part, or similar parts, and the specific alloy used or with empirical test data. Because of factors such as alloy composition and structure, size, mass, or design parameters, some parts may perform satisfactorily with no stress relief treatment. Class SR-0 treatment is therefore provided for parts that the purchaser wishes to exempt from treatment.

Note: 3—The use of inhibitors in acid pickling baths is not necessarily guaranteed to minimize hydrogen embrittlement.

Note: 4—Class SR-1, the longest treatment, is the default when the purchaser does not specify a class. The electroplater, supplier, or processor is not normally in possession of the necessary information, such as design considerations, induced stresses from manufacturing operations, etc., that must be considered when selecting the correct stress relief treatment. It is in the purchasers’ interest that their part designer, manufacturing engineer, or other technically qualified individual specify the treatment class on the part drawing or purchaser order in order to avoid the extra cost of the default treatment.

5. Categorization of Steels

5.1 With the exception of surface-hardened parts, heat treatment conditions shall be selected on the basis of actual tensile strength. When only the minimum tensile strength is specified, or if the tensile strength is not known, the heat treatment condition shall be selected by relating known or measured hardness values to equivalent actual tensile strengths. The tensile strength shall be supplied by the purchaser.

5.2 Steels that have been wholly or partly surface hardened shall be considered as being in the category appropriate to the hardness of the surface-hardened layer.

6. Stress Relief

6.1 For high-strength steels, the following conditions apply. Stress relief treatment is not essential for steels of actual tensile strength below 1000 MPa. The conditions given in Table 1 are applied for steels of actual tensile strength above 1000 MPa. The heat treatment shall be conducted before the commencement of any preparations or cleaning treatments using processes liable to cause embrittlement such as cathodic electro- cleaning or acid pickling. Other cleaning processes, such as those described in Practices B 242 or B 322, may be used.

6.1.1 Suitable combinations of a shorter time at appropriate higher temperatures may be used if they have been shown not to be detrimental. For tempered steels, items shall not be heated above a temperature that shall be at least 50°C below the tempering temperature.

6.1.2 If stress relief is given after shot peening in accordance with Specification B 851 or other cold working processes, the stress shall not exceed 230°C.

6.1.3 Items having surface-hardened areas that would suffer an unacceptable reduction in hardness by treatment in accordance with Table 1 shall be heat-treated at a lower temperature, but not below 130°C, for a minimum period of 8 h. This treatment is applicable for items made of steel with actual tensile strengths below 1400 MPa.

7. Keywords

7.1 delayed brittle failure; heat treatment; hydrogen embrittlement; hydrogen induced cracking; hydrogen stress cracking; pre-treatments of iron or steel; stress relief
Standard Guide for
Post-Coating Treatments of Steel for Reducing Risk of
Hydrogen Embrittlement

INTRODUCTION

When atomic hydrogen enters steel, it can cause a loss of ductility, load carrying ability, or cracking (usually as submicroscopic cracks), as well as catastrophic brittle failures at applied stresses well below the yield strength or even the normal design strength for the alloys. This phenomenon often occurs in alloys that show no significant loss in ductility, when measured by conventional tensile tests, and is referred to frequently as hydrogen-induced delayed brittle failure, hydrogen stress cracking, or hydrogen embrittlement. The hydrogen can be introduced during cleaning, pickling, phosphating, electroplating, autocatalytic processes, porcelain enameling, and in the service environment as a result of cathodic protection reactions or corrosion reactions. Hydrogen can also be introduced during fabrication, for example, during roll forming, machining, and drilling, due to the breakdown of unsuitable lubricants, as well as during welding or brazing operations.

1. Scope

1.1 This guide covers procedures for reducing the susceptibility in some steels to hydrogen embrittlement or degradation that may arise in the finishing processes.

1.2 The heat treatment procedures established herein may be effective for reducing susceptibility to hydrogen embrittlement. This heat-treatment procedure shall be used after plating operations but prior to any secondary conversion coating operation.

1.3 This guide has been coordinated with ISO/DIS 9588 and is technically equivalent.

Note 1—The heat treatment does not guarantee complete freedom from the adverse effects of hydrogen degradation.

2. Referenced Documents

2.1 ASTM Standards:
A 919 Terminology Relating to Heat Treatment of Metals
B 374 Terminology Relating to Electroplating
B 851 Specification for Automated Controlled Shot Peening of Metallic Articles Prior to Nickel, Autocatalytic Nickel, or Chromium Plating, or as Final Finish

2.2 ISO Standards:
ISO 2080 Electroplating and Related Processes—Vocabulary
ISO DIS 9588 Post-Coating Treatments of Iron or Steel for Reducing the Risk of Hydrogen Embrittlement

2.3 Federal Standard:
QQ-C-320 Chromium Plating (Electrodeposited)

3. Terminology

3.1 Definitions—Many of the terms used in this guide can be found in Terminology B 374, A 919, or ISO 2080.

4. Requirements

4.1 Heat treatment may be performed on coated metals to reduce the risk of hydrogen embrittlement. The duration of heat treatment in all cases shall commence from the time at which the whole of each part attains the specified temperature.

4.2 Parts made from steel with actual tensile strengths \( \geq 1000 \) MPa (with corresponding hardness values of 300 HV 10kgf, 303 HB, or 31 HRc) and surface-hardened parts may require heat treatment unless Class ER-0 is specified. Preparation involving cathodic treatments in alkaline or acid solutions shall be avoided. Additionally, the selection of electroplating solutions with high cathodic efficiencies is recommended for...
steel components with tensile strengths above 1400 MPa (with corresponding hardness values of 425 HV_{10kgf}, 401 HB, or 43 HR_{C}).

4.3 Table 1 provides a list of embrittlement-relief heat-treatment classes from which the purchaser may specify the treatment required to the electroplater, supplier, or processor on the part drawing or purchase order.

**Note 2**—The treatment class selected is based on experience with the part, or similar parts, and the specific alloy used or with empirical test data. Because of factors such as alloy composition and structure, type of coating, coating thickness, size, mass, or design parameters, some parts may perform satisfactorily with no embrittlement-relief treatment. Class ER-0 treatment is therefore provided for parts that the purchaser wishes to exempt from treatment.

**Note 3**—The use of inhibitors in acid pickling baths may not minimize hydrogen embrittlement.

4.4 The electroplater, supplier, or processor is not normally in possession of the necessary information, such as design considerations, operating stresses, etc., that must be considered when selecting the correct embrittlement relief treatment. It is in the purchaser’s interest that his or her part designer, manufacturing engineer, or other technically qualified individual specify the treatment class on the part drawing or purchase order.

### TABLE 1 Classes of Embrittlement-Relief Heat Treatment (See Sections 4-6 for details on the Use of Table 1)

<table>
<thead>
<tr>
<th>Class</th>
<th>Steels of Tensile Strength (Rm), MPa</th>
<th>Temperature, °C</th>
<th>Time, h</th>
</tr>
</thead>
<tbody>
<tr>
<td>ER-0</td>
<td>not applicable</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ER-1</td>
<td>1701 to 1800</td>
<td>190–220</td>
<td>min 22</td>
</tr>
<tr>
<td>ER-2</td>
<td>1601 to 1700</td>
<td>190–220</td>
<td>min 20</td>
</tr>
<tr>
<td>ER-3</td>
<td>1501 to 1600</td>
<td>190–220</td>
<td>min 18</td>
</tr>
<tr>
<td>ER-4</td>
<td>1401 to 1500</td>
<td>190–220</td>
<td>min 16</td>
</tr>
<tr>
<td>ER-5</td>
<td>1301 to 1400</td>
<td>190–220</td>
<td>min 14</td>
</tr>
<tr>
<td>ER-6</td>
<td>1201 to 1300</td>
<td>190–220</td>
<td>min 12</td>
</tr>
<tr>
<td>ER-7a</td>
<td>1525 or greater</td>
<td>177–205</td>
<td>min 12</td>
</tr>
<tr>
<td>ER-8</td>
<td>1101 to 1200</td>
<td>190–220</td>
<td>min 10</td>
</tr>
<tr>
<td>ER-9</td>
<td>1000 to 1100</td>
<td>190–220</td>
<td>min 8</td>
</tr>
<tr>
<td>ER-10a</td>
<td>1250 to 1525</td>
<td>177–205</td>
<td>min 8</td>
</tr>
<tr>
<td>ER-11a</td>
<td>1450 to 1800</td>
<td>190–220</td>
<td>min 6</td>
</tr>
<tr>
<td>ER-12a</td>
<td>1000 to 1500</td>
<td>177–205</td>
<td>min 4</td>
</tr>
<tr>
<td>ER-13</td>
<td>1000 to 1800 unpawned items</td>
<td>440–480</td>
<td>min 1</td>
</tr>
<tr>
<td></td>
<td>and for engineering chromium</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>plated items</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ER-14a</td>
<td>surface-hardened parts &lt;1401</td>
<td>130–160</td>
<td>min 8</td>
</tr>
<tr>
<td>ER-15a</td>
<td>surface-hardened parts 1401 to 1800 platted with cadmium, tin, zinc, or their alloys</td>
<td>130–160</td>
<td>min 8</td>
</tr>
<tr>
<td>ER-16</td>
<td>surface-hardened parts &lt;1401 plated with cadmium, tin, zinc, or their alloys</td>
<td>130–160</td>
<td>min 16</td>
</tr>
</tbody>
</table>

aClasses ER-7, ER-10, ER-11, ER-12, ER-14, and ER-15 are traditional treatments used in Federal Standard QQ-C-320. They do not apply to any other standard.

5. Embrittlement Relief Treatment Classes

5.1 With the exception of surface-hardened parts and parts that have been shot peened in accordance with Specification B 851, heat treatment conditions may be selected on the basis of actual tensile strength. When only the minimum tensile strength is specified, or if the tensile strength is not known, the heat treatment condition may be selected by relating known or measured hardness values to equivalent tensile strengths. It is recommended that the tensile strength be supplied by the purchaser.

5.2 Steels that have been wholly or partly surface hardened may be considered as being in the category appropriate to the hardness of the surface-hardened layer.

5.3 If the purchaser requires any tests to be performed in order to verify adequate embrittlement relief treatment, the test method and the sampling plan to be used shall be specified.

6. Heat Treatment After Processing

6.1 The heat treatment shall commence as soon as possible, preferably within 1 h but not later than 3 h after plating and before commencement of any grinding or other mechanical operation. For cadmium, tin, zinc, their alloys, or any other coating receiving a chromate treatment, heat treatment shall be conducted before chromate treatment.

**Note 4**—Chromate coatings undergo change at temperatures above 66°C. The coating changes from an amorphous structure to a crystalline structure and no longer exhibits self-healing properties. While the crystallized chromate coating will provide satisfactory corrosion protection under most natural environments, the chromate coating will no longer pass accelerated corrosion tests.

**Note 5**—The time period referred to is the length of time between the end of the plating operation and loading of the item concerned into the heat treatment processor.

6.2 For high-strength steels, the conditions given in Table 1 may be applied. For steels of actual tensile strength below 1000 MPa, heat treatment after plating is not essential.

6.3 Electroplated steel items having surface-hardened areas and through hardened or bearing steels, which would suffer an unacceptable reduction in hardness by treatment in accordance with Table 1 shall be heat treated at a lower temperature, but not below 130°C.

6.4 Treatment at 440 to 480°C will reduce the hardness of chromium deposits. It shall not be applied to steels that may be affected adversely by heat treatment at this temperature, and the lower temperature range shall be applied. For tempered steels, items shall not be heat treated above a temperature that shall be 50°C below the tempering temperature.

7. Keywords

7.1 delayed brittle failure; heat treatment; hydrogen embrittlement; hydrogen embrittlement relief; hydrogen induced cracking; hydrogen stress cracking; post-treatment of steel
Standard Specification for Automated Controlled Shot Peening of Metallic Articles Prior to Nickel, Autocatalytic Nickel, or Chromium Plating, or as Final Finish

1. Scope

1.1 This specification covers the requirements for automated, controlled shot peening of metallic articles prior to electrolytic or autocatalytic deposition of nickel or chromium, or as a final finish, using shot made of cast steel, conditioned cut wire, or ceramic media. The process is applicable to those materials on which test work has shown it to be beneficial within given intensity ranges. It is not suitable for brittle materials. Hand peening and rotary flap peening are excluded specifically.

1.2 Shot peening induces residual compressive stresses in the surface and near-surface layers of metallic articles, controlling or limiting the reduction in fatigue properties that occurs from nickel or chromium plating of the article, or the fatigue properties of unplated articles.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
   B 242 Guide for Preparation of High-Carbon Steel For Electroplating
   B 320 Practice for Preparation of Iron Castings for Electroplating
   B 322 Practice for Cleaning Metals Prior to Electroplating
   B 607 Specification for Autocatalytic Nickel Boron Coatings for Engineering Use
   B 650 Specification for Electrodeposited Engineering Chromium Coatings on Ferrous Substrates
   B 656 Guide for Autocatalytic (Electroless) Nickel-Phosphorus Deposition on Metals for Engineering Use
   B 689 Specification for Electroplated Engineering Nickel Coatings
   B 733 Specification for Autocatalytic (Electroless) Nickel-Phosphorus Coatings on Metal
   E 11 Specification for Wire Cloth and Sieves for Testing Purposes
   E 709 Guide for Magnetic Particle Examination
   2.2 Federal Standards:
   QQ-N-290 Nickel Plating (Electrodeposited)
   QQ-C-320 Chromium Plating (Electrodeposited)
   2.3 Military Standards:
   MIL-S-851 Steel Grit, Shot, and Cut Wire Shot, and Iron Grit and Shot Blast Cleaning and Peening
   MIL-S-13165 Shot Peening of Metal Parts
   MIL-C-26074 Coating, Electroless Nickel
   MIL-STD-45662 Calibration System Requirements
   2.4 SAE Standards:
   SAE J441 Cut Steel Wire Shot
   SAE J442 Test Strip, Holder and Gage for Shot Peening
   SAE J827 Cast Steel Shot
   SAE J1830 Size, Classification and Characteristics of Ceramic Shot for Peening

3. Terminology

3.1 Definitions:
   3.1.1 Almen strip—UNS G10700 carbon steel specimens that are used to calibrate the energy of a shot peening stream (see Fig. 1).
   3.1.2 Almen strip holding fixture—a fixture for holding Almen strips in suitable locations that represent the position and angular orientation of the surfaces of a part where intensity is to be determined and verified (see Fig. 2).
3.1.3 **arc height**—flat Almen strips, when subjected to a stream of shot moving at an adequate velocity, bending in an arc corresponding to the amount of energy transmitted by the shot stream. The height of the curved arc measured in millimeters is the arc height, measured by an Almen gage (see Fig. 3).

3.1.4 **automatic equipment**—shot peening equipment in which parts, fixtures, nozzles, and peening parameters are preset by hand or by locating fixtures and verified by inspection personnel. The peening time is monitored automatically, and the air pressure or wheel speed is set manually.

3.1.5 **compressive stresses**—cold working or stretching the surface beyond the elastic limit by shot peening, creating a layer in compression below the surface. The depth of compressive stresses is measured by the crown of the dimple to the depth.

3.1.6 **coverage**—the extent of obliteration of the original surface by dimples produced by impact from individual shot particles and expressed as a percentage. See Note 1.

3.1.7 **depth of compressive stresses**—where the stress profile passes through 0 stress.

3.1.8 **intensity**—the Almen strip arc height at saturation. Arc height is not termed intensity correctly unless saturation is achieved.

3.1.9 **liquid tracer system**—a liquid coating material bearing a pigment that fluoresces under an ultraviolet light and removes at a rate proportioned to peening coverage.

3.1.10 **microprocessor-controlled equipment**—peening equipment that has nozzle holding fixtures and is computer controlled for processing, monitoring, and documentation of the peening parameters critical to process certification.

3.1.11 **nozzle holding fixture**—a fixture that holds the nozzles at the required location, distance, and angle in a locked position during the peening operation.

3.1.12 **process interrupt parameters**—for critical peening operations, parameters such as shot flow, air pressure, part r/min, oscillation rate, and cycle time that must be monitored within process requirements.

3.1.13 **saturation**—the minimum duration of peening necessary to achieve the desired Almen intensity which, when doubled, does not increase the Almen strip arc height by more than 10%.
3.1.14 saturation curve—a curve that plots peening time on the Almen strip (abscissa) versus Almen strip arc height (ordinate) achieved for the peening time (see Fig. 4).

3.1.15 surface obliteration—the condition of a peened surface in which 100% of the surface has been dimpled with shot impressions.

NOTE 1—A 100% coverage is defined as that leaving unpeened 2% or less of the original surface because the estimation of coverage of the impressions is difficult when this is approximately 98% of the total surface. The 100% coverage is a theoretical limiting value. Hence, the term complete coverage is preferred. Complete coverage usually requires increasing the base time, that is, the time of peening to reach 98% coverage, by 15 to 20%. Values of 200%, 300%, etc. are obtained by multiplying this run time by 2, 3, etc.

4. Ordering Information

4.1 When ordering articles to be shot peened, the purchaser shall state the following:
4.2 ASTM designation.
4.3 Type, size, and hardness of shot to be used (see 6.1).
4.4 Number and frequency of the determinations of shot size and uniformity required, if other than those specified in 8.1.1.
4.5 Peening intensity to be used at each location (see 8.2).
4.6 Number, frequency, and location of Almen test specimens to be provided for intensity verification and monitoring of the process if other than those specified in 8.2, 8.2.1, and 8.2.2.
4.7 Areas on the part that are to be shot peened and those to be protected from the peening (see 7.5).
4.8 Whether magnetic particle or penetrant inspection is required before peening (see 7.2).
4.9 Amount (percent) of coverage required in the areas to be peened. A complete coverage is the minimum requirement (see 3.1.6 and 8.3).
4.10 Method for measuring coverage (see 8.3.1).
4.11 Type of equipment to be used, automated or computer-monitored microprocessor (see 6.3 and X1.10-X1.12).
4.12 Details of any post treatment such as corrosion protection (see 9.5).
4.13 Requirements of certification and test records, as specified in Section 10.

5. Significance and Use

5.1 Shot peening is a process for cold working surfaces by bombarding the product with shot of a solid and spherical nature propelled at a relatively high velocity. In general, shot peening will increase the fatigue life of a product that is subject to bending or torsional stress. It will improve resistance to stress corrosion cracking. It can be used to form parts or correct their shapes. See Appendix X1 for additional information.

5.2 It is essential that the shot peening process parameters be controlled rigidly to ensure repeatability from part to part and lot to lot.

5.3 This specification covers techniques and methods necessary for proper control of the shot peening process.

6. Materials and Equipment

6.1 Shot Material Composition:

6.1.1 Cast Steel—Cast steel shot shall conform to the requirements of SAE J827.
6.1.2 Cut Wire—Cut wire shot shall be made from cold finished, round wire, confirming to SAE J441.
6.1.3 Ceramic Shot—Ceramic beads shall conform to the chemical composition given in Table 1 and to SAE J1830.
6.1.4 Shot Form and Shape:

6.1.4.1 Cast Steel—Cast steel shot shall be spherical in shape and free of sharp edges, corners, and broken pieces. It shall conform to the acceptable shapes given in Fig. 5. The number of nonconforming shapes (see Fig. 6) shall not exceed the values given in Table 2.

6.1.4.2 Cut Wire—Cut wire shot shall be spherical in shape. It shall be free of sharp edges, corners, and broken pieces. The number of nonconforming shapes shall not exceed the values given in Table 2.

6.1.4.3 Ceramic Shot—Ceramic shot shall be spherical in shape and free of sharp edges, corners, and broken pieces. The number of nonconforming shapes shall not exceed the values given in Table 2.

6.1.5 Hardness—The hardness of the media shall exceed that of the material to be processed.

6.1.5.1 Cast Steel—Cast steel shot shall have a hardness of HRc 45 to HRc 55. Special hard cast steel shot shall be used on products harder than HRc 50 and shall have a hardness of HRc 55 to HRc 65.
6.1.5.2 Cut Wire—Cut wire shot shall have a hardness equal to or greater than that given in Table 3.
6.1.5.3 Ceramic Shot—Ceramic shot shall have a minimum hardness of 560 HV30 (30 kgf).

6.1.6 Size:

(1) The size of the media shall be capable of producing the required intensity in the required time.

(2) If a peened surface contains a fillet, the nominal size of the shot shall not exceed one-half of the radius of the fillet.

(3) If the shot must pass through an opening, such as a slot, to reach a peened surface the nominal diameter of the shot shall not exceed one-fourth of the width or the diameter of the opening.

6.1.6.1 Cast Steel—Cast steel shot charged into a machine shall conform to the screen requirements given in Table 4 for

<table>
<thead>
<tr>
<th>Table 1 Composition of Ceramic Shot</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZrO2, %</td>
</tr>
<tr>
<td>60.0–70.0</td>
</tr>
</tbody>
</table>
the nominal size selected. When a machine has a completely new charge of cast steel shot, conditioning shall be conducted, to remove the oxide layers on the shots, by bombarding onto a hardened steel surface for a minimum of two passes. Conditioning may not be required if the addition to the charge already in the machine is below 25%. If the addition of over 25% is made to the charge, conditioning is required.

6.1.6.2 Cut Wire—The diameter of cut wire shot charged into a machine shall conform to the requirements given in Table 4. Cut wire shot shall conform to the requirements of length and cumulative weight given in Table 5. It is mandatory that only preconditioned cut wire shot be used.

6.1.6.3 Ceramic Shot—Ceramic shot charged into the peening machine shall conform to the screen requirements of Table 6.

6.2 Almen Strips, Blocks, and Gages—Almen strips, blocks, and gages used shall meet the requirements of SAE J442.

6.3 Equipment—Shot peening shall be conducted in a machine that is designed for the purpose, propels shot at high speed against the product, moves the product through the shot stream in a way that ensures complete and uniform peening, and screens the shot continuously to remove broken or defective shot.

7. Pre-Peening Treatment

7.1 Prior Operations—Areas of parts to be shot peened shall be within dimensional requirements before peening. Except as otherwise permitted, all heat treatment, machining, and grinding shall be completed before shot peening. All filets shall be formed, all burrs removed, and all sharp edges and corners that require peening provided with sufficient radii prior to peening, in order to result in complete coverage without any distortion, chipping, or rollover.

7.2 Flaw and Crack Testing—When required, magnetic particle, penetrant, ultrasonic, or other flaw or crack detection processes shall be completed prior to peening. See Test Method E 165 and Guide E 709.

7.3 Corrosion and Damage—Parts shall not be peened if they show evidence of invasive corrosion or mechanical damage on the surface.

7.4 Cleaning—Cleaning prior to peening shall be accomplished by vapor degreasing, solvent wiping, warm solvent spray, or an acceptable water-base nonflammable product, as required, to remove all soils, scale, and coatings from the surface areas to be peened. See Practices B 183, B 242, B 320, and B 322.

7.5 Masking—Surfaces designated on the drawing to be free of shot peening marks shall be masked or otherwise protected from the shot stream or indirect impingement by shot.

7.5.1 Suitable masking materials are adhesive tape, sheet rubber, etc. If adhesive tape is used, it shall be coated on one face with adhesive, and when the tape is removed from the surface it shall not show any evidence of corrosion or leave any residue on the surface. Areas not requiring peening and not required to be masked shall be considered optional.

8. Procedure

8.1 Shot—Shot charged into the peening machine shall be as specified by the purchaser and meet the requirements of 6.1 for the particular type, size, and material required. Unless otherwise specified, all shot shall be maintained in the machine so that it conforms to the requirements of Table 7.

8.1.1 Uniformity Determination—At least one determination for shot size and uniformity in accordance with Table 7 shall be made before and after each production run and after each 8 h of production on long runs, when using cast or cut
wire steel shot. Ceramic shot size distribution shall be verified at least every 4 h of production and before and after each production run.

8.2 Peening Intensity—The peening intensity should be that specified by the purchaser as the arc height produced by the peening process at saturation, as measured on Almen strips placed in the required locations. Unless otherwise specified on the drawing or in the contract, the intensity of peening shall be as specified in Table 8 for the thickness involved.

8.2.1 Saturation Curve—For initial process development, a saturation curve shall be generated for each location where intensity is to be verified.

8.2.2 Intensity Determination—At least one intensity determination for all required locations shall be made immediately before and after each production run and at least every 8 h of continuous running. The intensity determination is also required after any replacement of shots, a new setting, or any other change of setting of the machine, as well as after any event that may affect the shot peening operation.

8.3 Peening Coverage—Peened surfaces shall be uniform in appearance and completely dent so that the original surface is obliterated entirely. The extent (in percent) of coverage shall be specified by the purchaser. Complete coverage is full and complete obliteration of the original surface.

8.3.1 Coverage Determination—Unless otherwise specified, at least one coverage determination for all areas requiring peening shall be made immediately before and after each production run and at least every 8 h of continuous running. Coverage shall be determined by either of the following methods, as specified by the purchaser:

8.3.1.1 Visual examination using a ten-power magnifying glass. This procedure is not recommended for large areas.

8.3.1.2 Visual examination using an approved impact-sensitive liquid fluorescent tracer system in accordance with the manufacturer’s recommendations.

8.4 Computer-Monitored Equipment—When auxiliary computer-monitored equipment is used for shot peening, calibration of the monitored systems shall be in accordance with MIL-STD-45662. Intensity verification in accordance with 8.2 shall be conducted prior to initial operation and after calibration.

9. Post-Peening Treatment

9.1 Residual Shot Removal—After shot peening and the removal of protecting masks, all shot and shot fragments shall be removed from the surfaces of articles by methods that will not erode, scratch, or degrade the surfaces in any way.

9.2 Surface Finish Improvement—It is permissible to improve the surface finish of a component after shot peening by polishing, lapping, or honing, provided that the surface temperature is not raised sufficiently to relax the compressive stresses and the amount of material removed is less than 10 % of the depth of the compressive layer induced by peening.

9.3 Nonferrous Materials—Nonferrous metals and their alloys that have been shot peened shall be cleaned by an approved chemical cleaning solution to remove all iron contaminants. Cleaning operations shall not degrade the surface or alter the dimensions of the part. Cleared surfaces shall be chemically tested for freedom from residual iron by the method given in Appendix X2.

9.4 Thermal and Mechanical Treatment Limits—No manufacturing operations that relieve compressive stresses or develop detrimental residual stresses shall be permitted after shot peening. When parts are heated after shot peening, as for baking of paint or protective coatings, embrittlement relief...
after electroplating, or other thermal treatment, the temperatures used shall be limited as shown in Table 9.

9.5 Protection From Corrosion—Shot peened parts shall be protected from corrosion during processing and until final preservation and packaging are complete. All shot peened parts shall be preserved, wrapped, or packaged, as specified by the purchaser, to ensure protection from corrosion and damage during handling, transportation, and storage.

10. Certification and Test Records

10.1 When specified in the purchase order or contract, the manufacturer’s or supplier’s certification shall be furnished to the purchaser stating that samples representing each lot have been manufactured, tested, and inspected in accordance with this specification, and that the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished. When specified in the purchase order or contract, test strip specimens and test records shall accompany peened parts and shall be inspected along with the appropriate lot. The following information shall be recorded for each specimen:

10.1.1 Lot number and other production control numbers.
10.1.2 Part number.
10.1.3 Number of parts in lot.
10.1.4 Date peened.
10.1.5 Shot peening machine used and machine setting.
10.1.6 Specified peening intensity and actual peening intensity by test strip identification numbers if the test fixture requires the use of more than one strip.
10.1.7 Shot size, type, hardness, standoff (distance), length of exposure to shot stream, and shot flow rate.
10.1.8 Percent coverage.
10.1.9 Shot velocity or air pressure.

11. Keywords

11.1 peening; shot; shot peening

### TABLE 6 Fused Ceramic Beads for Peening—Sizes (mm) (in accordance with Fig. 6).^4

<table>
<thead>
<tr>
<th>Designation</th>
<th>Nominal Size, mm</th>
<th>Sieve Number and Screen Opening Size, mm</th>
<th>Min % Beads with Sphericity No. of Beads with Sphericity &lt;0.5 % Acceptable per Area</th>
<th>Max No. of Broken or Angular Beads Acceptable per Area</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ceramic Size</td>
<td>Shot Size</td>
<td>Min</td>
<td>Max</td>
<td>Max 0.5% Retains</td>
</tr>
<tr>
<td>Z 850</td>
<td>330</td>
<td>0.850</td>
<td>1.18</td>
<td>14 (1.400)</td>
</tr>
<tr>
<td>Z 600</td>
<td>230</td>
<td>0.600</td>
<td>0.850</td>
<td>18 (1.000)</td>
</tr>
<tr>
<td>Z 425</td>
<td>170</td>
<td>0.425</td>
<td>0.600</td>
<td>25 (0.710)</td>
</tr>
<tr>
<td>Z 300</td>
<td>110</td>
<td>0.300</td>
<td>0.425</td>
<td>35 (0.335)</td>
</tr>
<tr>
<td>Z 210</td>
<td>70</td>
<td>0.212</td>
<td>0.300</td>
<td>45 (0.250)</td>
</tr>
<tr>
<td>Z 150</td>
<td>GP60</td>
<td>0.150</td>
<td>0.212</td>
<td>60 (0.250)</td>
</tr>
</tbody>
</table>

^4 The designated number for ceramic is the minimum bead diameter (in mm) × 1000 (conversion of mm into in.; divide mm/25.4 = U.S. in.).

### TABLE 7 Shot Maintenance Size and Form Maximum Allowable Nonconforming (in accordance with Fig. 6)

<table>
<thead>
<tr>
<th>Size</th>
<th>Max 2% On Screen (mm)</th>
<th>Min 80% On Screen (mm)</th>
<th>Maximum Allowable Nonconforming Shapes, per area 1 cm × 1 cm</th>
</tr>
</thead>
<tbody>
<tr>
<td>930</td>
<td>6 (3.353)</td>
<td>6 (2.380)</td>
<td>5</td>
</tr>
<tr>
<td>780</td>
<td>7 (2.819)</td>
<td>10 (1.999)</td>
<td>5</td>
</tr>
<tr>
<td>660</td>
<td>8 (2.880)</td>
<td>12 (1.679)</td>
<td>12</td>
</tr>
<tr>
<td>550</td>
<td>10 (1.999)</td>
<td>14 (1.410)</td>
<td>12</td>
</tr>
<tr>
<td>460</td>
<td>12 (1.679)</td>
<td>16 (1.191)</td>
<td>15</td>
</tr>
<tr>
<td>390</td>
<td>14 (1.410)</td>
<td>18 (1.000)</td>
<td>80</td>
</tr>
<tr>
<td>330</td>
<td>16 (1.191)</td>
<td>20 (0.841)</td>
<td>80</td>
</tr>
<tr>
<td>290</td>
<td>18 (1.000)</td>
<td>25 (0.711)</td>
<td>80</td>
</tr>
<tr>
<td>230</td>
<td>20 (0.841)</td>
<td>30 (0.589)</td>
<td>80</td>
</tr>
<tr>
<td>190</td>
<td>25 (0.711)</td>
<td>35 (0.500)</td>
<td>80</td>
</tr>
<tr>
<td>170</td>
<td>30 (0.589)</td>
<td>40 (0.419)</td>
<td>80</td>
</tr>
<tr>
<td>130</td>
<td>35 (0.500)</td>
<td>45 (0.351)</td>
<td>480</td>
</tr>
<tr>
<td>110</td>
<td>40 (0.419)</td>
<td>50 (0.297)</td>
<td>640</td>
</tr>
<tr>
<td>70</td>
<td>45 (0.351)</td>
<td>80 (0.178)</td>
<td>640</td>
</tr>
</tbody>
</table>

### TABLE 8 Intensity Versus Thickness and Ultimate Tensile Strength

<table>
<thead>
<tr>
<th>Material^a</th>
<th>Steel under 1380 MPa</th>
<th>Steel over 1380 MPa and Titanium</th>
<th>Aluminum Alloys (Stainless Steel Shot)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Under 2.5-mm thickness</td>
<td>...</td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>2.5 to 10.0-mm thickness</td>
<td>0.2 to 0.3 mm A^b</td>
<td>0.15 to 0.25 mm A</td>
<td>0.15 to 0.25 mm A</td>
</tr>
<tr>
<td>Over 10.0-mm thickness</td>
<td>0.3 to 0.4 mm A^c</td>
<td>0.15 to 0.25 mm A</td>
<td>0.25 to 0.35 mm A</td>
</tr>
</tbody>
</table>

^a Magnesium alloy’s response to shot peening is different from that of other materials. It is essential to avoid broken or deformed peening material. Peening must be conducted with materials and under conditions that do not induce cracks.

^b The suffix letter A indicates that the values have been determined by the use of Test Strip A.

^c Test Strip A is used for arc heights up to 0.6 mm A. Test Strip C should be used for greater peening intensity. Test Strip N is used if the intensity is below 0.1 mm A.
APPENDIXES
(Nonmandatory Information)

X1. General Information

X1.1 ASTM, Federal, and Military Specifications—Electrodeposits of nickel or chromium and autocatalytic nickel deposits applied in accordance with Specifications B 607, B 650, B 689, and B 733, Guide B 656, and QQ-N-290, QQ-C-320, and MIL-C-26074 to steel products can cause significant reductions in the fatigue strength of the product subjected to cyclical stress loading. Shot peening the steel prior to electroplating helps to control or limit the reduction of fatigue strength that can occur.

X1.2 Reduction of Crack Propagation—Shot peening induces compressive stresses in the surface of the product. Compressive stresses offset high tensile stresses that may be present in electrodeposited metal coatings, thereby impeding the propagation of cracks that cause fatigue failures under cyclical loads.

X1.3 Fatigue Life Improvement—Reductions in fatigue strength are also affected by the hardness and strength of the steel and by the thickness and internal tensile stress of the electrodeposit. Fatigue life may be enhanced by increasing the hardness and strength of the steel and by maintaining the deposit thickness at the minimum value consistent with design requirements. Eliminating or lowering the internal tensile stress of the electrodeposited coating is beneficial. The use of compressively stressed electrodeposited coatings may prevent a significant reduction in fatigue strength.

X1.4 Maintenance of Fatigue Strength—Shot peening, combined with proper selection of the steel and control of thickness and internal tensile stress of the electrodeposit, can be used to minimize or prevent the reduction of fatigue strength in plated steel.

X1.5 Intensity Reduction Indicator—The Almen strip will quickly indicate a reduction in intensity (lower arc height) caused by a reduction in wheel speed or drop in air pressure, by excessive breakdown of shot or other operational faults, such as non-removal of undersize shot.

X1.6 Efficiency and Cost—The smallest shot size capable of producing the desired effect is the most efficient and least costly. An intensity may be considered excessive if, as with very thin parts, it produces a condition in which the tensile stresses of the core material outweigh the beneficial compressive stresses induced at the surface. Table 8 provides a recommended peening intensity relative to cross-sectional thickness and strength of the steel.

X1.7 Test Strip Code—The suffix letter (A, C, or N) indicates that the intensity values have been determined by the use of a test strip of that value. Test Strip A is used for arc heights between 4 (0.1 mm)A and 24 (0.6 mm)A. If greater peening intensity than 24(0.6 mm)A is desired, Test Strip C should be used. Test Strip N is used if the intensity is below 4(0.1 mm)A.

X1.8 Masking Alternatives—When it is impractical to mask or otherwise protect areas designated to be free of shot peening marks, sufficient stock may be provided in these areas for the subsequent removal of affected material for compliance with dimensional requirements of the contract, provided that the temperature of Table 9 is not exceeded. If the beneficial effects of the compressive layer are required, do not remove more than 10% of the total depth of the compressive layer.

X1.9 Saturation Curve—A saturation curve is produced by exposing individual test strips for increasing time periods and plotting the results (exposure time versus arc height). A minimum of four points other than zero shall be used to define the curve; one of the four points used to indicate saturation shall be at least double the time of the saturation point. Saturation is achieved when, as the exposure time for the test strips is doubled, the arc height (does not increase by more than 10% (see Fig. 4). The arc height at saturation for each location must be within the required arc height range for that location. The reuse of test strips is not permitted. The test strip specimens as shown in Fig. 1 shall be attached as shown in Fig. 2, to holders of the form and dimensions also shown in Fig. 2, and mounted on a fixture or article and exposed to the shot stream in a manner that simulates conditions used for the articles. The test strips shall be run for the saturation time established by the saturation curve. After exposure, the test strips shall be removed from the holders and the amount of deflection measured with a micrometer gage, of the form and dimensions shown in Fig. 3. The arc height or amount of deflection measured on the test strips shall be within the specified intensity range. If the arc height measured is not within the intensity range specified, the process parameters must be adjusted, and new saturation curves must be run. In using the micrometer gage, the central portion of the unpeened side of the test strip shall be placed against the indicator stem of the gage. A peened test strip shall not be repainted after being removed from the test strip holder.

X1.10 Automatic Equipment—Automatic shot peening may be accomplished with equipment that propels shot by air pressure or centrifugal force against the product and moves the work through the shot stream in translation, rotation, or both. The equipment shall be capable of consistent reproduction of the shot peening intensities required. The equipment shall include a separator that removes broken or defective shot continuously during peening. The equipment shall be capable of controlling the peening cycle automatically.

X1.11 Computer-Monitored Equipment—Machines
equipped with a mechanical means with programmable speed selection for turning the part on its geometric center-line as closely as possible. The machine shall be equipped with mechanical means with programmable speed selection for translating the nozzle across the surface part (either horizontally or vertically). When run without nozzle translation, the machine shall be capable of programmable shutdown of each nozzle at any time during the peening cycle. The equipment shall have the capability to set and verify the rate of shot flow of each individual nozzle. The equipment shall be computer controlled for processing, monitoring, and documentation of the critical process interrupt parameters, which are air pressure of each nozzle, shot flow of each wheel, shot flow of each wheel, part rotation rate, nozzle reciprocation rate and amount, run time for each part, and total cycle time. This type of equipment is capable of programming maximum and minimum limits for each process interrupt parameter. Every second or less, all process interrupt parameters are scanned and evaluated against the pre-programmed maximum and minimum limits. If any deviation from the pre-programmed limits is found, the machine shall be shut down and the malfunction shall be indicated. The problem shall be corrected before the machine process cycle is resumed. The process is then restarted and completed from the exact point of shutdown. The machine shall be capable of storing in memory the data evaluated for each process interrupt parameter and providing that data in hard copy form, if required. The machine shall be able to document the details of any process interruptions in memory or hard copy form. The machine shall be capable of continuous separation of shot, both by size and shape, so that the shot being used conforms to the requirements of Table 7.

X1.12 Manual or Hand Peening and Rotary Flap Peening—Manual or hand peening and rotary flap peening shall not be permitted, except with the express written permission of the purchaser, since these processes are not as controllable and the results are less predictable than those obtained by automated shot peening.

X2. FREEDOM FROM IRON CONTAMINATION TEST

X2.1 The purpose of this test is to detect contamination by iron residues on the surfaces of aluminum and its alloys, corrosion and heat resisting alloys, etc.

X2.2 Materials:
X2.2.1 Degreasing agent.
X2.2.2 Five percent by volume aqueous solution of hydrochloric acid.
X2.2.3 Ten percent by weight aqueous solution of potassium ferrocyanide.
X2.2.4 Filter paper.

X2.3 Procedure—Degrease the area to be tested by wiping with an appropriate solvent such as isopropyl alcohol. Place a drop of the hydrochloric acid solution on the degreased surface and leave for approximately 2 min. Wet a filter paper with a drop of the potassium ferrocyanide solution and place it on the area of the part wetted with the hydrochloric acid solution. Rinse the area with water.

X2.4 Result—A deep blue color on the filter paper indicates the presence of iron. On some alloys, a pale blue color may be observed in the absence of iron residues. For comparison purposes, it is advisable to prepare a sample that is known to be free of iron contamination.
Standard Specification for Powder Metallurgy (P/M) Boron Stainless Steel Structural Components

1. Scope
1.1 This specification covers stainless steel powder metallurgy (P/M) structural components with a 7.7-g/cm³ minimum density that are fabricated from prealloyed powder consisting primarily of iron, chromium, nickel, molybdenum, and boron and are intended for use in corrosive service.
1.2 The values stated in inch-pound units are to be regarded as the standard. The SI values given in parentheses are for information only.

2. Referenced Documents
2.1 ASTM Standards:
A 262 Practices for Detecting Susceptibility to Intergranular Attack in Austenitic Stainless Steels
B 117 Practice for Operating Salt Spray (Fog) Apparatus
B 243 Terminology of Powder Metallurgy
B 311 Test Method for Density Determination for Powder Metallurgy (P/M) Materials Containing Less than Two Percent Porosity
E 8 Test Methods for Tension Testing of Metallic Materials
E 354 Test Methods for Chemical Analysis of High-Temperature, Electrical, Magnetic, and Other Similar Iron, Nickel, and Cobalt Alloys
E 572 Test Method for X-Ray Emission Spectrometric Analysis of Stainless Steel
E 1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel and in Iron, Nickel, and Cobalt Alloys
E 1086 Test Method for Optical Emission Vacuum Spectrometric Analysis of Stainless Steel by the Point-to-Plane Excitation Technique

G 48 Test Method for Pitting and Crevice Corrosion Resistance of Stainless Steels and Related Alloys by Use of Ferric Chloride Solution

3. Terminology
3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243. Additional descriptive information is in the Related Material section of Volume 02.05 of the Annual Book of ASTM Standards.

4. Ordering Information
4.1 Orders for components under this specification shall include the following information:
4.1.1 Dimensions (see Section 9),
4.1.2 Chemical composition (see Section 6 and Table 1),
4.1.3 Density (see Section 7),
4.1.4 Mechanical properties (see Section 8 and Table 2),
4.1.5 Certification (see Section 13),
4.1.6 Reference to the standard.

5. Materials and Manufacture
5.1 Structural components shall be made by cold pressing and sintering prealloyed powder.
5.2 The sintering temperature is dependent on the chemical composition of the powder.

6. Chemical Composition
6.1 The material shall conform to the composition limits specified in Table 1.
6.2 Chemical analysis should be made in accordance with Test Methods E 354, E 572, E 1019, and E 1086.

7. Physical Properties
7.1 Density:
7.1.1 The sintered density shall be 7.7-g/cm³ minimum.
7.1.2 Density shall be determined in accordance with Test Method B 311.

8. Mechanical Properties
8.1 The purchaser and manufacturer shall agree upon the method to be used to verify the typical yield or tensile strength in the finished parts. The preferred method for verifying the
tensile or yield strength is for the manufacturer and purchaser to agree upon a qualification test to be performed on the actual part. The specific test should be determined after consideration of the function of the part. An example would be measuring the force needed to break teeth off a gear, using a prescribed fixture.

8.2 The tensile properties of the material may also be verified using specifically prepared bars, compacted from the same mixed powder lot as the purchased parts, and sintered along with the parts.

8.3 Typical tensile values for 0.2 % offset yield strength, ultimate strength, and percent elongation in 1-in. gage length for as-sintered standard flat unmachined tension test specimens for powder metallurgy (P/M) products (see Fig. 19 of Test Methods E 8) are in Table 2.

9. Dimensions and Tolerances

9.1 Dimensions and tolerances of the structural components shall be indicated on drawings accompanying the purchase order or contract.

10. Sampling

10.1 Lot—Unless otherwise specified, a lot shall consist of components of the same form and dimensions made from the same mixed powder lot, compacted, and processed under the same conditions, and submitted for inspection at one time.

10.2 Chemical Analysis—If required by purchase agreement, at least one sample for chemical analysis shall be taken from each lot. A representative sample of chips may be obtained by dry-milling, drilling, or crushing at least two pieces without lubrication using clean dry tools.

10.3 Mechanical Testing—If required by the purchase agreement, the manufacturer and purchaser shall mutually agree on the representative number of specimens for mechanical testing from each lot. This number shall be based on good practice of statistical quality control.

11. Inspection

11.1 Unless otherwise specified, inspection of components supplied on contract shall be made by the purchaser.

12. Rejection and Rehearing

12.1 Components that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with test results, the producer or supplier may make a claim for rehearing.

13. Certification

13.1 When specified in the purchase order or contract, a producer’s certification of compliance document shall be reported to the purchaser, verifying that the components manufactured were sampled, tested, and inspected in accordance with this specification and have met the requirements. When specified in the purchase order or contract, a report of test results shall be furnished.

13.2 Certification by an independent third party indicating conformance to the requirements of this specification may be considered upon the request of the purchaser.

13.3 The purchase order or contract shall specify whether or not the certification includes a report of chemical analysis.

14. Keywords

14.1 cold pressing; density; powder metallurgy (P/M); pre-alloyed powder; sintering; stainless steel; structural (P/M) components
SUPPLEMENTARY REQUIREMENTS

The following supplementary requirements shall apply only when specified by the purchaser in the inquiry, contract, or order. The supplier and the purchaser should agree upon a minimum number of hours survival for the requested corrosion test.

S1. Intergranular Corrosion Test
   S1.1 When specified, material shall pass intergranular corrosion tests conducted by the manufacturer in accordance with Practices A 262.

S2. Salt Spray (Fog) Testing
   S2.1 When specified, material shall pass the salt spray corrosion test conducted by the manufacturer in accordance with Practice B 117.

S3. Pitting and Crevice Corrosion Test
   S3.1 When specified, material shall pass pitting and crevice corrosion tests conducted by the manufacturer in accordance with Test Methods G 48.
Standard Test Method for Volumetric Flow Rate of Metal Powders Using Arnold Meter and Hall Funnel

This standard is issued under the fixed designation B 855; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure for measuring the flow characteristics of a given volume of powder.

1.2 The values stated in SI units are to be regarded as the standard (except for the Hall Flowmeter Funnel, which is produced in inch-pound units). The values given in parentheses are for information only.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 213 Test Method for Flow Rate of Metal Powders
B 215 Practices for Sampling Finished Lots of Metal Powders
B 243 Terminology of Powder Metallurgy
B 703 Test Method for Apparent Density of Metal Powders using the Arnold Meter

3. Terminology

3.1 Definitions—Useful definitions of terms for metal powders and powder metallurgy are found in Terminology B 243.

3.2 Definitions of Terms Specific to This Standard:
3.2.1 volumetric flow rate—the time required for a given volume of powder to flow through an orifice in a standard instrument according to a specified procedure.

4. Summary of Test Method

4.1 This test method consists of slowly sliding a bushing partially filled with powder over a 20 cm³ hole in a hardened steel block. The volume of the powder obtained upon removal of the steel block is transferred to a Hall Flowmeter and the flow rate reported in seconds per 20 cm³.

5. Significance and Use

5.1 The volumetric flow rate is a true measure of the flow characteristics of a metal powder. Measuring flow by volume as compared to flow per unit mass eliminates the variable of the powder density.

5.2 The ability of a powder to flow and pack is a function of interparticle friction. As the surface area increases, the amount of friction in a powder mass also increases. Consequently, the friction between particles increases, giving less efficient flow and packing.

5.3 Knowledge of the volumetric flow rate is important to the parts’ producer in estimating production rates—compacts per hour, as well as for blending and mixing of powders.

5.4 This test method may be part of the purchase agreement between powder manufacturers and powder metallurgy (P/M) part producers, or it can be an internal quality control test by either the producer or the end user.

6. Apparatus

6.1 Arnold Density Meter—(Test Method B 703) a hardened tempered and demagnetized steel block having a center hole 31.6640 ± 0.0025 mm (1.2466 ± 0.0001 in.) and a height of 25.4000 ± 0.0025 mm (1.0000 ± 0.0001 in.) that corresponds to a volume of 20 cm³ (1.22 in.³) (Fig. 1).

6.2 Bushing—either brass or bronze. Approximately 38 mm (1.50 in.) inside diameter (ID) by 45 mm (1.75 in.) outside diameter (OD) by 38 mm (1.50 in.) long (Fig. 1).

6.3 Hall Flowmeter—(Test Method B 213) A standard flowmeter funnel having a calibrated orifice of 0.10 in. (2.54 mm) in diameter complete with stand (Fig. 2).

6.4 Base—A level, vibration-free base to support the Hall Flowmeter.

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1 This test method is under the jurisdiction of ASTM Committee B-9 on Metal Powders and Metal Powder Product and is the direct responsibility of Subcommittee B09.02 on Base Metal Powders.

2 Annual Book of ASTM Standards, Vol 02.05.

3 The Arnold Density Meter complete with bushing is available from Arnold P/M Consulting Services, 648 Cedar Road, St. Marys, PA 15857 and also from Alcan Powders & Pigments, 901 Lehigh Avenue, Union, NJ 07083-7632.

4 The complete Hall Flowmeter is available from Alcan Powders & Pigments, 901 Lehigh Avenue, Union NJ 07083-7632.
6.5 Paper—Glazed or waxed paper measuring approximately 150 mm by 150 mm square (6.0 in. by 6.0 in.).

6.6 Timing Device—Stopwatch or other suitable device.

7. Sampling

7.1 Obtain a test sample in accordance with Practice B 215.

7.2 The powder sample shall be of sufficient volume to fill the bushing to three-quarters of its height.

8. Preparation of Apparatus

8.1 Clean test block, bushing, and Hall funnel thoroughly to eliminate any loose powder particles.

9. Procedure

9.1 Place the steel block on a sheet of glazed or waxed paper.

9.2 Place the bushing onto the steel block on either side of the hole.

9.3 Fill the bushing slowly and carefully to three-quarters of its height with powder. (A ring on the ID of the bushing indicates the proper fill height.)

9.4 With downward pressure on the bushing, slowly slide the bushing towards the hole while twisting it. This gives a snowplow action to the powder as it falls slowly into the hole. Continue this motion until the bushing passes the hole. Stop, and again with downward pressure on the bushing slide it straight back over the hole to its starting position. The sliding action must be slow enough to allow for complete filling of the steel block cavity.

9.5 Remove the steel block from the paper, being careful not to tip the block and spill additional powder on to the paper.

9.6 The 20 cm$^3$ of powder is carefully poured from the paper into the Hall funnel while keeping the discharge orifice at the bottom of the funnel closed by placing a dry finger under it.

9.7 Start the timing device simultaneously with removal of the finger from the discharge orifice and stop it the instant the last of the powder leaves the funnel. Record the elapsed time to the nearest second.
10. Report

10.1 Multiply the flow time by the correction factor\(^5\) and report the results as the Arnold/Hall flow rate (s/20 cm\(^3\)).

11. Precision and Bias

11.1 Precision—Precision has been determined from an interlaboratory study performed by Subcommittee B09.02.

11.1.1 Volumetric Flow Rate Using the Arnold Meter and the Hall Funnel:

Repeatability \(r = 1 \text{ s (iron)}, 2 \text{ s (lubricated bronze and brass)}\) \(^1\)

\(^5\) The correction factor is a value supplied by the manufacturer of the funnel using a standardized powder. Periodically, the funnel must be calibrated with the standard powder to compensate for orifice wear. For the calibration procedure refer to Test Method B 213.

\(1\) In 95 % of such tests, on the basis of test error alone, duplicate tests in the same laboratory by the same operator, on one homogeneous lot of powder will differ by no more than the stated amount.

Reproducibility \(r = 2 \text{ s (iron)}, 3 \text{ s (lubricated bronze and brass)}\) \(^2\)

\(^2\) For 95 % of comparative trials done in two different laboratories and on the basis of test error alone, single tests on the same homogeneous lot of powder will differ by no more than the stated amount.

11.2 Bias—No bias statement can be made because there is no accepted standard or reference powder for volumetric flow.

12. Keywords

12.1 Arnold/Hall flow; interparticle friction; metal powders; volumetric flow rate

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Standard Practice for De-Agglomeration of Refractory Metal Powders and Their Compounds Prior to Particle Size Analysis

This standard is issued under the fixed designation B 859; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the de-agglomeration of refractory metal powders and their compounds in preparation for particle size analysis.

1.2 Experience has shown that this practice is satisfactory for the de-agglomeration of elemental tungsten, molybdenum, rhenium, and tantalum metal powders, and tungsten carbide. Other metal powders (for example, elemental metals, carbides, and nitrides) may be prepared for particle size analysis using this practice with caution as to effectiveness until actual satisfactory experience is developed.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific precautionary statements, see Note 2.

2. Referenced Documents

2.1 ASTM Standards:
B 243 Terminology of Powder Metallurgy
B 330 Test Method for Fisher Number of Metal Powders and Related Compounds
B 430 Test Method for Particle Size Distribution of Refractory Metal Powders and Related Compounds by Turbidimetry
B 761 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by X-Ray Monitoring of Gravity Sedimentation
B 821 Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis
B 822 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering

2.2 ASTM Adjunct:
ADJB0859 Detailed Drawings of Alternative Steel Milling Bottles

3. Terminology

3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243.

4. Significance and Use

4.1 Refractory metal powders, such as tungsten and molybdenum, are usually produced by hydrogen reduction at high temperatures. Thus, they usually contain numerous large, strongly-sintered agglomerates. Many of the manufacturing processes using these powders involve a milling step or some similar treatment or depend on the individual particulate size, not on the agglomerate size. Thus, a knowledge of the individual particulate size distribution, not the agglomerate size distribution, is usually desired from a particle size analysis of these powders. This practice provides a procedure for breaking down agglomerates into their constituent particles (de-agglomeration), without excessive fracture of the individual particles. The procedure is often referred to as laboratory milling or rod milling.

4.2 The laboratory milling conditions specified in this guide have been in use since 1965 as part of Test Method B 430. This guide was first published as a separate, stand-alone standard in 1995 because of its applicability in preparing powder samples for analysis by other methods (for example, Test Methods B 761 and B 822), in addition to Test Method B 430. Information on the development and establishment of the milling conditions here specified can be found in the footnoted reference.

4.3 The milling procedure described in this practice does not necessarily break down only agglomerates without fracturing individual particles; some particle fracture may occur in certain powders. However, use of this practice does provide...
consistent particle size analysis results that have been found to relate well to powder behavior in numerous manufacturing processes.

4.4 This practice shall be used for the de-agglomeration of the refractory metal powders and compounds listed in 1.1, when an evaluation of the individual particulate size distribution is required from the subsequent particle size analysis. It shall not be used when the agglomerate (as-is or as-supplied) size distribution is desired.

4.5 This practice may be used in preparing samples for Test Methods B 330, B 430, B 761, B 822, and other particle size analysis methods, prior to the dispersion procedure of Guide B 821, if used.

5. Apparatus

5.1 **Milling Bottle**—There are two alternative materials for the milling container:

5.1.1 **Glass Bottle**—Round laboratory solution bottle, 250-mL capacity, 60-mm diameter, wide-mouth, flat-bottom centrifuge bottle, with cap or stopper, or

5.1.2 **Stainless Steel Bottle**—Fabricated according to the detailed drawings in Adjunct No. ADJB0859.

5.2 **Tungsten Rods**—Fifty rods 75 ± 3 mm long by 4.0 ± 0.3 mm in diameter (approximately 3 in. by 0.15 in.), ground surface.

5.3 **Laboratory Jar Roll Mill,** capable of rotating the milling bottle at 145 rpm.

**Note 1**—If a jar roll mill is not available to give a bottle rpm of 145, the bottle can be either (1) set up on a lathe, or (2) built up in diameter and used on a faster rpm mill.

5.4 **Screen, No. 20 (850-µm), and Bottom Pan.**

6. Procedure

6.1 Place 30 ± 0.1 g of tungsten, molybdenum, rhenium, or tantalum metal, or 50 ± 0.1 g of tungsten carbide powder in the milling bottle containing the 50 tungsten milling rods.

6.2 Seal the milling bottle and rotate on the jar roll mill for 60 min + 15 s at a bottle speed of 145 ± 5 rpm. After the first 5 min, check to be sure the rods are cascading inside the bottle by listening for the fast-paced, repeated “clinking” sound that the cascading rods make. If the rods are not cascading, stop the mill, set the bottle upright momentarily, then replace the bottle on the mill and continue milling for the remaining 55 min (if the rods are now cascading; if not, repeat setting the bottle upright and restarting until they are).

6.3 After milling, immediately screen the powder through a No. 20 (850-µm) screen to remove the milling rods. Dislodge any milled powder that might remain in the bottle by placing a couple of screened rods in the bottle and “whipping” for a few turns.

**Note 2**—**Warning:** The fresh metal surfaces produced during milling may have a tendency to rapidly oxidize when the milling bottle is opened. Use caution when opening the bottle to avoid fire or explosion.

6.4 Remove all the milled powder from the bottom pan and place in a sample container.

7. Particle Size Analysis

7.1 If necessary, disperse the milled powder according to Guide B 821.

7.2 Immediately perform the desired particle size analysis.

**Note 3**—Since milled powder has a greater tendency than as-supplied powder to pick up moisture and oxidize, the analysis procedure should be initiated immediately after milling is completed. For all practical purposes, however, two runs can be made in succession on each milled powder. If more than two runs on the same milled powder are desired, provisions may be taken to lessen (elimination is not possible) the effect of humidity on the milled powder, such as immediate splitting of the sample and storage under dry nitrogen or in a desiccator.

8. Keywords

8.1 de-agglomeration; laboratory-milled; laboratory milling; molybdenum; particle size analysis; powders; refractory metals; rhenium; rod-milled; rod milling; tantalum; tungsten; tungsten carbide

**SUMMARY OF CHANGES**

Committee B09 has identified the location of selected changes made to this standard since the last issue, B 859–95 (2000), that may impact the use of this standard. (Approved Oct. 1, 2003.)

(1) Stainless steel milling bottles were added as an alternative to the glass bottles in Sections 5.1 and 5.1.2.

(2) The adjunct number for the drawings of the stainless steel milling bottles was added in Section 2.2.
Standard Test Method for Gross Defects and Mechanical Damage in Metallic Coatings by Polysulfide Immersion

1

This standard is issued under the fixed designation B 866; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers equipment and methods for detecting gross defects and mechanical damage (including wear-through) in metallic coatings where the breaks in the coating penetrate down to a copper or copper alloy substrate.

1.2 This test method is suitable for coatings consisting of single or combined layers of any coating that does not significantly tarnish in an alkaline polysulfide solution. Examples are gold, nickel, tin, tin-lead, and palladium, or their alloys.

1.3 Recent reviews of porosity testing (which include those for gross defects) and testing methods can be found in literature. An ASTM guide to the selection of porosity and gross defect tests for electrodeposits and related metallic coatings is available as Guide B 765. Other related porosity test standards are Test Methods B 735, B 741, B 798, B 799, and B 809.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 246 Specification for Tinned Hard-Drawn and Medium-Hard-Drawn Copper Wire for Electrical Purposes

B 374 Terminology Relating to Electroplating

B 488 Specification for Electrodeposited Coatings of Gold for Engineering Uses

B 542 Terminology Relating to Electrical Contacts and Their Use

B 545 Specification for Electrodeposited Coatings of Tin

B 605 Specification for Electrodeposited Coatings of Tin-Nickel Alloy

B 679 Specification for Electrodeposited Coatings of Palladium for Engineering Use

B 689 Specification for Electrodeposited Engineering Nickel Coatings

B 733 Specification for Autocatalytic (Electroless) Nickel-Phosphorus Coatings on Metal

B 735 Test Method for Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor

B 741 Test Method for Porosity in Gold Coatings on Metal Substrates by Paper Electrography

B 765 Guide for Selection of Porosity Tests for Electrodeposits and Related Metallic Coatings

B 798 Test Method for Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography

B 799 Test Method for Porosity in Gold and Palladium Coatings by Sulfurous Acid/Sulfur Dioxide Vapor

B 809 Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor (“Flowers-of-Sulfur”)

3. Terminology

3.1 Definitions: Many terms used in this test method are defined in Terminologies B 374 or B 542.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 defect indications—black or dark colored products resulting from the reaction between the alkaline polysulfide reagent and exposed copper or copper alloy underlying metal.

3.2.2 gross defects—breaks in the coating that expose relatively large areas of underlying metal to the environment (compare with intrinsic porosity (3.2.3)). Gross defects include those produced by mechanical damage and wear, in addition to as-plated large pores (with diameters an order of magnitude greater than intrinsic porosity) and networks of microcracks.

Note 1—Such large pores and microcrack networks indicate serious deviations from acceptable coating practice (as, for example, dirty
basis-metal substrates and contaminated or out-of-balance plating baths).

3.2.3 intrinsic porosity—the “normal” porosity that is present, to some degree, in all commercial thin platings (such as in precious-metal coatings for engineering purposes) and will generally follow an inverse relationship with thickness.

NOTE 2—Intrinsic porosity is due primarily to small deviations from ideal plating and surface preparation conditions. Scanning electron microscope (SEM) studies have shown that the diameter of such pores, at the plating surface, is of the order of micrometres, so that only small areas of underlying metal are exposed to the environment.

3.2.4 measurement area—the portion or portions of the surface examined for the presence of gross defects or mechanical damage (and wear-through). The measurement area shall be indicated on the drawings of the parts, or by the provision of suitably marked samples.

3.2.5 metallic coatings—platings, claddings, or other metallic coatings applied to the basis-metal substrate. The coating can comprise a single metallic layer or a combination of metallic layers.

3.2.6 porosity (general)—in a coating, the presence of any hole, crack, or other defect that exposes the underlying metal to the environment.

3.2.7 underplate—a metallic coating layer between the basis metal and the topmost metallic coating. The thickness of an underplating is usually greater than 1 µm, in contrast to a strike or flash, which is usually thinner.

3.2.8 wear-through—the exposure of underplate or basis metal as a direct result of wear. Wear-through is an observable phenomenon.

3.2.9 wear track—a mark that indicates the path along which physical contact had been made during a sliding process (such as the mating and unmatting of an electrical contact).

4. Summary of Test Method

4.1 The test samples are immersed in an alkaline polysulfide solution at 74°C (165°F) for 60 s. After rinsing and drying, the samples are examined for dark or discolored areas which indicate exposure of copper or copper alloys to the solution through breaks in the coating.

5. Significance and Use

5.1 The purpose of the alkaline polysulfide immersion test is to determine the presence of mechanical damage, wear-through, and other gross defects in the coating. Most metallic coatings are intended to be protective and the presence of gross defects indicates a serious reduction of such protection.

5.2 The protection afforded by well applied coatings may be diminished by improper handling following plating or as a result of wear or mechanical damage during testing or while in service. The alkaline polysulfide test serves to indicate if the damage has extended down to the copper or copper alloy basis metal since it will not detect exposed nickel underplate.

5.3 The alkaline polysulfide test has been specified in several ASTM specifications for tin-plated coatings, namely Specifications B 246 and B 545. This test could also be used to detect gross defects and mechanical damage in other metallic coatings, such as tin-nickel alloy (Specification B 605), nickel (Specification B 689), gold (Specification B 488), palladium (Specification B 679), and autocatalytic nickel-phosphorous coatings (Specification B 733).

5.4 This test detects mechanical damage that exposes copper underplate and copper basis metal. Such damage may occur in any post-plating operation or even towards the end of the plating operation. It is most often seen to occur in product assembly operations.

5.5 If properly performed, this test will also detect wear-through, provided the wear-through reaches a copper or copper-alloy layer.

5.6 Many types of gross defects are too small to be seen, except at magnifications so high (as in SEM) that a realistic assessment of the measurement area cannot be easily made. Other defects, such as many types of wear-through, provide insufficient contrast with the coating surface. Gross defects tests (as with porosity tests) are, therefore, used to magnify the defect sites by producing visible reaction products in and around the defects.

5.7 The polysulfide solution will react with copper and copper alloys to produce a dark brown or black stain (the defect indications) at the site of the defect. Silver also turns black under the same conditions. The test solution will not react with nickel and is only useful when the presence or absence of copper exposure is a specific requirement.

5.8 The polysulfide immersion test is relatively insensitive to the presence of small pores. It shall not be used as a general porosity test. (Test Method B 809 should be used instead.)

5.9 The extent and location of the gross defects or mechanical damage (revealed by this test) may or may not be detrimental to product performance or service life. Such determinations shall be made by the user of the test through practical experience or judgment.

5.10 The present test can be used on samples of various geometries, such as curved surfaces. It can also be used for selective area coating if allowance is made for tarnish creepage from bare copper alloy areas.

5.11 This test is destructive in that it reveals the presence of gross defects by contaminating the surface with reaction-product films. Any parts exposed to this test shall not be placed in service.

5.12 However, the defect indications on the sample surfaces that result from this test are stable; samples may be retained for reference purposes.

5.13 This test is neither recommended for predictions of product performance nor is it intended to simulate field failure mechanisms. For such product performance evaluations, an environmental test that is known to simulate actual failure mechanisms should be used.

6. Apparatus

6.1 In addition to the normal equipment (beakers, bottles, weighing balances, funnels, and so forth) that are part of every chemical laboratory, the following apparatus are required:

6.1.1 Microscope—Optical, stereo, 10 to 30×. It is preferred that one eyepiece contain a graduated reticle for measuring the diameter of tarnish spots. The reticle shall be calibrated for the magnification at which the microscope is to be used, preferably 10×.
6.1.2 Hydrometer, 1.120 to 1.190 specific gravity, 150–mm scale.
6.1.3 Light Source (Illuminator) for Microscope, incandescent, or circular fluorescent.

7. Reagents
7.1 Sodium Hydroxide, pellet, ACS certified grade or better.
7.2 Sodium Sulfide, 9-hydrate, ACS "Analytical Reagent" (AR) grade, or better.
7.3 Sulfur, precipitated, USP grade.

8. Hazards
8.1 All of the normal precautions shall be observed in handling the materials required for this test. This shall also include, but not be limited to, procuring and reviewing Material Safety Data Sheets that meet the minimum requirements of the OSHA Hazard Communication Standard for all chemicals used in cleaning and testing, and observing the recommendations given.

9. Preparation
9.1 Preparation of Solutions:
9.1.1 Polysulfide Solution—Warning—All work shall be done under an operating fume hood since the gases emitted and the polysulfide solution are toxic.
9.1.1.1 Make a saturated solution of sodium sulfide by dissolving 20 to 25 g of sodium sulfide in 100 mL of deionized water. Stir for 30 min at minimum. Make sure that undissolved crystals are present in the solution. If not present, continue adding increments of approximately 0.5–g sodium sulfide, with stirring, until the solution is saturated (excess solids present).
9.1.1.2 With stirring, slowly add 30 to 35 g of sulfur to the saturated sodium sulfide solution.
9.1.1.3 Cover the beaker. Stir for 60 min at minimum.
9.1.1.4 Allow solution to stand for 24 h without stirring.
9.1.1.5 Filter solution through qualitative grade filter paper into a 250-mL beaker.
9.1.1.6 Set aside about 10 mL of filtered solution in a small stoppered vial. Label the vial, “Concentrated Polysulfide Solution,” and date it.
9.1.1.7 Pour remaining solution into a 250–mL graduated cylinder or hydrometer cylinder. Adjust the specific gravity using a hydrometer to 1.142 ± 0.005, at 20 to 30°C, by adding a few millilitres of deionized water and stirring with a glass rod to mix thoroughly. Recheck specific gravity. Continue adding water and mixing until desired specific gravity is reached. If solution becomes too dilute (less than 1.142), add the concentrated polysulfide solution (see 9.1.1.6) as needed.
9.1.1.8 Store solution in a tightly capped 250-mL plastic bottle labeled, “Polysulfide Solution, sp gr 1.142,” and date it.
9.1.2 Alkaline Polysulfide Reagent:
9.1.2.1 Measure 75 mL of the polysulfide solution, sp gr 1.142 into a 600-mL beaker containing a teflon-coated stirring bar.
9.1.2.2 Weigh out 75 g of sodium hydroxide pellets into a plastic weighing dish.
9.1.2.3 Add the sodium hydroxide carefully to the polysulfide solution. Cover beaker. Stir to dissolve.
9.1.2.4 Add 375 mL of deionized water to the beaker, cover, and stir to mix.
9.1.2.5 Store solution in a tightly stoppered 500-mL plastic bottle labeled, “Alkaline Polysulfide Reagent,” and date it.
9.2 Preparation of Test Samples:
9.2.1 Handle samples as little as possible, even before cleaning, and only with tweezers, microscope-lens tissue, or clean soft cotton gloves.
9.2.2 Before being cleaned, the samples shall be prepared so that the measurement areas may be viewed easily through the microscope. If samples are part of assembled products, they may need to be disassembled to ensure proper access to these areas and to enable the part to be immersed in the alkaline polysulfide solution.

Note 3—Since the test is specific to the plated metallic portions of the product, the latter should be separated from plastic housings, etc., whenever possible, before cleaning. Also, nonmetallic materials, such as paper tags, string, tape, and so forth, shall be removed, but take care to maintain sample identity.

9.2.3 Cleaning:
9.2.3.1 Inspect the samples under 10× magnification for evidence of particulate matter. If present, such particles should be removed by “dusting” (that is, blowing them off the sample) with clean, oil-free air.
9.2.3.2 Thoroughly clean the particle-free samples with solvents or solutions that do not contain CFCs, chlorinated hydrocarbons, or other known ozone-destroying compounds. The procedure outlined in Note 4 has been found to give satisfactory results for coatings with mild to moderate surface contamination.

Note 4—Suggested Cleaning Procedure:
(1) Keep individual pieces separated if there is a possibility of damage to the measurement areas during the various cleaning steps.
(2) Clean samples for 5 min in an ultrasonic cleaner which contains a hot (65 to 85°C) 2% aqueous solution of a mildly alkaline (pH 7.5 to 10) detergent (such as Micro or Sparkleen).
(3) After ultrasonic cleaning, rinse samples thoroughly under warm running tap water for at least 5 s.
(4) Rinse samples ultrasonically for 2 min in fresh deionized water to remove the last detergent residues.
(5) Immerse samples in fresh analytical reagent-grade methanol or isopropanol and ultrasonically “agitator” for at least 30 s to remove the water from the samples.
(6) Remove and dry samples until the alcohol has completely evaporated. If an air blast is used as an aid to drying, the air shall be oil free, clean, and dry.
(7) Do not touch measurement area of the samples with bare fingers after cleaning.
9.2.3.3 Reinspect samples (under 10× magnification) for particulate matter on the surface. If particulates are found, repeat the cleaning step. Surface cleanliness is extremely important. Contaminants, such as plating salts and flakes of metal, may give erroneous indications of defects.

10. Procedure
10.1 Warning—All work shall be performed in a fume hood since the vapors are noxious and toxic.
10.2 Before each test, check the effectiveness of the alkaline polysulfide reagent (9.1.2.5) by dipping a clean copper or copper alloy (>95% copper) wire or piece into the reagent at
room temperature. If the copper does not blacken within 10 s, discard solution and make fresh alkaline polysulfide reagent.

NOTE 5—If bare copper pieces are not available, scratch a plated sample heavily through to the copper and test the reagent.

10.3 Place sufficient alkaline polysulfide reagent in a beaker so that the measurement area may be immersed. Cover beaker with a watch glass to prevent evaporation. Heat the solution, with gentle stirring, to 74 ± 3°C.

10.4 Immerse the examination area by any convenient method in the hot reagent for 60 s. Agitate samples if stirring is insufficient to remove air bubbles.

10.5 Remove and gently rinse with warm running tap water and thoroughly dry with clean dry air.

10.6 Cool the reagent. Filter it back into the bottle for reuse. The reagent may be reused as long as it passes the copper wire test described in 10.2.

11. Examination and Evaluation

11.1 Using an incandescent or ring fluorescent lamp, examine the measurement areas at 10× magnification for the presence of dark brown or black stains or spots, which will usually not protrude from the surface. Because burnished gold can appear black under certain lighting conditions, be certain to rotate the sample to ensure that the black is not a result of the light being reflected out of the field of view.

NOTE 6—One limitation of this test is that copper exposed in a deep recess, such as a crack or gouge, will not be detected unless the copper itself is easily visible under low magnification.

11.2 If the defect indications are to be counted, the following additional instructions are recommended.

11.2.1 A defect indication should be measured and counted when at least one half of the indication falls within the measurement area. Unless otherwise specified, products that initiate outside the measurement area but fall within it and are irregular in shape should not be counted. However, for small measurement areas or where the migrating indication covers a significant portion of this area, the presence of such products should be recorded.

11.2.2 Indication size shall be defined by the longest diameter of the product. Unless otherwise specified, products less than 0.05 mm (0.002 in.) in diameter shall not be counted. A graduated reticle in the microscope eyepiece is useful as an aid to counting and sizing.

NOTE 7—A useful sizing technique is to tabulate the defect indications in accordance with three size ranges. These are (approximately): (a) 0.12 mm diameter (0.005 in.) or less, (b) between 0.12 and 0.40 mm diameter (0.005 and 0.015 in.), and (c) greater than 0.40 mm diameter (0.015 in.).

11.2.3 The acceptable number, sizes, and locations of the defect indications shall be as specified on the appropriate drawing or specification, or as agreed upon by the producer and the user.

12. Precision and Bias

12.1 Precision—The precision of this test method will be investigated with samples containing deliberately damaged areas.

12.2 Bias—The procedure in this test method has no bias because the presence and size of the gross defects are defined only in terms of this test method.

13. Keywords

13.1 electrodeposits; gross defects; mechanical damage; metallic coatings; polysulfide immersion; porosity testing; wear-through
Standard Specification for Electrodeposited Coatings of Palladium-Nickel for Engineering Use

1. Scope

1.1 Composition—This specification covers requirements for electrodeposited palladium-nickel coatings containing between 70 and 95 mass % of palladium metal. Composite coatings consisting of palladium-nickel and a thin gold overplate for applications involving electrical contacts are also covered.

1.2 Properties—Palladium is the lightest and least noble of the platinum group metals. Palladium-nickel is a solid solution alloy of palladium and nickel. Electroplated palladium-nickel alloys have a density between 10 and 11.5, which is substantially less than electroplated gold (17.0 to 19.3) and comparable to electroplated pure palladium (10.5 to 11.8). This yields a greater volume or thickness of coating per unit mass and, consequently, some saving of metal weight. The hardness range of electrodeposited palladium-nickel compares favorably with electroplated noble metals and their alloys (1, 2).

NOTE 1—Electroplated deposits generally have a lower density than their wrought metal counterparts.

Approximate Hardness (HK_{25})

<table>
<thead>
<tr>
<th>Material</th>
<th>Hardness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gold</td>
<td>50–250</td>
</tr>
<tr>
<td>Palladium</td>
<td>75–600</td>
</tr>
<tr>
<td>Platinum</td>
<td>150–550</td>
</tr>
<tr>
<td>Palladium-Nickel</td>
<td>300–650</td>
</tr>
<tr>
<td>Rhodium</td>
<td>750–1100</td>
</tr>
<tr>
<td>Ruthenium</td>
<td>600–1300</td>
</tr>
</tbody>
</table>

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

- B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
- B 242 Practice for Preparation of High-Carbon Steel for Electroplating
- B 254 Practice for Preparation of and Electroplating on Stainless Steel
- B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings
- B 322 Practice for Cleaning Metals Prior to Electroplating
- B 343 Practice for Preparation of Nickel for Electroplating with Nickel
- B 374 Terminology Relating to Electroplating
- B 481 Practice for Preparation of Titanium and Titanium Alloys for Electroplating
- B 482 Practice for Preparation of Tungsten and Tungsten Alloys for Electroplating
- B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section
- B 488 Standard Specification for Electrodeposited Coatings of Gold for Engineering Uses
- B 489 Practice for Bend Test for Ductility of Electrodeposited and Autocatalytically Deposited Metal Coatings on Metals
- B 507 Practice for Design of Articles to Be Electroplated on Racks
- B 542 Terminology Relating to Electrical Contacts and Their Use
- B 558 Practice for Preparation of Nickel Alloys for Electroplating
- B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
- B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings
- B 578 Test Method for Microhardness of Electroplated Coatings
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
- B 689 Specification for Electroplated Engineering Nickel Coatings

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1 This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is under the direct responsibility of Subcommittee B08.08 on Engineering Coatings.


2 The boldface numbers in parentheses refer to the list of references at the end of this specification.

Annual Book of ASTM Standards, Vol 02.05.

3. Terminology

3.1 Definitions: Many terms used in this specification are defined in Terminology B 374 or B 542.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 overplating, n—a coating applied onto the topmost palladium-nickel coating. The thickness of an overplating or “flash” is usually less than 0.25 µm.

3.2.2 significant surfaces, n—those surfaces normally visible (directly or by reflection) or which are essential to the serviceability or function of the article; or which can be the source of corrosion products or tarnish films that interfere with serviceability or function of the article; or which can be the source of residual salts or that may adversely affect the appearance and performance of the deposit, despite the absence of residual salts. See Appendix X6.

3.2.3 underplating, n—a metallic coating layer or layers between the basis metal or substrate and the palladium-nickel coating. The thickness of an underplating is usually greater than 1 µm, in contrast to a strike which is thinner.

4. Classification

4.1 Orders for articles to be plated in accordance with this specification shall specify the coating system, indicating the basis metal, the thicknesses of the underplatings, the type and thickness class of the palladium-nickel coating, and the grade of the gold overplating according to Table 1, Table 2, and Table 3. See Section 7.

5. Ordering Information

5.1 In order to make the application of this specification complete, the purchaser shall supply the following information to the seller in the purchase order or other governing document:

5.1.1 The name, designation, and date of issue of this specification;

5.1.2 The coating system including basis metal, composition type, thickness class and gold overplate grade (see 4.1 and Table 1, Table 2, and Table 3);

5.1.3 Presence, composition, and thickness of underplating (see 3.2.1). For nickel underplating see 6.5.1; for copper underplating see 6.5.2; for silver underplating see 6.5.3; for gold underplating see 6.5.4; for palladium underplating see 6.5.5; and for an underplating consisting of any combination of the above see 6.5.6.

5.1.4 Significant surfaces shall be defined (see 3.2.3);

5.1.5 Requirements, if any, for porosity testing (see 9.6);

5.1.6 (Steel parts only) Stress relief if required (see Specification B 489).

5.1.7 (Steel parts only) Hydrogen embrittlement relief (see B 850 );

5.1.8 Sampling plan employed (see Section 8); and,

5.1.9 Requirement, if any, for surface coating cleanliness (absence of residual salts). See Appendix X6.

6. Manufacture

6.1 Any process that provides an electrodeposition capable of meeting the specified requirements will be acceptable.

6.2 Substrate:

6.2.1 The surface condition of the basis metal should be specified and should meet this specification prior to the plating of the parts.

6.2.2 Defects in the surface of the basis metal, such as scratches, porosity, pits, inclusions, roll and die marks, laps, cracks, burrs, cold shuts, and roughness may adversely affect the appearance and performance of the deposit, despite the presence of underplating.
observance of the best plating practice. Any such defects on significant surfaces should be brought to the attention of the supplier and the purchaser.

6.2.3 Clean the basis metal as necessary to ensure a satisfactory surface for subsequent electroplating in accordance with Practices B 183, B 242, B 254, B 281, B 322, B 343, B 481, B 482 and B 558.

6.2.4 Proper preparatory procedures and thorough cleaning of the basis metal are essential for satisfactory adhesion and performance of these coatings. The surface must be chemically clean and continuously conductive, that is, without inclusions or other contaminants. The coatings must be smooth and as free of scratches, gouges, nicks, and similar imperfections as possible.

NOTE 2—A metal finisher can often remove defects through special treatments such as grinding, polishing, abrasive blasting, chemical treatments, and electropolishing. However, these may not be normal in the treatment steps preceding the plating, and a special agreement is indicated.

6.3 If required (see 5.1.6), steel parts with a hardness greater than 1000 MPa (31 HRC) shall be given a suitable stress relief heat treatment prior to plating in accordance with Specification B 849. Such stress relief shall not reduce the hardness to a value below the specified minimum. Avoid acid pickling of high strength steels.

6.3.1 Apply the coating after all basis metal preparatory heat treatments and mechanical operations on significant surfaces have been completed.

6.4 Racking:
6.4.1 Position parts to allow free circulation of solution over all surfaces. The location of rack or wire marks in the coating should be agreed upon between the producer and supplier.

6.5 Plating Process:
6.5.1 Nickel Underplating—Apply a nickel underplating before the palladium-nickel when the product is made from copper or copper alloy. Nickel underplatings are also applied for other reasons. See Appendix X5.

NOTE 3—In certain instances where high frequency analog signals are employed, such as wave guides, the magnetic properties of nickel may attenuate the signal. Palladium-nickel itself is non-ferromagnetic when the nickel content is less than 14 mass %. 

NOTE 4—In applications where forming or flaring operations are to be applied to the plated component, a ductile nickel electrodeposit should be specified.

6.5.2 Strikes—Good practice suggests the use of a palladium strike to follow any underplate or substrate (other than silver or platinum) immediately prior to applying the palladium-nickel.

6.5.3 Plating—Good practice calls for the work to be electrically connected when entering the palladium-nickel solution.

NOTE 5—Some palladium-nickel electroplating solutions attack copper. This can result in codeposition of copper impurity. The situation is further aggravated when low current densities are utilized. Copper can be removed from solutions by low current density electrolysis (0.1 to 0.3 mA/cm^2).

6.5.4 Gold Overplating—Apply a thin gold overplating after the palladium-nickel in any application in which palladium-nickel plated electrical connectors are mated together in a contact pair. This process is necessary to preserve the performance of the contact surface. See Appendix X1 for other reasons for using a gold overplate.

NOTE 6—When using Type 1 gold, the thickness of the gold overplate shall not exceed 0.12 µm (5 µin.) due to increased risk of degrading durability and increasing the coefficient of friction.

6.5.5 Residual Salts—For rack and barrel plating applications, residual plating salts can be removed from the articles by a clean, hot (50 to 100°C) water rinse. A minimum rinse time of 2.5 min (racks) or 5 min (barrel) is suggested. Best practice calls for a minimum of three dragout rinses and one running rinse with dwell times of 40 s in each station when rack plating and 80 s when barrel plating. Modern high-velocity impingement type rinses can reduce this time to a few seconds. This is particularly useful in automatic reel-to-reel applications where dwell times are significantly reduced. See Appendix X6.

7. Coating Requirements
7.1 Nature of Coating—The palladium-nickel deposit shall have a minimum purity of 70 mass % palladium.

7.2 Composition—The composition of the palladium-nickel electrodeposits shall be within ±5 mass % of the specified type.

7.3 Appearance—Palladium-nickel coatings shall be coherent, continuous, and have a uniform appearance to the extent that the nature of the basis metal and good commercial practices permit.

7.4 Thickness—Everywhere on the significant surface (see 5.1), the thickness of the palladium-nickel coating shall be equal to or exceed the specified thickness. The maximum thickness, however, shall not exceed the drawing tolerance.

NOTE 7—The coating thickness requirement of this specification is a minimum requirement, that is, the coating thickness is required to equal or exceed the specified thickness everywhere on the significant surfaces while conforming to all maximum thickness tolerances given in the engineering drawing. Variation in the coating thickness from point to point on a coated article is an inherent characteristic of electroplating processes. The coating thickness at any single point on the significant surface, therefore, will sometimes have to exceed the specified value in order to ensure that the thickness equals or exceeds the specified value at all points. Hence, most average coating thicknesses will be greater than the specified value. How much greater is largely determined by the shape of the article (see Practice B 507) and the characteristics of the plating process. In addition, the average coating thickness on products will vary from article to article within a production lot. If all of the articles in a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the average necessary to assure that a single article meets the requirement. See 8.1.

7.5 Adhesion—The palladium-nickel coatings shall be adherent to the substrate or underplate when tested by one of the procedures summarized in 9.5.

7.6 Integrity of the Coating:
7.6.1 Gross Defects/Mechanical Damage—The coatings shall be free of visible mechanical damage and similar gross defects when viewed at magnifications up to 10X. For some applications this requirement may be relaxed to allow for a small number of such defects (per unit area), especially if they are outside of or on the periphery of the significant surfaces. See 7.6.2.

7.6.2 Porosity—Almost all as-plated electrodeposits contain some porosity, and the amount of porosity to be expected for
any one type of coating will increase with decreasing the thickness of that particular coating type. The amount of porosity in the coating that may be tolerable depends on the severity of the environment that the article is likely to encounter during service or storage. If the pores are few in number, or away from the significant surfaces, their presence can often be tolerated. Acceptance or pass-fail criteria, if required, shall be part of the product specification for the particular article or coating requiring the porosity test. See 9.6.

NOTE 8—Extensive reviews of porosity and porosity testing can be found in the literature (3, 4).

8. Sampling

8.1 The purchaser and producer are urged to employ statistical process control in the coating process. Properly performed, statistical process control will assure coated products of satisfactory quality and will reduce the amount of acceptance inspection. The sampling plan used for the inspection of the quality of the coated articles shall be as agreed upon between the purchaser and the supplier.

8.1.1 When a collection of coated articles (the inspection lot (see 8.2)) is examined for compliance with the requirements placed on the articles, a relatively small number of the articles (the sample) is selected at random and is inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for the sampling inspection of coatings.

8.1.2 Test Method B 602 contains four sampling plans, three for use with tests that are non-destructive and one when they are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 identifies the plan to be used.

8.1.3 Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and seller need to agree on the plan to be used.

8.1.4 Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numerical value and certain statistical requirements must be met. Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Method B 762 identifies the plan to be used.

8.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specifications, coated by a single supplier at one time, or at approximately the same time, under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

9. Test Methods

9.1 Appearance—The coating shall be examined at up to 10× magnification for conformance to the requirements of appearance.

9.2 Alloy Composition—Alloy composition of the palladium-nickel can be determined by a wet method, X-ray Fluorescence (XRF), Scanning Electron Microscopy (SEM)/Energy Dispersive Spectroscopy (EDS), Auger, or by Electron Probe X-ray Microanalysis (EPMA)/Wavelength Dispersive Spectroscopy (WDS).

9.2.1 The method chosen for determination of alloy composition shall not be the same method used for determination of deposit thickness if the deposit is over a nickel underplate or as a referee method. The reason for this is that the determination of alloy composition and the determination of deposit thickness by spectrographic analysis are to some extent interdependent. See 9.2.4.1 and 9.4.1.

9.2.2 Wet Method—Use any recognized method to determine quantitatively the relative concentrations of palladium and nickel present. Atomic absorption spectrophotometry (or any other methods with demonstrated uncertainty less than 10 %) may be used to determine the alloy composition.

NOTE 9—Determination of alloy composition by dissolving the coating from a test specimen must be obtained by electroplating the palladium-nickel directly over a non-nickel containing alloy substrate with no intermediate layer. Copper alloy substrates are preferred. Alloy composition is best determined on a special test specimen. One must be careful to arrange the specimen so as to electroplate at a typical current density, similar to what is used in production. Palladium-nickel may be stripped by utilizing a 90 volume % (reagent grade) sulfuric acid, 10 volume % (reagent grade) nitric acid solution.

9.2.3 XRF—XRF can be used for composition analysis of palladium-nickel alloy coatings deposited directly onto copper or a copper alloy that does not contain nickel. This method is not suitable for composition analysis of palladium-nickel alloy coatings less than 60 µm in thickness when deposited over nickel or nickel containing substrates.

NOTE 10—If the palladium-nickel coating is less than 60 µm, palladium-nickel alloy composition measurements in the presence of an intermediate nickel layer or nickel containing substrate is degraded by the fact that the nickel X-ray emission of the alloy layer and the intermediate layer (or substrate) cannot be accurately distinguished from one another.

9.2.4 EPMA:

9.2.4.1 EPMA based on electron beam excitation of X-rays characteristic of the elements present can be used to measure composition of palladium-nickel alloy coatings on top of any undercoat or any substrate to an accuracy of 0.1 mass % palladium if the thickness of the coating is ≥1.5 µm. See Appendix X8.

9.2.4.2 EPMA shall be used as the referee method for the determination of alloy composition.

9.2.5 SEM/EDS—the SEM/EDS technique is capable of determining composition of palladium-nickel coatings that are ≥1.5 µm thick to an accuracy and precision of ±0.2 mass % palladium. A procedure for calibration of a conventional SEM equipped with an X-ray EDS for routine analysis of palladium-nickel alloy composition appears in Appendix X7.

9.2.6 Auger Electron Spectroscopy (AES) and X-ray Photo-electron Spectroscopy (XPS)—AES and XPS are capable of analyzing regions that are of the order of 0.002 µm thick. These techniques are potential candidates for analysis of electrodeposited palladium-nickel alloy coatings with a thickness of ≥0.03 µm.
NOTE 11—The use of AES and XPS to determine bulk coating composition requires the sputter removal of 0.01 to 0.02 μm of material from the surface to remove surface contaminants and surface composition gradients. Some, but not all, commercial AES and XPS instruments are capable of accurate analyses of palladium-nickel alloy composition using an internal procedure for determining correction factors similar to that described in Appendix X7 for an SEM/EDS instrument.

9.3 Deposit Purity—Use any recognized method to determine quantitatively the metallic impurities present. Atomic absorption spectrophotometry (or any other methods with demonstrated uncertainty less than 10 %) may be used to determine the metallic impurities. Initial scanning should be carried out for all elements, in order to detect any unknown or unexpected impurities. Determine deposit purity by subtracting total impurities from 100 %.

NOTE 12—Deposit purity is best determined on a special test specimen. One shall be careful to arrange the specimen so as to electroplate at a typical current density, similar to what is used in production. Palladium-nickel may be stripped by utilizing a 90 volume % (reagent grade) sulfuric acid, 10 volume % (reagent grade) nitric acid solution. The test specimen substrate should be platinum, gold, or an electrodeposited not attacked by the strip solution. For the determination of impurities, the total palladium-nickel deposit should be over 100 mg and the sample weight is determined by a weigh-strip-weigh procedure. The strip solution is then used for quantitative analysis of impurities.

9.4 Thickness:

9.4.1 Measure thickness by methods outlined in Test Methods B 487, B 568, or B 748, or any other test method that has an uncertainty less than 10 %. See Appendix X2 for specific information on thickness measurement of palladium-nickel alloy coatings by XRF.

9.4.2 Use Test Method B 748 as the referee method for the determination of deposit thickness.

9.5 Adhesion—Determine adhesion by one of the following procedures (see Test Methods B 571 for full details):

9.5.1 Bend Test—Bend the electroplated article repeatedly through an angle of 180° on a diameter equal to the thickness of the article until fracture of the basis metal occurs. Examine the fracture at a magnification of 10×. Cracking without separation does not indicate poor adhesion unless the coating can be peeled back with a sharp instrument.

9.5.2 Heat Test—No flaking, blistering, or peeling shall be apparent at a magnification of 10× after the palladium-nickel electroplated parts are heated to 300 to 350°C (570 to 660°F) for 30 min and allowed to air cool.

9.5.3 Cutting Test—Make a cut with a sharp instrument and then probe with a sharp point and examine at a magnification of 10×. No separation of the coating from the substrate or intermediate layers shall occur.

9.6 Plating Integrity—Porosity and microcracks shall be determined by either Test Methods B 741, B 798, B 799, or B 809 unless otherwise specified. Do not use the nitric acid vapor test (palladium-nickel can dissolve in nitric acid).

NOTE 13—The test to be selected will depend on the palladium-nickel thickness, the nature of the basis metal, the nature and thickness of any intermediate layers or underplate, and the shape of the palladium-nickel plated part. Guide B 765 is suitable to assist in the selection of porosity tests for electrodeposits of palladium-nickel alloys.

9.7 Ductility—When required, determine ductility in accordance with Practice B 489.

10. Special Government Requirements

10.1 The following special requirements shall apply when the ultimate purchaser is the federal government or an agent of the federal government.

10.1.1 Sampling—For government acceptance, the sampling plan specified in MIL-STD-105 is to be used instead of the ASTM standards specified in 8.1.

10.1.2 Thickness Testing:

10.1.2.1 In addition to the nondestructive methods outlined in Test Method B 568, a cross-sectioning method, such as that specified by Test Methods B 487 or B 748, shall be used as a referee method to confirm the precision and bias of the particular nondestructive technique that is used.

10.1.2.2 The palladium-nickel thickness on significant surfaces shall be at least 1.3 μm (50 μin.), unless otherwise specified on the drawings or in the contract. The coating on nonsignificant surfaces shall be of sufficient thickness to ensure plating continuity and uniform utility, appearance, and protection. The thickness of plating on nonsignificant surfaces, unless specifically exempted, shall be a minimum of 60 % of that specified for significant surfaces.

10.1.3 Packaging—The packaging and packing requirements shall be in accordance with Practice D 3951 or as specified in the contract or order.

APPENDIXES

(Nonmandatory Information)

X1. SOME REASONS FOR USING A GOLD OVERPLATE

X1.1 A gold overplate is employed to enhance the performance of the palladium-nickel surface. Two types of gold are used:

X1.1.1 Type 1 gold is used in the critical areas in thickness ranges of 0.05 to 0.12 μm.

X1.1.2 Type 2 gold is used in the critical areas in thickness ranges of 0.05 to 0.25 μm or higher.

X1.2 The gold overplate offers the following performance enhancements to palladium-nickel:

X1.2.1 Durability—Palladium-nickel has a higher coefficient of friction than gold. A gold overplate of proper thickness, therefore, reduces friction and enhances durability. The gold overplate actually provides a low shear strength solid lubricant that reduces friction wear (5, 6). Either Type 1 or Type 2 gold
works in this application (6-8). Type 1 gold should be used at a thickness no greater than 0.12 µm to maintain a low coefficient of friction. Palladium-nickel should not be mated against itself in a sliding contact pair when durability and resistance to fretting is desired.

X1.2.2 Mating Force—Application of Type 1 or Type 2 gold reduces friction and mating force. Type 1 should be no more than 0.12 µm thick.

X1.2.3 Fretting—Fretting occurs when two surfaces undergo low amplitude, repetitive motions. Depending on conditions and contact surface materials, fretting wear or fretting corrosion can occur. Fretting wear is loss of material along the wear track. Fretting corrosion is the formation of surface oxides at the contact surface. The addition of a Type 1 or Type 2 gold can often reduce fretting corrosion that is due to fretting motions (9). The occurrence of fretting is influenced greatly by contact design. See Terminology B 542.

X1.2.4 Frictional Polymerization—Frictional polymerization is the formation of insulating polymeric films at the contact spot. Such occurrences have been documented for palladium-nickel, pure palladium, and other metals (5). The addition of a Type 1 or Type 2 gold overplate can often reduce frictional polymer formation (9).

X1.2.5 Solderability—The addition of a Type 1 or Type 2 gold overplate enhances the solderability and shelf life of palladium-nickel. Type 1 gold is more solderable than Type 2.

X1.2.6 Thermal Stability—Gold overplating of palladium-nickel plated surfaces greatly improves the stability of contact resistance during prolonged exposure to temperatures ranging from 105° to 120°C (10). A Type 1 gold overplate imparts better thermal stability than a Type 2 gold overplate.

X2. THICKNESS MEASUREMENTS OF PALLADIUM-NICKEL ALLOY COATINGS BY XRF

X2.1 These guidelines are intended to aid purchasers of palladium-nickel alloy electroplating systems in properly setting up XRF instruments to measure the deposit thickness. This calibration method and measurement mode is for the determination of coating thickness only. The procedure is not intended for alloy composition measurement.

X2.2 Base metal type (substrate composition) must be considered for each product to be measured. Due to X-ray interference from the tin in phosphor-bronze substrates, some X-ray units must be calibrated using standards having the same substrate material as the product to be measured. Some commercially available X-ray units have substrate correction capability and do not require different standards for different copper alloy substrates. Follow the instrument manufacturer’s instructions when calibrating the instrument for measurement on copper alloy substrates.

X2.3 A bare substrate, a palladium-nickel alloy saturation thickness standard, and at least two palladium-nickel alloy thickness standards having the same alloy composition as the product to be measured are required for calibration of the X-ray unit. The thickness of the alloy standards must bracket the production plating thickness range to be measured. The thickness saturation standard should have a thickness greater than 60 µm (240 µin.).

X2.3.1 Alternatively, pure palladium standards could be used in place of the palladium-nickel standards of X2.3. By this method, the XRF will measure only the palladium counts emitted from the palladium-nickel sample. Providing the alloy composition is known, the palladium-nickel thickness can be determined by multiplying the XRF value by the correction factor given in Table 4.

X2.4 In calibrating the X-ray unit, the following guidelines are recommended:

X2.4.1 Select single coating excitation measurement and calibration mode;

X2.4.2 Do not use a filter (absorber);

X2.4.3 Calibrate with the same collimator size as will be needed to measure product;

X2.4.4 Total calibration measurement time per standard (number of measurements per standard multiplied by the calibration measurement time) should be at least 120 s. Multiple readings should be taken from each standard, for example, 4 measurements per standard multiplied by a 30 s measurement time each equals 120 s.

X2.4.5 Verify that a proper region of interest window (R.O.I.) has been set. Some instruments set the R.O.I. automatically from a list of applications (older instruments require manual R.O.I. setting).

X2.4.6 Ensure that the instrument has been adjusted for intensity variations (intensity correction or reference measurement, depending on instrument manufacturer).

X2.4.7 Following the calibration, measure the calibration standards to verify that the instrument is properly calibrated.

---

### Table 4: Correction Factors

<table>
<thead>
<tr>
<th>Mass % Pd</th>
<th>Pd-Ni Density Factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>60.0</td>
<td>10.58</td>
</tr>
<tr>
<td>62.0</td>
<td>10.64</td>
</tr>
<tr>
<td>64.0</td>
<td>10.69</td>
</tr>
<tr>
<td>66.0</td>
<td>10.75</td>
</tr>
<tr>
<td>68.0</td>
<td>10.80</td>
</tr>
<tr>
<td>70.0</td>
<td>10.86</td>
</tr>
<tr>
<td>72.0</td>
<td>10.92</td>
</tr>
<tr>
<td>74.0</td>
<td>10.97</td>
</tr>
<tr>
<td>76.0</td>
<td>11.03</td>
</tr>
<tr>
<td>78.0</td>
<td>11.08</td>
</tr>
<tr>
<td>80.0</td>
<td>11.14</td>
</tr>
<tr>
<td>82.0</td>
<td>11.20</td>
</tr>
<tr>
<td>84.0</td>
<td>11.25</td>
</tr>
<tr>
<td>86.0</td>
<td>11.31</td>
</tr>
<tr>
<td>88.0</td>
<td>11.36</td>
</tr>
<tr>
<td>90.0</td>
<td>11.42</td>
</tr>
<tr>
<td>92.0</td>
<td>11.48</td>
</tr>
<tr>
<td>94.0</td>
<td>11.53</td>
</tr>
<tr>
<td>96.0</td>
<td>11.59</td>
</tr>
<tr>
<td>98.0</td>
<td>11.64</td>
</tr>
<tr>
<td>100.0</td>
<td>11.70</td>
</tr>
</tbody>
</table>

---
Measure each standard ten times. The mean value of the ten measurements should fall within the predicted theoretical instrument precision for a mean of ten readings.

X2.4.8 Ensure the optical alignment (collimator to optics) of the XRF instrument is correct.

X2.4.9 Carefully focus on the measurement area of the test specimen.

X2.4.10 The test specimen should be properly oriented with respect to the X-ray detector.

X2.4.11 The surface to be measured should be flat and level with respect to the measurement stage. Curved surfaces may be measured subject to proper collimator size selection and proper orientation of the axis of curvature. Follow instrument manufacturer’s instructions.

X2.4.12 Handle the calibration standards and product carefully since surface damage will affect accuracy.

X2.4.13 Make adjustments periodically to the instrument to correct for drift effects as instructed by the manufacturer. Following this adjustment, verify the instrument calibration by measuring a known thickness standard or reference specimen.

X2.5 Prior to measuring the product containing a gold flash overplate, remove the gold with a suitable gold stripping solution. Potassium or sodium cyanide based gold stripping solutions are commercially available.

X2.6 Recommended measurement times to ensure accurate and reproducible results are as follows:

<table>
<thead>
<tr>
<th>Plating Thickness (µm)</th>
<th>Measurement Time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0–0.25</td>
<td>25</td>
</tr>
<tr>
<td>0.25–1.0</td>
<td>20</td>
</tr>
<tr>
<td>&gt;1.0</td>
<td>15</td>
</tr>
</tbody>
</table>

X3. ELECTRICAL CONTACT PERFORMANCE VERSUS THICKNESS CLASS

X3.1 General—It is difficult to specify electrical contact performance as a function of plating thickness because performance is also strongly affected by such things as metallurgy, contact design, quality, and fabrication techniques. It is possible, therefore, to have a wide range of thickness classes for a particular application.

X3.2 Electrical contact performance is generally defined in terms of the ability of a contact system to maintain a low and stable interfacial resistance when exposed to mating cycles in the product’s service or storage environment. This is usually determined by some type of environmental performance test such as those involving mixed flowing gas, for example Test Methods B 827 and B 845.

X3.3 Palladium-nickel thickness ranges that are often used in the electronics industry for specific applications are as follows:

<table>
<thead>
<tr>
<th>Thickness (µm)</th>
<th>Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.8–3.0</td>
<td>Printed circuit edge card connectors</td>
</tr>
<tr>
<td>0.8–1.5</td>
<td>Low energy electrical contacts</td>
</tr>
<tr>
<td>0.4–0.5</td>
<td>Electrical contacts where little adverse environmental, electrical, or mechanical action is expected.</td>
</tr>
</tbody>
</table>

X4. INTERMATEABILITY GUIDELINES

X4.1 Most mating combinations are acceptable when both mating halves have either precious metal or non-precious metal. The mixture of precious metal with non-precious metal is generally not recommended.

X4.2 The keys to good contact functioning are contact force, stability through good design, and the proper selection of coatings. Lubrication, with its advantages and disadvantages, also can play a beneficial role.

X4.3 The use of special lubricants can serve to improve environmental resistance, wear resistance, and reduce mating force. Lubrication can also reduce the severity of fretting wear and fretting corrosion (11).

X4.4 In unlubricated situations, the following combinations are recommended:

| Contact 1 Coating Metallurgy With: Contact 2 Coating Metallurgy |
|------------------|------------------|
| PdNi             | PdNi             |
| PdNi with gold overplate (Type 1 or 2 gold) | PdNi | PdNi | PdNi | Solder or tin |
| PdNi with gold overplate (Type 1 or 2 gold) | PdNi with gold overplate (Type 1 or 2 gold) |

X4.5 Specific applications with wide differences in thickness class, high normal forces, or unusual performance requirements should be investigated to ensure functionality.

X4.6 In unlubricated situations, the following combinations are not recommended:

<table>
<thead>
<tr>
<th>Contact 1</th>
<th>With:</th>
<th>Contact 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>PdNi</td>
<td>PdNi</td>
<td>PdNi</td>
</tr>
<tr>
<td>PdNi</td>
<td>PdNi</td>
<td>Solder or tin</td>
</tr>
<tr>
<td>PdNi with gold overplate (Type 1 or 2 gold)</td>
<td>PdNi with gold overplate (Type 1 or 2 gold)</td>
<td></td>
</tr>
</tbody>
</table>

X4.7 When suitable lubricants are applied to one or both mating halves, the guidelines in X4.4 still apply. Lubricants, however, can change the performance and alter the guidelines expressed in X4.6, with the exception of those for solder or tin.
X5. SOME REASONS FOR USING A NICKEL UNDERPLATE FOR PALLADIUM-NICKEL ELECTROPLATING

X5.1 *Diffusion Barrier*—To inhibit diffusion of copper from the basis metal into the palladium-nickel.

X5.2 *Levelling Layer*—To produce a smoother surface than the basis metal in order to ensure a lower porosity palladium-nickel top coat, for example, levelling nickel over a rough substrate.

X5.3 *Pore Corrosion Inhibitor*—A nickel underplate under the palladium-nickel top coat will form passive oxides at the base of pores in humid air, provided the environment does not contain significant amounts of acidic pollutants, such as SO₂ or HCl.

X5.4 *Load Bearing Underlayer for Contacting Surfaces*—A hard nickel underplate can serve as a load bearing foundation for the palladium-nickel top coat and reduce the wear of the precious metal during sliding of the contacting surfaces.

X5.5 For all of these purposes, the nickel underplating must be intact, that is, not cracked, and must have sufficient thickness to achieve the particular function for which it was intended. As a general rule, the minimum thickness should be 1.3 µm (50 µin.), preferably greater. For some levelling purposes, a greater thickness may be required.

X5.6 The use of nickel also reduces the potential for copper dissolution by protecting copper based substrates against chemical attack. Some palladium-nickel electroplating solutions are prone to attacking copper and subsequent codeposition of copper impurities.

X6. RESIDUAL SALTS

X6.1 Electroplated parts are placed in water of known conductivity and agitated for a specific time. The conductivity of the water extract is measured and the increase in conductivity due to residual salts and other conducting impurities is calculated. A suggested water extract conductivity test method uses a procedure in accordance with Test Methods D 1125, Method A.

X6.2 Conductivity of water for extract test shall be 1 µS/cm or less (resistivity 1 MΩ·cm or more).

X6.2.1 A sample of the coated parts having a total surface area of 30 cm² shall ordinarily be used and extracted in 100 cm³ of equilibrated water. To prepare equilibrated water, fill a clean polyethylene bottle half-way with high-purity water (X6.1), replace the bottle cap and shake the bottle vigorously for 2 min to equilibrate the water with the CO₂ in the air. CO₂ is a component of air, is soluble in water, and forms carbonic acid, which ionizes and is at equilibrium at 0.8 µS/cm. Slowly agitate the solution for 10 min before determining the conductivity of the extract. In a closed polyethylene bottle, the equilibrated water will remain in the range from 0.8 to 1 µS/cm for at least 1 week.

X6.3 Inspection under a source of ultraviolet light is often employed to determine whether electroplating salts have been removed by the rinsing following gold electroplating. The presence of salts is evidenced by a characteristic fluorescence and should not be confused with fluorescing dirt or dirt particles.

X6.4 Water or purging stains, resulting from blind holes or from parts that were assembled before electroplating, as normally obtained in good commercial practice, are permissible except where they occur on surfaces to which electrical contact is to be made or on which subsequent soldering operations are performed.

X7. PROCEDURE FOR ALLOY COMPOSITION ANALYSIS BY SCANNING ELECTRON MICROSCOPY/X-RAY ENERGY DISPERSIVE SPECTROSCOPY

X7.1 The composition of electrodeposited palladium-nickel alloy coatings containing 70 to 95 mass % Pd that are ≥1.5 µm can be analyzed to a precision of ±0.2 mass % by the procedure outlined below:

X7.1.1 Obtain two EPMA certified composition standards that bracket the desired range of palladium-nickel alloy compositions (see 9.2.4).

X7.1.2 Make three EDS analyses of mass percent palladium in the certified region of each standard under the conditions cited below:

| Accelerating Voltage: 20 kV |
| Working Distance: Optimum for the microscope |
| Tilt Angle: 0° |
| Count Time: 100 s |
| Beryllium Window: In |

X7.1.3 The sample should be moved slightly between readings and obvious coating defects, such as pores or mechanical damage, should be avoided.

X7.1.4 Average the three EDS composition analyses on each standard and calculate a correction factor (CF) for each composition standard from the equation below:
X7.1.5 Calculate the average correction factor, \((CF)_{ave}\) for the two standards.

X7.1.6 Make three EDS mass percent palladium composition analyses on the unknown coating samples according to the procedure outlined in X7.1.2.

X7.1.7 Multiply the average EDS mass percent palladium for the unknown sample times \((CF)_{ave}\) for day X to obtain the correct alloy composition.

X7.1.8 The standard deviation for the \((CF)_{ave}\) should be \(\pm 0.005\) based on measurements made on a large number of different commercial SEM/EDS instruments. If the standard deviation for the \((CF)_{ave}\) is \(\pm 0.010\), the procedure described above is not satisfactory and Reference 13 should be consulted concerning possible alternatives.

X7.2 For palladium-nickel alloy coatings that are 1.0 µm thick and deposited directly onto a 1.5 µm thick coating of pure nickel, the palladium content measured by this technique will be about 0.3 to 1.0 mass % less than the actual coating composition. For palladium-nickel alloy coatings that are 0.75 µm thick and deposited directly onto a 1.5 µm thick coating of pure nickel, the palladium content measured by this technique will be about 1 to 2 mass % less than the actual coating composition.

\[
(CF) = \frac{\text{Certified Mass % Pd for Standard}}{\text{Average Measured Mass % Pd on Day X}}
\]

X8. ELECTRON PROBE X-RAY MICROANALYSIS (EPMA)

X8.1 The EPMA can be used to measure composition of palladium-nickel alloy coatings on top of any undercoat or any substrate if the thickness of the coating is \(\geq 1.5\) µm and the accelerating voltage for the electrons is in the range of 16 to 22 kV (an accelerating voltage of 20 kV is preferred). Under these conditions, there will be no significant excitation of characteristic X-rays from elements in substrates or undercoatings (12).

X8.2 The composition of palladium-nickel alloy coatings can be accurately analyzed by EPMA to a precision of \(\pm 0.1\) mass % using an X-ray WDS and a computer program developed by NIST that corrects for background, absorption, secondary fluorescence, and atomic number effects (12, 13). This technique is suitable for the certification of alloy standards for composition analyses by other techniques based on excitation of X-ray and electron radiations such as those cited in 9.2.5 and 9.2.6. The recommended palladium-nickel alloy coating thickness for an EPMA certified standard is \(\geq 2.3\) µm.

REFERENCES


1. Scope

1.1 This test method covers a procedure for measuring the volume of the apparent density cups used in Test Methods B 212, B 329, and B 417.

1.2 The apparent density cup, particularly its rim, may become worn during use, and it is recommended that the volume of the cup be checked periodically (at least every 6 months) in order to ensure that it complies with the specified volume.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 212 Test Method for Apparent Density of Free-Flowing Metal Powders Using the Hall Flowmeter Funnel
B 243 Terminology of Powder Metallurgy
B 329 Test Method for Apparent Density of Metal Powders and Compounds Using the Scott Volumeter
B 417 Test Method for Apparent Density of Non-Free-Flowing Metal Powders Using the Carney Funnel

3. Terminology

3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243. Additional descriptive information is available in the Related Materials section of Vol 02.05 of the Annual Book of ASTM Standards.

4. Summary of Test Method

4.1 The apparent density cup is filled with water and weighed to obtain the mass of water contained, which is then converted to a volume.

5. Significance and Use

5.1 This test method enables the measurement of the volume of the apparent density cup to ensure that it complies with the specified volume of 25.00 ± 0.03 cm³ (cylindrical cup), or 16.39 ± 0.05 cm³ (square cup). Use of an out-of-specification cup will give erroneous apparent density values using the formulae in Test Methods B 212, B 329, and B 417.

6. Apparatus

6.1 Density Cup—A cylindrical cup (see Fig. 1) designed to have a capacity of 25.00 ± 0.03 cm³ (as in Test Methods B 212, B 329, and B 417) or a square cup designed to have a capacity of 16.39 ± 0.05 cm³ (as in Test Method B 329).

6.2 Balance, suitable for weighing at least 200 g to the nearest 0.001 g.

6.3 Microscope Slide—A transparent microscope slide at least as wide as the outer diameter of the apparent density cup.

6.4 Water—Distilled or deionized water boiled to remove dissolved air.

6.5 Alcohol—Low residue ethyl alcohol.

6.6 Wire—A wire not exceeding 2.5 mm in diameter by 150 mm long.

6.7 Thermometer—A thermometer capable of measuring the temperature of the water to the nearest 1°C.

7. Procedure

7.1 Boil at least 150 mL of distilled or deionized water for 5 min to remove dissolved air. Cool to room temperature and handle so as to avoid the introduction of air bubbles.

7.2 Clean the density cup with alcohol and dry thoroughly.

7.3 Weigh, to the nearest 0.001 g, the density cup along with a clean, clear microscope slide. This is the tare mass, T.

7.4 Fill the density cup with the prepared water until the water overflows.

7.5 Dislodge any air bubbles inside the density cup with a clean wire.

7.6 Slide the clean, clear microscope slide horizontally across the rim of the density cup, spilling the excess water over the sides of the cup and center the microscope slide on top of the cup.

7.7 If any air bubbles appear, remove the microscope slide and return to 7.4.
7.8 Dry the outside of the density cup and the exposed surfaces of the microscope slide with paper toweling. Ensure that the absorbency of the toweling does not wick water from inside the cup.

7.9 Weigh, to the nearest 0.001 g, the dried density cup filled with water with the microscope slide on top. This is the gross mass, $G$.

7.10 Measure the temperature of the water to the nearest 1°C and determine the density of the water, $\rho_w$, from Table 1.

8. Calculation

8.1 Calculate the mass of water contained in the density cup from the following formula:

$$M = G - T$$

where:
- $M$ = mass of water in the density cup,
- $G$ = gross mass of density cup, plus water, plus microscope slide, and
- $T$ = tare mass of density cup plus microscope slide.

8.2 Calculate the volume of the density cup, $V$, from the following relationship:

$$V = \frac{M}{\rho_w}$$

where:
- $V$ = volume of the density cup,
- $M$ = mass of water in the density cup, and
- $\rho_w$ = density of the water from Table 1.

9. Report

9.1 Report the volume of the density cup to the nearest 0.01 cm$^3$.

9.1.1 Cylindrical Cup:

9.1.1.1 If the measured volume of the cylindrical density cup falls within the specified range 25.00 ± 0.03 cm$^3$, it is permissible to use 25 cm$^3$ in the formula for calculating apparent density in Test Methods B 212, B 329, and B 417.

9.1.1.2 If the measured volume of the cylindrical density cup falls outside the specified range of 25.00 ± 0.03 cm$^3$ but falls within the broader range of 24.8 to 25.2 cm$^3$, then the measured volume should be used in the formula for calculating apparent density in Test Methods B 212, B 329, and B 417.

9.1.2 Square Cup:

9.1.2.1 If the measured volume of the square density cup falls within the specified range 16.39 ± 0.05 cm$^3$, it is permissible to use 16.39 cm$^3$ in the formula for calculating apparent density in Test Method B 329.

9.1.2.2 If the measured volume of the square density cup falls outside the specified range of 16.39 ± 0.05 cm$^3$ but falls within the broader range of 16.2 to 16.6 cm$^3$, then the measured volume should be used in the formula for calculating apparent density in Test Method B 329.

10. Precision and Bias

10.1 The precision and bias of this test method is currently under development in Subcommittee B09.02 on Base Metal Powders.

11. Keywords

11.1 apparent density cup; cup volume; density cup; measuring cup volume; volume of cup
Standard Specification for Chromium Diffusion Coating Applied by Pack Cementation Process

1. Scope

1.1 This specification covers the requirements for chromium diffusion of metals by the pack cementation method. Pack diffusion employs the chemical vapor deposition of a metal which is subsequently diffused into the surface of a substrate at high temperature. The material to be coated (substrate) is immersed or suspended in a powder containing chromium (source), a halide salt (activator), and an inert diluent such as alumina (filler). When the mixture is heated, the activator reacts to produce an atmosphere of chromium halides which transfers chromium to the substrate for subsequent diffusion. The chromium-rich surface enhances corrosion, thermal stability, and wear-resistant properties.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 374 Terminology Relating to Electroplating
B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 762 Test Method of Variables Sampling of Metallic and Inorganic Coatings
C 664 Test Methods for Thickness of Diffusion Coating
D 3951 Practice for Commercial Packaging

3. Terminology

3.1 Definitions used in this specification are in accordance with Terminology B 374.

3.2 Definitions of Terms Specific to This Standard:
3.2.1 diffusion coating—a diffusion coating is one produced by causing an element to react with or diffuse into, or both, the surface of a metallic substrate, thus, chemically altering the surface of the substrate.
3.2.2 retorts—containers in which powder and parts are packed for processing. They can be constructed of carbon, stainless, or high alloys and fabricated in all shapes and sizes to accommodate parts being processed.
3.2.3 significant surface—areas that are essential to the serviceability or function of the article. These surfaces must be identified on a drawing or marked-up sample of product. Areas can fall into one of three categories as follows:
3.2.4 coating required—these surfaces must be in accordance with all quality requirements of this specification.
3.2.5 no coating required—these surfaces are areas where no coating is allowed due to a number of reasons including dimensional, fabrication, and welding, as well as others. Materials used for masking are commercially available.
3.2.6 optional—these surfaces do not require coating, but at the same time do not require masking.

4. Classification

4.1 There are four classes of chromium diffusion defined by base (basis) metal category.
4.1.1 Class I—Carbon steel.
4.1.2 Class II—Low-alloy steels.
4.1.3 Class III—Stainless steels.
4.1.4 Class IV—Nickel-based alloys.

5. Ordering Information
5.1 In order to make the application of this specification complete, the purchaser shall supply the following information to the vendor through a purchase order and drawings:
5.1.1 Title, ASTM designation number, and date of issue of this specification.
5.1.2 Deposit by classification (see Section 4).
5.1.3 Composition and metallurgical condition of substrate to be coated.
5.1.4 Location of significant surfaces (see 3.2.3).
5.1.5 Samples for destructive metallurgical test (see 8.1).
5.1.6 Any post heat treatment required.
5.1.7 Acceptance inspection procedure to be used (see Section 9).
5.1.8 Any requirement for certification (see Section 11).

6. Processing Requirements
6.1 Substrate Preparation—The metal to be chromized shall be free of flaws and defects that will be detrimental to the coating. Thorough cleaning is essential to ensure satisfactory diffusion. Materials used for cleaning should not damage the base metal. Oils, dirt, grease, and stains must be removed. When blasting is also required, use aluminum oxide (90 to 220 mesh) at 60 to 80 psi from 4 to 6 in. standoff. (Refer to Guide F 1330.)
6.2 Materials:
6.2.1 Masteralloy with 50 to 100 % Pure Chromium—Sold under a number of trade names, in various mesh sizes. Percentage in mix depends on material being processed.
6.2.2 Activator—Most commonly used is ammonium chloride, but many others are available. Percentage in mix depends on material and type of activity.
6.2.3 Inert Filler—Aluminum oxide or calcined alumina is used and commercially available in numerous mesh sizes. Percentage in mix depends on material being processed and mesh size being used.

NOTE 1—It is important that both proper equipment is used and time established to guarantee uniform blend of pack mix.

NOTE 2—It is common practice to rejuvenate used mixes by addition of coating elements, alloys, and activators. Chemical analysis of used mixes facilitates control of critical constituents. Disposal of used mixes must be performed in compliance with environmental regulations.

6.3 Loading—Place pack mix and parts in retorts. Retort size is limited by dimensions of furnace used for processing.
6.4 Furnace Cycle—Parts are heated to a temperature between 1900 and 2400°F depending on base material and pack mix and held for amount of time needed to produce desired depth of diffusion.

NOTE 3—Due to the high temperature of the process, a positive pressure of an inert gas, usually argon, must be maintained in retorts throughout the entire furnace cycle.

6.5 Post-Cleaning—Retorts should be allowed to cool sufficiently before opening. When parts are removed from retorts, residual pack mix is removed from surfaces by a supplier-approved method.

6.6 Post-Straightening—Long tubes may have distortion due to the high temperature of process. Use rotary straightener or hydraulic press to restore straightness and ovality.
6.7 Visual Inspection—Inspect in accordance with 7.3.
6.8 Marking and Packaging—to be defined by the purchaser in the purchase order. Parts processed for the U.S. government and military, including subcontract, shall be packaged in accordance with Practice D 3951.

7. Coating Requirements
7.1 Diffusion Thickness—The following are minimums for the different classes of materials based on standard process parameters. Thicker coatings require special processing and must be called out for in the purchase order if required.

<table>
<thead>
<tr>
<th>Class</th>
<th>Minimum, in.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Class I (carbon)</td>
<td>0.003</td>
</tr>
<tr>
<td>Class II (low alloys)</td>
<td>0.003</td>
</tr>
<tr>
<td>Class III (stainless steels)</td>
<td>0.002</td>
</tr>
<tr>
<td>Class IV (nickel base alloys)</td>
<td>0.001</td>
</tr>
</tbody>
</table>

7.2 Decarburization—For certain Class I and Class II materials, the maximum depth of decarburization to be defined by the purchaser.
7.3 Chromium Content—The outer 15 % of coating shall contain a minimum of 20 % (by weight) chromium.
7.4 Appearance—The diffusion zone shall be nonporous and adherent to the base metal and shall have a uniform surface free from objectionable imperfections. Minor variations in color and surface appearance shall be considered acceptable, providing the requirements of 7.1 and 7.2 are met (see 8.7).

8. Test Methods
8.1 Special Test Specimens:
8.1.1 The permission or the requirements to use special test specimens, the number to be used, the material from which they are to be made, and their shape and size shall be stated by the purchaser.

NOTE 4—Test specimens are often used to represent coated articles in a test if these articles are of a size, shape, or material that is not suitable for the test, or if it is preferred not to submit articles to a destructive test because, for example, the articles are expensive or few in number. The specimen should duplicate the characteristics of the article that influence the property being tested.

8.1.2 Special test specimens used to represent articles in an adhesion, weldability, corrosion resistance, or appearance test shall be made of the same material, shall be in the same metallurgical condition, and shall have the same surface condition as the articles they represent, and they shall be placed and processed in the production lot along with the articles they represent.

8.2 Thickness—Perform the destructive test on samples in accordance with Test Methods C 664. Read and record the thickness of samples in accordance with Test Method B 487.
8.3 Decarburization—Determined by metallographic examination in accordance with Test Method E 1077.
8.4 Chromium Content—Perform by scanning electron microscope (refer to Practice E 766).
8.5 Mechanical Properties (if required)—Tensile testing on samples from each process batch.
8.6 Hardness Testing (if required)—Macrohardness and microhardness testing are both acceptable means.

8.7 Appearance—The coating can be examined at up to $5 \times$ magnification for conformance to appearance requirements.

9. Sampling

9.1 The sampling plan used for inspection of a quantity of coated articles shall be agreed upon by the purchaser and the supplier.

NOTE 5—Usually, when a collection of coated articles, the inspection lot (9.2), is examined for compliance with the requirements placed on the coating, a relatively small number of the articles, the sample, is selected at random and is inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria of compliance are determined by the application of statistics. This procedure is known as sampling inspection. Three standards, Test Method B 602, Guide B 697, and Method B 762, contain sampling plans that are designed for the sampling inspection of coatings.

Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one for tests that are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 designates the plan to be used.

Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and the seller need to agree on the plan to be used.

Method B 762 can be used only for coating requirements that have a numerical limit, such as a coating thickness. The test must yield a numerical value, and certain statistical requirements must be met. Method B 762 contains several plans and also gives instructions for designating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Method B 762 designates the plan to be used.

NOTE 6—When both destructive and nondestructive tests exist for the measurement of a characteristic, the purchaser needs to state which one is to be used so that the proper sampling plan is selected. A test may destroy the coating, but in a noncritical area; or, although it may destroy the coating, a tested part may be reclaimed by stripping and recoating. The purchaser needs to state whether the test is to be considered destructive or nondestructive.

9.2 An inspection lot shall be defined as a collection of coated articles of the same kind that have been produced to the same specifications, coated by a single supplier at one time or approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

9.3 If special test specimens are used to represent the coated articles in a test, the number used shall be that required in 8.1.1.

10. Rejection and Rehearing

10.1 Parts that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer promptly in writing. In the case of dissatisfaction with the results of a test, the producer may make a claim for a rehearing. Coatings that show imperfections may be rejected.

11. Certification

11.1 When specified in the purchase order or contract, the purchaser shall be furnished with certifications that the samples representing each lot have been either tested or inspected as directed by this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results must be furnished.

12. Record Retention

12.1 The seller is to maintain all job records and samples for a period of two years unless the purchaser requires a longer period.

13. Keywords

13.1 chromizing; corrosion-resistant coatings; diffusion coating; pack cementation
Standard Specification for Aluminum Diffusion Coating Applied by Pack Cementation Process\(^1\)

This standard is issued under the fixed designation B 875; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (\(\epsilon\)) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers the requirements for aluminum diffusion of metals by the pack cementation method. Pack diffusion employs the chemical vapor deposition of a metal which is subsequently diffused into the surface of a substrate at high temperature. The material to be coated (substrate) is immersed or suspended in a powder containing aluminum (source), a halide salt (activator), and an inert diluent such as alumina (filler). When the mixture is heated, the activator reacts to produce an atmosphere of aluminum halides which transfers aluminum to the substrate for subsequent diffusion. The aluminum-rich surface enhances corrosion, thermal stability, and wear-resistant properties.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
- B 374 Terminology Relating to Electroplating\(^2\)
- B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of Cross Section\(^2\)
- B 567 Test Method for Measurement of Coating Thickness by Beta Backscatter Method\(^2\)
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings\(^2\)
- B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings\(^2\)
- B 762 Test Method of Variables Sampling of Metallic and Inorganic Coatings\(^2\)
- C 664 Test Methods for Thickness of Diffusion Coating\(^2\)
- D 3951 Practice for Commercial Packaging\(^5\)
- E 3 Practice for Preparation of Metallographic Specimens\(^4\)
- F 1330 Guide for Metallic Abrasive Blasting to Descle

3. Terminology

3.1 Definitions used in this specification are in accordance with Terminology B 374.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 diffusion coating—a diffusion coating is one produced by causing an element to react with or diffuse into, or both, the surface of a metallic substrate, thus, chemically altering the surface of the substrate.

3.2.2 retorts—containers in which powder and parts are packed for processing. They can be constructed of carbon, stainless, or high alloys and fabricated in all shapes and sizes to accommodate parts being processed.

3.2.3 significant surface—areas that are essential to the serviceability or function of the article. These surfaces must be identified on a drawing or marked-up sample of product. Areas can fall into one of three categories as follows:

3.2.4 coating required—these surfaces must be in accordance with all quality requirements of this specification.

3.2.5 no coating required—these surfaces are areas where no coating is allowed due to a number of reasons including dimensional, fabrication, and welding, as well as others. Materials used for masking are commercially available.

3.2.6 optional—these surfaces do not require coating, but at the same time do not require masking.

4. Classification

4.1 There are three classes of aluminum diffusion defined by base (basis) metal category.

4.1.1 Class I—Carbon steel and low alloy.

4.1.2 Class II—Stainless steels.

4.1.3 Class III—Nickel-based alloys.

5. Ordering Information

5.1 In order to make the application of this specification complete, the purchaser shall supply the following information to the vendor through a purchase order and drawings:

\(^1\) This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.15 on Diffusion Coatings.
\(^2\) Annual Book of ASTM Standards, Vol 02.05.
\(^3\) Annual Book of ASTM Standards, Vol 15.09.
\(^4\) Annual Book of ASTM Standards, Vol 03.01.
\(^5\) Annual Book of ASTM Standards, Vol 01.07.
6.4.1 High-activity packs are heated to a temperature between 1100 and 1500°F and held for the amount of time needed to produce a sufficient deposit of aluminum which after a high-temperature diffusion cycle meets thickness requirements. Time is also dependent on activator(s) being used.

6.4.2 Low-activity packs are heated to a temperature between 1650 and 2100°F and held for the amount of time needed to produce the desired level of diffusion.

6.5 Post-Cleaning—Retorts should be allowed to cool sufficiently before opening. When parts are removed from retorts, residual pack mix is removed from surfaces by a supplier-approved method.

6.6 Diffusion Heat Treatment—Parts processed in high activity packs are placed in the retort and under inert atmosphere of argon or hydrogen are heated to 1900 to 2000°F and held for the amount of time needed to produce the desired level of diffusion.

6.7 Post-Straightening—Long tubes may have distortion due to the high temperature of process. Use rotary straightener or hydraulic press to restore straightness and ovality.

6.8 Visual Inspection—Inspect in accordance with 7.3.

6.9 Marking and Packaging—To be defined by the purchaser in the purchase order. Parts processed for the U.S. government and military, including subcontracts, shall be packaged in accordance with Practice D 3951.

7. Coating Requirements

7.1 Diffusion Thickness—The following are minimums for the different classes of materials based on standard process parameters. Thicker coatings require special processing and must be called out for in the purchase order if required.

<table>
<thead>
<tr>
<th>Class</th>
<th>Minimum, in.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Class I (carbon and low alloy)</td>
<td>0.005</td>
</tr>
<tr>
<td>Class II (stainless steels)</td>
<td>0.003</td>
</tr>
<tr>
<td>Class III (nickel-base alloys)</td>
<td>0.001</td>
</tr>
</tbody>
</table>

7.2 Aluminum Content—The outer 15% of coating shall contain a minimum of 28% (by weight) aluminum.

7.3 Appearance—The diffusion zone shall be nonporous and adherent to the base metal and shall have a uniform surface free from objectionable imperfections. Minor variations in color and surface appearance shall be considered acceptable, providing the requirements of 7.1 and 7.2 are met (see 8.6).

8. Test Methods

8.1 Special Test Specimens:

8.1.1 The permission or the requirements to use special test specimens, the number to be used, the material from which they are to be made, and their shape and size shall be stated by the purchaser.

NOTE 4—Test specimens are often used to represent coated articles in a test if these articles are of a size, shape, or material that is not suitable for the test, or if it is preferred not to submit articles to a destructive test, because, for example, the articles are expensive or few in number. The specimen should duplicate the characteristics of the article that influence the property being tested.

8.1.2 Special test specimens used to represent articles in an adhesion, weldability, corrosion resistance, or appearance test shall be made of the same material, shall be in the same metallurgical condition, and shall have the same surface condition as the articles they represent, and they shall be placed and processed in the production lot along with the articles they represent.

8.2 Thickness—Perform the destructive test on samples in accordance with Test Methods C 664. Read and record the results in accordance with Test Method B 487.

8.3 Aluminum Content—Shall be performed by beta backscatter or X-ray fluorescence.
8.4 **Mechanical Properties** (if required)—Tensile testing on samples from each process batch.

8.5 **Hardness Testing** (if required)—Macrohardness and microhardness testing are both acceptable means.

8.6 **Appearance**—The coating can be examined at up to 5× magnification for conformance to appearance requirements.

### 9. Sampling

9.1 The sampling plan used for inspection of a quantity of coated articles shall be agreed upon by the purchaser and the supplier.

**NOTE 5**—Usually, when a collection of coated articles, the inspection lot (9.2), is examined for compliance with the requirements placed on the coating, a relatively small number of the articles, the sample, is selected at random and is inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria of compliance are determined by the application of statistics. This procedure is known as sampling inspection. Three standards, Test Method B 602, Guide B 697, and Method B 762, contain sampling plans that are designed for the sampling inspection of coatings.

Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one for tests that are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 designates the plan to be used.

Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and the seller need to agree on the plan to be used.

Method B 762 can be used only for coating requirements that have a numerical limit, such as a coating thickness. The test must yield a numerical value, and certain statistical requirements must be met. Method B 762 contains several plans and also gives instructions for designating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Method B 762 designates the plan to be used.

**NOTE 6**—When both destructive and nondestructive tests exist for the measurement of a characteristic, the purchaser needs to state which one is to be used so that the proper sampling plan is selected. A test may destroy the coating, but in a noncritical area; or, although it may destroy the coating, a tested part may be reclaimed by stripping and recoating. The purchaser needs to state whether the test is to be considered destructive or nondestructive.

9.2 An inspection lot shall be defined as a collection of coated articles of the same kind that have been produced to the same specifications, coated by a single supplier at one time or approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

9.3 If special test specimens are used to represent the coated articles in a test, the number used shall be that required in 8.1.1.

### 10. Rejection and Rehearing

10.1 Parts that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer promptly in writing. In the case of dissatisfaction with the results of a test, the producer may make a claim for a rehearing. Coatings that show imperfections may be rejected.

### 11. Certification

11.1 When specified in the purchase order or contract, the purchaser shall be furnished with certifications that the samples representing each lot have been either tested or inspected as directed by this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results must be furnished.

### 12. Record Retention

12.1 The seller is to maintain all job records and samples for a period of two years unless the purchaser requires a longer period.

### 13. Keywords

13.1 aluminizing; corrosion-resistant coatings; diffusion coating; pack cementation
Standard Test Method for Gross Defects and Mechanical Damage in Metallic Coatings by the Phosphomolybdic Acid (PMA) Method

This standard is issued under the fixed designation B 877; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test standard covers equipment and methods for using phosphomolybdic acid (PMA) to detect gross defects and mechanical damage including wear through in metallic coatings of gold, silver, or palladium. These metals comprise the topmost metallic layers over substrates of nickel, copper, or copper alloys.

1.2 Recent reviews of porosity testing, which include those for gross defects, and testing methods can be found in the literature. An ASTM guide to the selection of porosity and gross defect tests for electrodeposits and related metallic coatings is available as Guide B 765. Other related porosity and gross defects test standards are Test Methods B 735, B 741, B 798, B 799, B 809, and B 866.

1.3 The values stated in SI units are the preferred units. Those in parentheses are for information only.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 374 Terminology Relating to Electroplating
B 488 Specification for Electrodeposited Coatings of Gold for Engineering Uses
B 542 Terminology Relating to Electrical Contacts and Their Use
B 679 Specification for Electrodeposited Coatings of Palladium for Engineering Use
B 689 Specification for Electroplated Engineering Nickel Coatings
B 735 Test Method for Porosity in Gold Coatings on Metal Substrates by Nitric Acid Vapor
B 741 Test Method for Porosity in Gold Coatings on Metal Substrates by Paper Electrography
B 765 Guide for Selection of Porosity Tests for Electrodeposits and Related Metallic Coatings
B 798 Test Method for Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography
B 799 Test Method for Porosity in Gold and Palladium Coatings by Sulfurous Acid/Sulfur-Dioxide Vapor
B 809 Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor (“Flowers-of-Sulfur”)
B 866 Test Method for Gross Defects and Mechanical Damage in Metallic Coatings by Polysulfide Immersion

3. Terminology

3.1 Definitions—Many terms in this test method are defined in Terminology B 374 or B 542.

3.2 Definitions of Terms Specific to This Standard:
3.2.1 base metal, n—any metal other than gold, silver, platinum, palladium, iridium, or rhodium. Typical base metals used as underplates or substrates are copper, nickel, tin, lead, and their alloys.

3.2.2 defect indications, n—colored droplets resulting from the reaction between the PMA reagent and the underlying metal.

3.2.3 gross defects, n—those breaks in the coating that expose relatively large areas of underlying metal to the environment. Gross defects include those produced by mechanical damage and wear, as well as as-plated large pores with diameters an order of magnitude greater than intrinsic porosity and networks of microcracks.

 NOTE 1—Large pores and microcrack networks indicate serious deviations from acceptable coating practice (dirty substrates and contaminated or out-of-balance plating baths).

3.2.4 intrinsic porosity, n—the normal porosity that is present, to some degree, in all commercial thin electrodeposits (precious metal coatings for engineering purposes) that will generally follow an inverse relationship with thickness.
NOTE 2—Intrinsic porosity is due to small deviations from ideal plating and surface preparation conditions. Scanning electron microscope (SEM) studies have shown the diameter of such pores at the plating surface is 1 to 2 µm so only small areas of underlying metal are exposed to the environment.

3.2.5 measurement area, n—that portion or portions of the surface that is examined for the presence of gross defects or mechanical damage and wear through. The measurement area shall be indicated on the drawings of the parts or by the provision of suitably marked samples.

3.2.6 metallic coatings, n—include electrodeposits, claddings, or other metallic layers applied to the substrate. The coating can comprise a single metallic layer or a combination of metallic layers (gold over palladium).

3.2.7 porosity (general), n—the presence of any hole, crack, or other defect that exposes the underlying metal to the environment.

3.2.8 underplate, n—a metallic coating layer between the substrate and the topmost metallic coating. The thickness of an underplate is usually greater than 1 µm, in contrast to a strike or flash, which is usually thinner.

3.2.9 wear through, n—the exposure of underplate or substrate as a direct result of wear. Wear through is an observable phenomenon.

3.2.10 wear track, n—a mark that indicates the path along which physical contact has been made during a sliding process (the mating and unmuting of an electrical contact).

4. Summary of Test Method

4.1 This test method involves the use of a solution of phosphomolybdic acid (PMA), which is a solid complex of molybdenum trioxide, Mo₂O₃, and phosphoric acid, H₃PO₄. In this state, molybdenum is very reactive with many free metals and may be used to detect exposed underplates and substrate metals. The part is exposed briefly to fumes of hydrochloric acid to remove oxides in the defect region. A small drop of the aqueous PMA solution is applied to the spot in question using an applicator. If it contacts base metal from exposed underplate or substrate, the Mo₂O₃ will immediately be reduced to lower oxides, forming the intensely colored, molybdenum blue complex (heteropoly blue).

4.2 This test may not be suitable for some precious metal alloy coatings that contain significant concentrations of non-precious metals (base metals) like nickel or copper. (See .)

4.3 The reagents in this test also react with tin, lead, and tin-lead solder.

5. Significance and Use

5.1 The primary purpose of the PMA test is to determine the presence of mechanical damage, wear through, and other gross defects in the coating. Most metallic coatings are intended to be protective, and the presence of gross defects indicates a serious reduction of such protection.

5.2 The protection afforded by well applied coatings may be diminished by improper handling following plating or as a result of wear or mechanical damage during testing or while in service. The PMA test can serve to indicate the existence of such damage.

5.3 This test is used to detect underplate and substrate metal exposed through normal wear during relative motions (mating of electrical contacts) or through mechanical damage. As such, it is a sensitive pass/fail test and, if properly performed, will rapidly detect wear through to base metals or scratches that enter the base metal layers.

5.4 This test is relatively insensitive to small pores. It is not designed to be a general porosity test and shall not be used as such. The detection of pores will depend upon their sizes and the length of time that the reagent remains a liquid.

5.5 This test cannot distinguish degrees of wear through or whether the wear through is to nickel or copper. Once base metal is exposed, the colored molybdenum complex is formed. While relatively small area defects (compared to the area of the droplet) may be seen at the bottom of the drop as tiny colored regions immediately after applying the PMA, any larger areas of exposed base metal will cause the entire droplet to turn dark instantly.

5.6 The PMA test also detects mechanical damage that exposes underplate and substrate metal. Such damage may occur in any postplating operation or even at the end of the plating operation. It can often occur in assembly operations where plated parts are assembled into larger units by mechanical equipment.

5.7 The PMA test identifies the locations of exposed base metal. The extent and location of these exposed areas may or may not be detrimental to performance. The PMA test is not recommended for predictions of product performance, nor is it intended to simulate field failure mechanisms. For such contact performance evaluations, an environmental test known to simulate actual failure mechanisms should be used.

5.8 The PMA test is primarily intended for the evaluation of individual samples rather than large sample lots, since evaluations are normally carried out one at a time under the microscope (see Section 10).

5.9 This test is destructive. Any parts exposed to the PMA test shall not be placed in service.

6. Apparatus

6.1 In addition to the normal equipment (beakers, weighing balances, funnels, etc.) that are a part of every chemical laboratory.

6.2 Microscope, Optical, Stereo, 10 to 30×—It is preferred that one eyepiece contain a graduated reticle for measuring the defect location. The reticle shall be calibrated for the magnification at which the microscope is to be used, preferably 10×.7

6.3 Light source (illuminator) for microscope, incandescent.

6.4 Glass volumetric flask, 10 mL.

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7 Magnification standards suitable for calibrating optical microscopes may be purchased from U.S. National Institute of Standards and Technology, Office of Standard Reference Materials.
6.5 Glass bottle of a stable shape and with glass stopper. The bottle opening shall be 2.5 cm (1 in) minimum. An example is a 50-mL low-form weighing bottle or a flask-shaped weighing bottle.

6.6 Applicators (see 9.2)—Platinum wire, 32 AWG, or disposable glass micropipets, 1 or 0.5 µL size.

7. Reagents and Materials

7.1 Phosphomolybdic Acid (PMA)—Crystalline, ACS certified grade.
7.2 Concentrated Hydrochloric Acid—ACS analytical reagent (AR) grade or better.

8. Specific Safety and Health Precautions

8.1 All the normal precautions shall be observed in handling the materials required for this test. This shall include, but is not limited to, procuring and reviewing Material Safety Data Sheets that meet the minimum requirements of the OSHA Hazard Communication Standard for all chemicals used in cleaning and testing and observing the recommendations given.

9. Preparations

9.1 Preparation of solutions:
9.1.1 Two types of PMA solutions can be used with this method.
9.1.1.1 Method A, the preferred method, uses a dilute 8 % solution of PMA in water.
9.1.1.2 Method B, uses a saturated solution of PMA in water.

Note 3—The dilute solution is preferred because it works well with silver, gold, and palladium coatings, while the saturated solution reacts with silver to give false indications. In addition, the saturated solution has a tendency to dry up quickly on the test surface before proper evaluations can be made.

9.1.2 Dilute (8 %) PMA solution (for Method A):
9.1.2.1 Place a small, clean, and dry glass funnel in the neck of a clean, dry 10 mL volumetric flask.
9.1.2.2 Tare out the weight of the funnel and flask on a balance.
9.1.2.3 Weigh 0.8 (±0.1) g PMA into the flask, using a plastic or glass spatula.
9.1.2.4 Rinse the funnel with distilled or deionized water to drain any adhering PMA into the flask.
9.1.2.5 Dilute to mark with deionized water.
9.1.2.6 Place stopper in flask and mix thoroughly. Cloudy solution will clear after standing 10 to 15 min.
9.1.2.7 Pour clear solution into a clean glass bottle and seal with glass stopper. Label bottle with PMA concentration and date of preparation.
9.1.2.8 Store bottle in refrigerator. Solution may be used for one week.
9.1.3 Saturated PMA solution (for Method B):
9.1.3.1 Prepare solution in accordance with 9.1.2.1-9.1.2.6, except use approximately 5 g of PMA instead of 0.8 g. (Filter out sediment, if necessary.)
9.1.3.2 Mix thoroughly for at least 10 min.

Note 4—There shall be a small excess of PMA, seen as a sediment in the bottom of the flask. This indicates saturation.

9.1.3.3 Pour into a clean bottle and label bottle with contents and preparation date.
9.1.3.4 Solution may be used for one week. Store in refrigerator when not in use.
9.1.4 Hydrochloric acid (for both methods):
9.1.4.1 Fill the special glass bottle (see 6.4) to approximately halfway from the top.
9.1.4.2 Label glass bottle with contents.
9.1.4.3 Keep stoppered and under a fume hood when not in use.

9.2 Preparation of applicators:
9.2.1 The applicator shall not react with the PMA solution. Examples are as follows:
9.2.1.1 Platinum—Make a small loop using a 32 AWG platinum wire and an appropriate size mandrel (such as a needle). Leave a small gap to facilitate release of the PMA droplet (see Fig. 1). Attach loop to a wooden or plastic handle.
9.2.1.2 Platinum inoculating loops with handles may be purchased. Cut the loop with a knife to create a small gap (Fig. 1), which will facilitate the release of the PMA droplet.
9.2.1.3 Glass capillary micropipets in the 1-µL size or smaller.
9.2.2 If a platinum loop is used as the applicator, the loop diameter shall preferably be 1 mm and shall not exceed 2 mm. The loop diameter is kept small for the following reasons:
9.2.2.1 The small dimensions of many examination areas.
9.2.2.2 The ability of the loop to release a rounded droplet instead of a thin sheet of solution, which dries too fast.
9.2.2.3 Difficulty in controlling flow and observing reactions in large drops.

9.3 Preparation of test samples:
9.3.1 Handle samples as little as possible even prior to cleaning and only with tweezers, microscope-lens tissue, or clean, soft cotton gloves.
9.3.2 Prior to being cleaned, the samples shall be prepared so the measurement area is accessible and can be placed in a basically horizontal plane. This allows for easy viewing through the microscope and prevents the PMA solution from running off during application.
9.3.3 Masking:
9.3.3.1 The PMA solution will react with any exposed base metal such as nickel, copper, tin, lead, or solder. If the examination area is within a millimetre of exposed or thinly plated substrate metal, masking may be necessary.
9.3.3.2 If masking is necessary, clean per 9.3.4. Carefully paint the nonmeasurement areas with stop-off lacquer under a microscope using a fine artist’s brush. Allow the samples to dry thoroughly.
9.3.4 Cleaning the Test Samples:

9.3.4.1 Inspect the samples under 10× magnification for evidence of particulate matter. If present, such particles should be removed by dusting (blowing them off the sample) with clean, oil-free air.

9.3.4.2 Thoroughly clean the particle-free samples with solvents or solutions that do not contain CFC’s, chlorinated hydrocarbons, or other known ozone-destroying compounds. The procedure outlined in Note 5 has been found to give satisfactory results for coatings with mild to moderate surface contamination.

**Note 5—**Suggested cleaning procedure: (1) Keep individual pieces separated if there is a possibility of damage to the measurement areas during the various cleaning steps; (2) Clean samples for 5 min in an ultrasonic cleaner that contains a hot (65 to 85°C) 2% aqueous solution of a mildly alkaline (pH 7.5 to 10) detergent; (3) After ultrasonic cleaning, rinse samples thoroughly under warm running tap water for at least 5 s; (4) Rinse samples ultrasonically for 2 min in fresh deionized water to remove the last detergent residues; (5) Immerse in fresh analytical reagent grade methanol or isopropanol, and ultrasonically agitate for at least 30 s to remove the water from the samples; (6) Remove and dry samples until the alcohol has completely evaporated. If an air blast is used as an aid to drying, the air shall be oil-free, clean, and dry; and (7) Do not touch measurement area of the samples with bare fingers after cleaning.

9.3.4.3 Reinspect samples under 10× magnification for particulate matter on the surface. If particulates are found, repeat the cleaning step. Surface cleanliness is extremely important. Contaminants, such as plating salts and flakes of metal, may give erroneous indications of defects.

10. Test Procedure

10.1 Allow the appropriate PMA solution (see 9.1.1) to come to room temperature (approximately 10 min).

10.2 In the meantime, calibrate the microscope reticle at a convenient magnification in the 10 to 20× range using a stage micrometre or other device. Do not change magnification after calibration.

10.3 Fill a 10-mL beaker with deionized water and another 10-mL beaker with PMA solution.

10.4 Check PMA solution by applying a drop to a piece of copper. It should turn blue instantly. Alternately, test a deeply colored molybdenum blue. Watch the droplet for 20 to 40 s after its application, as specified in 10.8. Note all color changes. When multiple drops are applied, note location of any color changes. For example, the starting point of a burnish mark may be gouged, turning the droplet dark blue instantly, while a shallower burnish mark may not give any significant color change.

10.5 For samples with nickel underplates, hold sample inside top of bottle containing the hydrochloric acid in a working fume hood. Expose the measurement area to the HCl fumes for 3 to 5 s.

**Note 7—**If multiple samples are exposed at the same time, they should all be tested within 10 min of the acid fume exposure using a timer.

10.6 Place sample under microscope so that the measurement area is in a horizontal plane. If necessary, use a fixture or press into modeling clay to hold sample in place.

10.7 Use the applicator to apply a rounded droplet to the measurement area. For areas larger than the loop diameter, apply more drops to different parts of the measurement areas as needed. It is preferable not to have the drops run together.

**Note 8—**If a loop is used, clean any dried PMA or blue PMA solution from the loop using deionized water and tissue before continuing. Apply droplets to visible burnish marks and any other suspected defect areas. Make note if these fall outside the measurement areas.

10.8 Observe each droplet. Note any color changes within the following time limits: (1) 20 s for a loop diameter less than 0.6 mm; (2) 30 s for micropipets or for a loop diameter of 0.6 to 1.5 mm; and (3) 40 s for a loop diameter greater than 1.5 mm.

11. Examination and Evaluation

11.1 When base metals or alloys are attacked by PMA, Mo(VI) oxide is reduced to lower oxides forming intensely colored molybdenum blue. Watch the droplet for 20 to 40 s after its application, as specified in 10.8. Note all color changes. When multiple drops are applied, note location of any color changes. For example, the starting point of a burnish mark may be gouged, turning the droplet dark blue instantly, while a shallower burnish mark may not give any significant color change.

11.1.1 Unplated areas and worn spots will show up immediately upon application of the droplet as a color change from yellow to dark blue.

**Note 9—**The entire droplet will turn blue. This test does not pinpoint the exact location of the defect(s) within the droplet.

11.1.2 Smaller defects and cracks will tend to turn the droplet blue more slowly, but still within the time periods specified in 10.8.

11.1.3 Large pores may show up as minute blue spots within the droplet. The droplet may gradually change to green (small amounts of blue color in a yellow drop).

11.1.4 Thinly plated areas or areas with very small pores may not be seen immediately as blue spots, but the droplet may gradually change color from yellow to green.

**Note 10—**Observations 11.1.3 and 11.1.4 may or may not be significant, depending upon the reason for the test.

11.1.5 When the droplet is applied, distinct spots may turn blue instantly and then disappear; or they remain blue without turning the whole droplet blue. This appears to be due to a salt or metal flake on the surface of the examination area. To check into this, (1) Use the edge of the applicator to rub the spot gently. A flake will either break up or come loose and float around the droplet. The small particles may disappear by being dissolved by the phosphoric acid present in the PMA solution; (2) Rinse the sample thoroughly with deionized water, drying with methanol and then air. Expose the examination area to acid fumes and retest in accordance with 10.5-10.8. Flakes should be gone. Wear through or defects will show up normally if actually present.

12. Precision and Bias

12.1 Precision—This test method is essentially a pass/fail test to detect or identify large breaks in the plating primarily due to nonuniform coverage and mechanical damage, as well
as after wear testing. Deliberately damaged platings will be used to determine the precision of this test method.

12.2 Bias—The procedure in this test has no bias because the presence and size of gross defects are defined only in terms of this test method.

13. Keywords

13.1 electrodeposits; gross defects; mechanical damage; metallic coatings; porosity; testing for defects; wear through
Standard Practice for Applying Non-Electrolytic Conversion Coatings on Magnesium and Magnesium Alloys

This standard is issued under the fixed designation B 879; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε1 NOTE—Sections X2.2 and X2.4.2 were editorially updated in May 2003.

1. Scope

1.1 This practice covers a guide for metal finishers to clean and then provide a paint base for the finishing of magnesium and magnesium alloys using chemical conversion coatings. Where applicable (for example, aerospace) secondary supplementary coatings (for example, surface sealing) can be used (see Appendix X1).

1.2 Although primarily intended as a base for paint, chemical conversion coatings provide varying degrees of surface protection for magnesium parts exposed to indoor atmosphere either in storage or in service under mild exposure conditions. An example is the extensive use of the dichromate treatment (see section 5.2) as a final coating for machined surfaces of die cast magnesium components in the computer industry.

1.3 The traditional numbering of the coating is used throughout.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 The following documents form a part of this practice to the extent referenced herein.

2.2 ASTM Standards:

D 1732 Practices for Preparation of Magnesium Alloy Surfaces for Painting2

2.3 SAE Standard:

AMS 2475 Protective Treatments—Magnesium Alloys3

2.4 Military Specifications:

MIL-M-3171 Magnesium Alloy, Processes for Pretreatment and Prevention of Corrosion on4

DTD 911 (British), Protection of Magnesium-Rich Alloys Against Corrosion4

DTD 5562 (British), Clear Baking Resin for Surface Sealing Magnesium4

DTD 935 (British), Surface Sealing of Magnesium Rich Alloys4

3. Significance and Use

3.1 The processes described in this practice clean and provide a paint base for the finishing of magnesium and magnesium alloys. Service conditions will determine, to some degree, the specific process to be applied.

4. Reagents

4.1 The chemicals that are used to formulate and control the processing solutions are listed in Table 1. Commercial grade chemicals are satisfactory. The concentrations stated for chemicals that are normally supplied at less than a nominal 100 % strength are those typically available. Other strengths may be used in the proportions that yield the specified processing concentrations. Unless otherwise stated all solutions are made up using water.

5. Types of Coating

5.1 Chrome Pickle (Traditional Number 1) Treatment (See Practices D 1732):

5.1.1 With slight variations this treatment can be applied to all alloys and forms of magnesium. The treatment removes up to 15 µm of metal per surface, 30 µm per diameter. Therefore, it may not be applicable to machined surfaces with close tolerances. Parts with steel inserts may be processed, but some slight etching of the steel surface may occur.

5.1.2 The color, luster, and etch produced by the treatment will vary with the age and usage of the solution, alloy...
TABLE 1 Processing Chemicals

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetic acid glacial, (CH₃COOH)</td>
<td></td>
</tr>
<tr>
<td>Aluminum sulfate (Al₂(SO₄)₃·14H₂O)</td>
<td></td>
</tr>
<tr>
<td>Ammonium bifluoride (NH₄HF₂)</td>
<td></td>
</tr>
<tr>
<td>Ammonium hydroxide (NH₄OH), 30 %</td>
<td></td>
</tr>
<tr>
<td>Ammonium phosphate monobasic (NH₄H₂PO₄)</td>
<td></td>
</tr>
<tr>
<td>Ammonium sulfate (NH₄)₂SO₄</td>
<td></td>
</tr>
<tr>
<td>Ammonium sulfate (NH₄)₂SO₄·H₂O</td>
<td></td>
</tr>
<tr>
<td>Calcium chromate (CaCrO₄)</td>
<td></td>
</tr>
<tr>
<td>Calcium fluoride (CaF₂)</td>
<td></td>
</tr>
<tr>
<td>Calcium sulfate (CaSO₄·2H₂O)</td>
<td></td>
</tr>
<tr>
<td>Chromic acid (Cr₂O₇·2H₂O)</td>
<td></td>
</tr>
<tr>
<td>Ferric nitrate (Fe(NO₃)₃·9H₂O)</td>
<td></td>
</tr>
<tr>
<td>Glycolic acid (HOCH₂COOH), 70 %</td>
<td></td>
</tr>
<tr>
<td>Hydrofluoric acid (HF), 60 %</td>
<td></td>
</tr>
<tr>
<td>Magnesium fluoride (MgF₂)</td>
<td></td>
</tr>
<tr>
<td>Magnesium nitrate (Mg(NO₃)₂·6H₂O)</td>
<td></td>
</tr>
<tr>
<td>Magnesium sulfate (MgSO₄·7H₂O)</td>
<td></td>
</tr>
<tr>
<td>Manganese sulfate (MnSO₄·5H₂O)</td>
<td></td>
</tr>
<tr>
<td>Nitric acid (HNO₃), sp gr 1.42</td>
<td></td>
</tr>
<tr>
<td>Phosphoric acid (H₃PO₄), 85 %</td>
<td></td>
</tr>
<tr>
<td>Potassium fluoride (KF)</td>
<td></td>
</tr>
<tr>
<td>Potassium bifluoride (KHF₂)</td>
<td></td>
</tr>
<tr>
<td>Sodium bifluoride (NaHF₂)</td>
<td></td>
</tr>
<tr>
<td>Sodium bisulfate (NaHSO₄)</td>
<td></td>
</tr>
<tr>
<td>Sodium carbonate (Na₂CO₃)</td>
<td></td>
</tr>
<tr>
<td>Sodium dichromate (Na₂Cr₂O₇·2H₂O)</td>
<td></td>
</tr>
<tr>
<td>Sodium hydroxide (NaOH)</td>
<td></td>
</tr>
<tr>
<td>Sodium metasilicate (Na₂SiO₃ or Na₂SiO₄·4H₂O)</td>
<td></td>
</tr>
<tr>
<td>Sodium nitrate (NaNO₃)</td>
<td></td>
</tr>
<tr>
<td>Sulfuric acid (H₂SO₄), sp gr 1.84</td>
<td></td>
</tr>
</tbody>
</table>

The table lists various processing chemicals used in magnesium alloy treatments, including those for pickle, chromate, and phosphate treatments. The text explains the properties and uses of these treatments.

5.2 Dichromate (Traditional Number 7) Treatment (see Practices D 1732):

5.2.1 This treatment provides an improved paint base compared with the chrome pickle treatment, and for temporary protection on all standard alloys except, EK41A, HM31A, HM21A, HK31A, WE54, WE43, and M1A on which the coating does not form. The treatment causes no appreciable dimensional changes, is normally applied after machining, and is suitable for close clearance parts. Parts containing inserts of bronze, brass, steel, or cadmium plated steel should not be treated unless the dissimilar metals are masked or it is demonstrated that the treatment will not adversely affect them.

5.2.2 Coatings vary from light to dark brown depending upon the alloy. On AZ91C-T6 and AZ92A-T6 castings the coating is grey.

5.3 Galvanic Chromate (Traditional Number 9) Treatment (see Practices D 1732):

5.3.1 This treatment can be used for all alloys and is specifically used for those alloys which do not react or form satisfactory conversion coatings in other baths. The treatment requires no external current but utilizes the relatively high potential difference between suitably racked magnesium components and steel tank walls or other cathodes. As with the dichromate treatment, a prior immersion in acid fluoride solution is required to condition the magnesium surface. The galvanic chromate treatment causes no appreciable dimensional change and is normally applied after machining.

5.3.2 Properly applied coatings vary from dark brown to a dense black color depending on the alloy. The treatment is particularly useful for application to optical equipment requiring a nonreflective black coating.

5.4 Chromic Acid Brush-On (Traditional Number 19) Treatment:

5.4.1 This treatment can be applied to parts that require touch up. It is generally used in refinishing procedures or where parts or assemblies are too large to be immersed. It is effective on most alloys and causes negligible dimensional changes.

5.4.2 Coatings produced by this treatment can vary from a brassy iridescence to a dark brown depending upon treatment time. Prolonged treatment produces powdery coatings. For best adhesion, dark brown coatings are preferred.

5.5 Chromate Treatment (see DTD 911):

5.5.1 This treatment is suitable for all magnesium alloys. The treatment causes no dimensional change and is normally applied after machining. The pickling procedures and the composition of the treating solution generally vary with the alloy being processed.

5.5.2 The coating will vary from dark brown to light reddish-brown depending on the alloy.

5.6 Chrome-Manganese Treatment:

5.6.1 This treatment provides an improved paint base compared with the chrome pickle treatment and protection on all standard alloys except EK41A, HM31A, HM21A, HK31A, and M1A on which the coating does not form. The treatment causes no appreciable dimensional change, and is normally applied after machining. It is suitable for close clearance parts. Parts containing inserts of bronze, brass, steel, or cadmium plated steel should not be treated unless the dissimilar metals are masked or it is demonstrated that the treatment will not adversely affect them.

5.6.2 The bath generally gives dark brown to black films on both cast and wrought magnesium alloys. Treatment of aluminum containing alloys may require bath temperatures above 50°C.

5.7 SemiBright Pickle (Traditional Number 21) Treatment:

5.7.1 This treatment provides a semibright silvery surface on magnesium parts that prevents tarnishing and corrosion for indoor storage up to six months in non-air-conditioned environments. Extended storage times can be obtained by using air conditioning. This process causes negligible dimensional change. It is a simple, economical way to apply an attractive shelf-life finish and is a good base for clear lacquers. The treatment greatly reduces or eliminates "filiform or worm-tracking" corrosion usually experienced when clear paints are used directly over polished metal surfaces.

5.8 Phosphate Treatment:

5.8.1 Phosphate treatments can provide a satisfactory paint base on magnesium for many applications when it is necessary to avoid the use of chromates. Commercial iron phosphate treatments applied by spray or dipping have been successfully...
used on magnesium die castings for automotive and other consumer product applications. The suitability of a particular phosphatizing process for magnesium should be verified by testing. Iron phosphate treatments containing nickel or copper salts as accelerators are detrimental to the corrosion resistance of magnesium and should not be used.

5.8.2 Phosphate treatments do not provide interim stand-alone protection against atmospheric oxidation and tarnish equal to that provided by some chromate conversion coatings.

6. Part Preparation

6.1 Cleaning—General:
6.1.1 Before considering the use of solvent degreasing, consult federal and state safety and environmental laws and regulations. Many of the commonly used solvents are now being banned from use. Exposure to their vapor (VOC) is being strictly regulated for health, safety, and environmental reasons. Obtain current safe exposure levels for various solvents before use. Follow all federal, state, and local regulations for the disposal of solvents.

6.1.2 Solvent Cleaning—Grease or oil may be removed by means of vapor degreasing, ultrasonic cleaning, solvent washing, or an emulsion cleaning process that utilizes a mineral oil distillate and an emulsifying agent. Chlorinated solvents, petroleum spirits, naphths, lacquer thinner, and similar solvents that do not attack magnesium may be used. Methyl alcohol (CH₃OH) should not be used because it may react with the magnesium surface.

6.1.3 Mechanical Cleaning—Mechanical cleaning may consist of sand, shot, pumice, grit or vapor blasting, sodium carbonate slurry, sanding, hard bristle brushing, grinding and rough polishing. Sand, shot, or grit blasting leaves surface contamination that will greatly increase the corrosion rate of the magnesium on exposure to salt water or humid environment. If these methods are used, specific pickling procedures must be employed after blasting (see 6.4.2).

6.1.4 Alkaline Cleaning—Cleaning prior to application of treatments other than the chrome pickle treatment (see 5.1), when used for protection during shipment or storage, should be done in an alkaline cleaner recommended for steel or in a cleaning solution as specified in 6.1.4.1. Maintain the solution pH above 8.0. Alkaline cleaning prior to the application of the chrome pickle treatment (see 5.1), when used for protection during shipment and storage only, may be omitted provided the parts are free of grease, oil, and other deleterious deposits at the time of application. Alkaline cleaning solutions containing more than 2% sodium hydroxide will etch ZK60A, ZK60B, and some other magnesium alloys producing a change in dimensions. If such a dimensional change is undesirable, use cleaners with lower alkali content.

6.1.4.1 Alkaline cleaning may be carried out in solutions of proprietary cleaners. In this case the operating conditions should be as specified by the supplier. In no case should a cleaner having pH lower than 8.0 be used. Most recommended cleaners are used by simple immersion. After alkaline cleaning, rinse parts thoroughly in cold running water. No water breaks should be observed in the rinse.

6.1.5 Electrolytic Cleaning—Use of anodic current for cleaning is not generally recommended because of the possible formation of oxide films, pitting of the magnesium surface, or both. However, electrolytic cleaning using cathodic current at 1 to 4 A/dm² may be carried out in properly formulated cleaners.

6.2 Graphite Lubricant Removal:
6.2.1 Remove graphite-based lubricants from hot formed magnesium sheet parts by soaking the parts for 10 to 20 min in 100 g/L sodium hydroxide maintained at 88 to 100°C. The pH should be above 13.0. Add wetting agent (0.75 g/L), if needed, for the removal of heavy films of mineral oil. Then rinse parts thoroughly in cold water and immerse for 3 min in a chromic-nitrate pickle as specified in 6.5.2. Repeat the cycle until all parts are clean.

6.2.2 Because of the difficulty of removing graphite from chrome pickled sheet, such sheet should not be used for forming unless the chrome pickle is removed as outlined in 6.3 before forming.

6.3 Previously Applied Chemical Finishes:
6.3.1 Magnesium base alloys are often supplied with a chrome pickle treatment to protect them during shipment, storage, and machining. The coating from this treatment remaining on unmachined areas will impair the film produced by any subsequent chromate treatment and therefore must be removed.

6.3.2 Previously applied coatings may be removed with the alkaline cleaners recommended in 6.1.4.

6.3.3 If the finish is difficult to remove, immerse the part in the chromic acid pickle given in 6.5.1. Alternate immersion in the alkaline cleaner and the chromic acid pickle may be required to remove aged finishes. Rinse well in water between acid and alkaline pickling.

6.3.4 The chromic acid brush-on treatment (see 5.4) may be applied over the chrome pickle finish or over previously applied brush-on coatings without removing the previously applied coating.

6.4 Acid Pickling:
6.4.1 General pickling to remove oxide layers, old chemical finishes, burned-on drawing and forming lubricants, and other water insoluble or non-emulsifiable substances is preferably carried out using a chromic acid type pickle as described in 6.5.1 or 6.5.2.

6.4.2 Pickle sand and permanent mold castings that have been mechanically cleaned as described in 6.1.3 in the sulfuric acid pickle described in 6.5.4 or in the nitric-sulfuric acid pickle described in 6.5.5. The pickling operations should be of such duration that 50 µm of surface is removed whenever dimensional tolerances permit.

6.4.3 Magnesium sheet and plate up through 12.5 mm thick should have the mill scale removed to ensure a clean surface for the subsequent treatments. Where dimensional tolerances permit, remove 12 to 25 µm per metal surface. The acetic-nitrate pickle described in 6.5.8, or an equivalent solution, should be used.

6.4.4 Pickle die castings that are to be pickled for maximum corrosion resistance and a more uniform surface for applying subsequent coatings in the chromic-nitric-hydrofluoric acid pickle described in 6.5.6 or the phosphoric acid pickle described in 6.5.7.
6.5 Acid Pickling Solutions:

6.5.1 Chromic Acid Pickling—Chromic acid pickling causes no dimensional change and therefore may be used on parts with close tolerances. It may be used for removal of previously applied chemical finishes (see 6.3.3). It is satisfactory for the removal of surface oxidation and corrosion products and for general cleaning. It is not satisfactory for the removal of sand or effects of blasting and should not be used on parts that contain copper inserts unless the copper can be completely masked off. Excessive concentrations of chloride, sulfate, and fluoride must be avoided in the solution since these will cause etching or film formation rather than cleaning. The solution contains 180 g/L of chromic acid. Treatment time is 1 to 15 min at 88 to 95°C. Lower temperatures are permissible provided the treatment time is suitably increased.

6.5.2 Chromic-Nitrate Pickle—The chromic-nitrate pickle is not generally used for the removal of corrosion products of surface oxidation but may be substituted for the chromic acid pickle described in 6.5.1. It is mainly used for the removal of burned on graphite lubricants as described in 6.2. It is not suitable for the removal of sand from castings or for correcting the effects of blasting. It should not be used for parts containing copper inserts unless the inserts can be completely masked off. The solution contains 180 g/L chromic acid and 30 g/L sodium nitrate. Immersion time is 2 to 20 min at 15 to 30°C which normally removes 12.5 µm of metal/surface. Lack of chemical action and a pH of 1.7 or higher indicates depletion of the bath. The addition of sufficient chromic acid to restore a pH of 0.5 to 0.7 will rejuvenate the solution. After three to four replenishments, the additions of chromic acid required to rejuvenate become excessive, and the bath should be discarded and replaced.

6.5.3 Nitric Acid Pickle—Nitric acid pickle is used to clean and brighten alloys on which chromate films are not desired. The solution contains 70 mL/L nitric acid. Immersion time is 30 to 60 s at 20 to 27°C.

6.5.4 Sulfuric Acid Pickle—The sulfuric acid pickle is used on magnesium sand castings to remove the effects of blasting operations. Use the pickle before any machining operations since the amount of metal removed is likely to exceed permissible tolerances. The solution contains 32 mL/L sulfuric acid in water. Immersion time is 10 to 15 s at 20 to 32°C or as required to remove approximately 50 µm of metal per surface.

6.5.5 Nitric-Sulfuric Acid Pickle—As an alternative for the sulfuric acid pickle (see 6.5.4) the nitric-sulfuric acid pickle may be used. The solution contains 80 mL/L nitric acid and 20 mL/L sulfuric acid. Conditions of operation and time of immersion are the same as for the sulfuric acid pickle.

6.5.6 Chromic-Nitric-Hydrofluoric Acid Pickle—This pickle may be used on any casting, but is especially effective on die castings. It will remove metal at approximately 12.5 µm per minute per surface. The solution contains 280 g/L chromic acid, 25 mL/L nitric acid, and either 7.5 mL/L HF or 6.5 g/L ammonium bifluoride. Immersion time is 0.5 to 2 min at 21 to 32°C.

6.5.7 Phosphoric Acid Pickle—The phosphoric acid pickle may be used for pickling all castings, particularly die castings. It is especially effective at removing segregated aluminum from the surface of AZ91A, AZ91B, and AZ91D alloys. It has been used for some wrought alloys, such as HK31A. Dip the parts for 10 to 15 s in 50 to 85 v/o phosphoric acid and allow to drain and react in air for 30 to 60 s. Without rinsing, dip in sodium hydroxide 80 to 120 g/L for 30 s at 20 to 25°C. Follow with a cold-water rinse. Sodium hydroxide corrects the powdering effect of the phosphoric acid. Metal loss is 12.5 to 25 µm. Contain the phosphoric acid solution in tanks with rubber, glass, ceramic or lead lining.

6.5.8 Acetic-Nitrate Pickle—The acetic-nitrate pickle is suitable for the removal of mill scale and other surface contamination from sheet to ensure maximum effectiveness of the protective coating. This solution can be used on wrought forms and on solution heat treated castings. Castings in the as-cast condition (-F) or in the solution heat treated and aged condition (-T6) should not be pickled in this solution because a loose grey smut forms. Treat castings in the -F or -T6 conditions in the chromic-nitric-hydrofluoric acid pickle (see 6.5.6). For best results, the pickle should be allowed to remove 10 to 25 µm of metal per surface. The treatment may not therefore be suitable for the treatment of parts with close tolerances. The solution contains 200 mL/L acetic acid and 50 g/L sodium nitrate. Immersion time at 20 to 30°C is 0.5 to 1 min.

6.5.9 Glycolic-Nitrate Pickle—In cases where spray pickling is used or fumes are a problem, the glycolic-nitrate pickle may be substituted for the acetic-nitrate pickle. This will also reduce acid loss from vaporization. The solution contains 250 mL/L commercial glycolic acid, 200 g/L magnesium nitrate and 37.5 mL/L nitric acid. Immersion time at 20 to 30°C is 3 to 4 min.

6.5.10 Pickling Prior to Spot Welding—A special cleaning and pickling sequence is used to prepare parts for spot welding. The treatment results in a surface that has a very low surface resistance and that will retain this low value for some time. The sequence consists of: alkaline cleaning and rinsing as in 6.1.4; alkali neutralization in a dilute acid solution such as 0.5 to 1.0 volume % sulfuric acid or a 7.5 to 15 g/L sodium acid sulfate solution; treatment in a chromic-sulfuric acid pickle containing 180 g/L chromic acid and 0.5 mL/L sulfuric acid. Immersion time is 3 min at 20 to 30°C.

6.5.11 Hydrofluoric-Sulfuric Acid Pickle—This pickle is used (particularly in brightening die castings) when the 12.5 µm metal loss caused by the phosphoric acid pickle cannot be tolerated. This solution contains 15 to 20 volume % hydrofluoric acid and 5 volume % sulfuric acid. Immersion time at room temperature is 2 to 5 min. After the dip, rinse the parts in cold water.

6.6 Fluoride Anodizing Cleaning (see MIL-M-3171 and DTD 911):

6.6.1 Fluoride Anodizing Cleaning—This electrolytic process was developed for the cleaning of magnesium parts prior to conversion coating. While the process is normally used to clean raw sand castings, it is also particularly useful for the
cleaning of components because the process does not significantly alter dimensions.

6.6.2 The electrolytic bath contains 150 to 250 g/L ammonium bifluoride in a tank lined with non-conducting hard rubber or suitable plastic material. A power source is required capable of delivering a progressively increasing ac output up to a maximum of 120 V. Current requirements will vary depending upon the size of the installation, but a minimum of 150 A/m² on one electrode, when the bath is being used to capacity is recommended. The power is applied via normal bus bars and clamps. All sections of fixing clamps immersed in the electrolyte must be of magnesium alloy, and parts to be cleaned should not contain any dissimilar metal inserts.

6.6.3 Parts are fixed in good electrical contact with the bus bars and located in the tank so that they are at least 0.25 m below the electrolyte surface and there is approximately equal surface area on each electrode. Alternating current is applied and the voltage progressively increased until 120 V is reached. Current flow is heavy at first but rapidly diminishes as the surface impurities are removed and an insulating coating of magnesium fluoride is formed. The treatment is complete when the current density falls below 50 A/m².

6.6.4 Certain alloys, however, may be etched at 120 V, particularly if the ammonium bifluoride concentration is towards the lower end of the range. For these alloys the process should be terminated at about 90 V. The magnesium surface, when satisfactorily anodized, should be a uniform gray-white color.

6.6.5 Thoroughly rinse parts in cold water after treatment, and strip the fluoride film in hot chromic acid as detailed in 6.5.1. If fluoride anodizing is followed by an hard anodizing process, such as the HAE treatment, removal of the fluoride film is not necessary.

7. Application of Coatings

7.1 Chrome Pickle (Traditional Number 1) Treatment (see Practices D 1732):

7.1.1 Cleaning of parts to receive this treatment should be in accordance with the procedures outlined in Section 6.

7.1.2 Procedures for Wrought Parts—The solution for treating wrought parts contains 180 g/L sodium dichromate and 190 mL/L nitric acid. The bath is operated at 20 to 45°C and immersion times can range from 30 s to 2 min depending on the activity of the solution. Immerse the part and agitate in the solution under the conditions indicated above. Following treatment, drain the parts for at least 5 s and then rinse thoroughly in cold running water. Drying may be facilitated by a dip in hot water or with a hot air blast. Avoid heating above 200°C. Under some conditions, the coating may be applied by spraying the solution over the part rather than immersing the part in the solution.

7.1.3 Procedures for Sand, Permanent Hold and Die Castings—Chrome pickling of the widely used sand, permanent mold and die castings of high aluminum content (AZ91 types) requires modified solutions and procedures. The aluminum-rich phase present in these alloys can cause problems ranging from failure to react to selective reaction leaving a loose dark aluminum-rich smut deposit on the surface. Preventive action includes the addition of fluoride and sulfate to the solution, adjustment of temperature and treatment times, and the use of pretreatment pickles (chromic-nitric-hydrofluoric, phosphoric, or hydrofluoric-sulfuric) where necessary.

7.1.3.1 The solution for treating these types of castings contains of sodium bifluoride 15 g/L, sodium dichromate 120 g/L, aluminum sulfate 10 g/L, nitric acid 90 mL/L. Operating temperatures can range from 21 to 60°C. Preheat die castings, permanent mold, and aged sand castings by dipping in hot water (80 to 95°C) followed by immediate immersion in the treating solution. A 10 s immersion is sufficient if the bath is operated at 50 to 60°C. Lower temperatures require longer treating times.

7.1.3.2 Excessive treatment time will produce a powdery coating. Failure to preheat the part in hot water may result in failure of the coating to form. If a separate solution for die cast, permanent mold, and aged sand castings is not available, the bath used for wrought parts may be used. Sand castings in the solution heat treated condition may be chrome pickled in this solution at room temperature with no preheating. After treatment, drain the parts and rinse for at least 5 s and then rinse thoroughly in cold water. Drying may be facilitated by a dip in hot water.

7.1.3.3 Optional Hydrofluoric Sulfuric Pre-Pickle—Hydrofluoric acid 30 to 40 volume %, sulfuric acid 5 volume %, 2 to 5 min, at room temperature, followed by a cold water rinse.

7.1.3.4 Dilute Chrome Pickle Treatment—The hexavalent chromium content of this bath is about 20 % lower than the commonly used chrome pickle baths. This results in lower initial cost, less dragout loss, and lower effluent treatment costs. The paint base created is equivalent to that of a standard chrome pickle. The dimensional change is about 2 µm per surface for a treatment time of 20 to 30 s at 20 to 30°C. The bath contains: sodium bifluoride 2.5 g/L, sodium dichromate 35.0 g/L, magnesium sulfate 3.0 g/L, and nitric acid 30 mL/L.

7.1.4 Precautions—Chrome pickle baths may remove as much as 15 µm of metal per surface during treatment. Therefore, they can not be used on machined surfaces unless tolerances will permit or allowances are made. Magnesium parts containing steel inserts can be given the chrome pickle treatment. An excess of nitric acid or buildup of nitrate salts in the solution can result in the formation of bright brassy coatings as described in 5.1.2.

7.1.5 Brush Application—Parts too large to be immersed may be carefully brushed with a generous amount of fresh pickle solution made as specified in 7.1.2 or 7.1.3. The solution should remain on the surface for at least one minute while brushing is continued and washed off immediately thereafter with cold running water. The coating formed by this procedure is less uniform in color than that produced by immersion but is an equally good paint base.

7.1.5.1 Powdery coatings are not good paint bases and indicate poor rinsing or failure to keep the surface wet with solution during the one minute treatment time. The area being treated must be continually brushed with solution so that the surface never dries.
7.1.5.2 Brush application is suitable for touchup of all types of treatments that have been damaged. This type of coating is also best for touchup of bare areas before making an electrical bonding joint and where no primer can be applied.

7.1.6 Parts, such as tanks, that must be treated internally and that take a long time to fill and empty may be treated with the chrome pickle solution for wrought parts diluted with an equal quantity of water. The treatment time should be sufficient to produce a complete coating on all areas. The pickle bath may be diluted in a similar manner to increase treatment times to fit the time cycles of automated processing equipment.

7.1.7 Control of the Chrome Pickle Treatment—Control the process in accordance with the principles outlined in X1.1.

7.2 Dichromate (Traditional Number 7) Treatment (see Practices D 1732 and AMS 2475):

7.2.1 Close control is required when applying this treatment to AZ31B-H24. Where maximum corrosion resistance is required on magnesium alloy sheet, use acid pickling as described in 6.5.2, 6.5.7, or 6.5.8.

7.2.2 Hydrofluoric Acid Treatment—After parts have been cleaned as described in Section 6, give them a hydrofluoric acid treatment. The solution contains 300 mL/L hydrofluoric acid operated at 20 to 30°C for 30 s to 5 min. This both cleans and activates the surface. Immerse parts made of AZ31B alloy for 30 s. Immerse all other wrought and cast alloys for 5 min. Following treatment, give the parts a thorough rinse in cold running water. Drag-over of fluoride ion will render the dichromate solution inoperative.

7.2.3 Acid Fluoride Treatment—Use this treatment for all parts containing aluminum inserts, rivets, etc. and as an alternative treatment to the hydrofluoric acid treatment (see 7.2.2). It is the preferred treatment for AZ31B and AZ31C alloy parts. It is generally more economical and safer than the hydrofluoric acid treatment, however, it will not remove the dark smudge that forms on some castings after sandblasting and pickling. If a dark smudge forms on the casting, use the hydrofluoric acid treatment in 7.2.2.

7.2.3.1 The acid fluoride solution contains 50 g/L of sodium, potassium, or ammonium bifluoride. It is operated at 20 to 30°C for a minimum of 5 min. After treatment, rinse the parts thoroughly in cold running water. See 7.2.2 on dragover of fluoride.

7.2.4 Dichromate Treatment—The solution consists of 120 to 180 g/L of sodium dichromate and 2.5 g/L of calcium or magnesium fluoride operated at boiling point for 30 to 45 min. After treatment, rinse the parts thoroughly in cold running water and dip in hot water or blow dry with hot air to facilitate drying. Apply required paint coatings as soon as practical after treated parts are thoroughly dry. Because ZK60A accepts the coatings more easily, a 15 min treatment is adequate for this alloy.

7.2.5 Control of Dichromate Treatments—Control the process as outlined in X2.2.

7.3 Galvanic Chromate (Traditional Number 9) Treatment (see Practices D 1732):

7.3.1 Acid Fluoride Treatment—After parts have been cleaned as described in Section 4, immerse them for 5 min in a solution that contains 50 g/L sodium, potassium or ammonium bifluoride at 20 to 30°C. Alternatively, the hydrofluoric acid solution detailed in 7.2.2 may be used. After treatment rinse the parts thoroughly in cold running water.

7.3.2 Galvanic Chromating—The chromating solution, that is contained in a steel tank, contains 30 g/L ammonium sulphate, 30 g/L sodium dichromate, and 7.8 mL/L ammonium hydroxide, maintained at 50 to 60°C. The magnesium parts are electrically connected to the tank by an external circuit containing an Ammeter and rheostat. The magnesium parts are immersed in the solution and galvanically chromated at a current density of 21.5 to 105 A/m². A total of 750 to 1600 amperes per square metre of magnesium is usually required to develop a uniform coating. After treatment rinse the parts thoroughly in cold running water followed by hot water to facilitate drying.

7.3.3 Control of the Galvanic Chromate Treatment—The process should be controlled as outlined in X2.5.

7.4 Chrome-Manganese Treatment (see DTD 911):

7.4.1 Cleaning of parts to receive this treatment should be in accordance with the procedures outlined in Section 6. It is however advantageous to precede the chromate treatment with an alkaline clean and, after rinsing, to immediately transfer parts, while still wet, to the chrome-manganese bath.

7.4.2 The solution contains of 100 g/L sodium dichromate, 50 g/L manganese sulphate, and 50 g/L magnesium sulphate. The bath is normally operated at room temperature (20 to 30°C) with immersion times of 1.5 to 2 h. The bath may be operated at higher temperatures to shorten treatment time, for example, 30 min at 50 to 60°C, 15 min at 70 to 80°C, and 10 min at 90 to 95°C. Satisfactory chromating of the magnesium-aluminum series alloys, for example, AZ91, AZ92, AZ61, and AZ31, may require elevated temperature bath operation. After treatment thoroughly rinse the parts in cold then warm water and dried.

7.4.3 Control of the Chrome Manganese Treatment—Control the process as outlined in X1.4.

7.5 Semi-Bright Pickle (Traditional Number 21) Treatment:

7.5.1 Any of the usual cleaning and degreasing processes used for magnesium alloys can be used. However, the bright appearance given by this treatment can be enhanced by giving the part a nitric acid pickle (see 6.5.3) or mechanical cleaning.

7.5.2 The solution consists of 180 g/L of chromic acid, 40 g/L ferric nitrate, and 3.5 g/L potassium fluoride. The bath is operated at 18 to 38°C. Lower bath temperature reduce staining in air during transfer from pickle to rinse. Longer pickling times yield maximum brightness. Immersion times are 15 s to 3 min. The solution, castings, or both, must be agitated during treatment. After removal from the pickling solution, rinse immediately in cold running water followed by a hot water rinse to speed drying.

7.5.3 If stains develop due to the length of time between pickling and rinsing, go directly from the pickling tank to a tank containing the following solution: 50 g/L of sodium metasilicate, sodium carbonate, or sodium hydroxide. Immerse the parts for 30 s to 1 min in the hot solution (70 to 80°C). After removing the parts from the dip solution, rinse thoroughly in cold running water followed by a hot water rinse to aid in drying.
7.6 Phosphate Treatment:

7.6.1 Any of the de-greasing processes used for magnesium alloys are satisfactory, followed by an alkaline cleaning.

7.6.2 The solution consists of 120 g/L ammonium phosphate monobasic, 30 g/L ammonium sulfite, and 16 mL/L ammonium hydroxide. The bath is operated at room temperature with mild mechanical agitation. Immersion time is 1.5 to 2 min or until gassing stops. Immediately rinse the treated parts in cold water after removal from the bath and before the surface dries.

7.7 Chromic Acid Brush-On (Traditional Number 19) Treatment:

7.7.1 The brush-on technique for the chromic acid treatment is less critical than for the chrome pickle treatment. While the treatment solution will not cause problems if trapped between faying surfaces, such traps should be avoided in design or properly sealed to prevent entrance of corrosive liquids in the service environment. The lower concentrations used reduce the toxicity and waste disposal hazards.

7.7.2 Pretreatment—Normal methods of removing oil, grease, etc. as given in Section 6 are used prior to treatment.

7.7.3 Application—The solution contains of 10 g/L of chromic acid and 7.5 g/L of calcium sulfate and is used at 20 to 30°C for the times indicated in 5.7.4. Add the chemicals in the order given above, and agitate the solution vigorously, mechanically, or by air agitation, for at least 15 min before use.

7.7.4 Brush-On Application—Proper application requires that the parts be kept wet with the brush-on solution for a sufficient length of time, usually 1 to 2 min, so that a brown film is produced. Then rinse the parts in cold running water and dry either in an oven or hot air blast. In no case should the parts be rinsed in hot water.

7.7.4.1 Unlike application of the chrome pickle treatment by brush-on techniques, the time between treatment and rinsing is not critical. In fact, if running water is not available, the rinsing step can be eliminated without affecting the coatings.

7.7.4.2 The paint base properties are substantially equal to those of the chrome pickle treatment. The color of the coating may vary from pale brown to almost black, depending on the alloy and treatment conditions. The coating may be applied over previously applied thin films of all the chromate treatments.

7.7.5 Operating Cautions—Coatings applied by this method are not adequate for interfaces used in electrical bonding but are satisfactory for use on entrapment areas of magnesium assemblies. Parts processed with this treatment require painting or sealing before exposure to outdoor, overnight atmospheric conditions. The color of the coatings can vary from brassy iridescent to dark brown by altering the treatment time. Up to 1 min of treatment time produces brassy coatings, 2 to 3 min produces a dark brown coating. Prolonged treatment produces powdery coatings. The dark brown coatings are preferred for best paint adhesion.

7.8 Chromate Treatment—Proprietary Iridite 15:

7.8.1 Alloys Containing More Than 1% Aluminum—Clean parts as described in Section 6. For alloys containing 3.5% aluminum or less, such as AZ31, the chromic-nitrate pickle (see 6.5.2) should follow alkaline cleaning. For those alloys having an aluminum content greater that 3.5%, such as AZ61, AZ81, and AZ91, use the chromic-nitrate-hydrofluoric acid pickle (see 6.5.6). If mill pickled stock is to be treated, immerse parts in the pickle for 15 to 30 s. If unpickled or cast materials are to be processed, the immersion time should be 2 to 3 min.

7.8.1.1 The use of the bath should be strictly in accordance with the manufacturer’s instructions.

8. Keywords

8.1 chemical conversion coatings; chromates; cleaning magnesium; preparation for painting; protection; surface sealing

APPENDIXES

(Nonmandatory Information)

X1. SURFACE SEALING OF MAGNESIUM ALLOY COMPONENTS

X1.1 For severe environmental conditions such as near the ocean or in high humidity areas, the use of the surface sealing process greatly improves the resistance of conversion coated magnesium to corrosion and enhances the performance of the entire protection system.

X1.2 Surface Sealing (see DTD 5562 and DTD 935)—Surface sealing is a technique developed to produce a thin, flexible, extremely water-impermeable resin coating on all suitably treated magnesium alloy surfaces. This technique should not be confused with “impregnation” techniques used to seal metal porosity. The coating should be applied as soon as possible after the conversion coating is formed.

X1.3 Coating Procedure:

X1.3.1 Prebake the components at 200 to 220°C for 30 min or until this temperature has been maintained for at least 10 min.

X1.3.2 Allow to cool to 60°C then spray with, or preferably dip in, surface coating resin to DTD 5562 specification. If dipping is used hold the part in the resin for the first coat until all bubbling.

X1.3.3 Allow to air dry for 15 to 30 min. Ensure uniform drainage and remove any runs or tears that form with a small brush.

X1.3.4 Bake at 200 to 220°C for 10 to 15 min.

X1.3.5 Remove any drips or tears that may have formed with a sharp knife or sand paper taking care not to damage the conversion coating.
X1.3.6 Repeat X1.3.2-X1.3.5 twice more making three coats in all.

X1.3.7 Bake the final coat at 200 to 220°C for 45 min, or if the part is large, until the temperature of the part has been maintained for 45 min.

Note X1.1—The complete resin coating is approximately 25 µm thick when applied over chromate chemical conversion coatings. Application onto electrolytic conversion coatings (see Practices D 1732) produces significantly less build-up due to the very porous nature of the anodic film.

Note X1.2—Prebaking the component is essential in order to ensure that all surface moisture and moisture in surface porosity is boiled off. Dipping is preferred to spraying since it leads to a complete and more uniform resin coverage, and dipping the component while still warm assists the resin to permeate and fill the surface cracking or crazing that is present in the conversion coating.

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**X2. CONTROL OF TREATMENT PROCESSES**

Note X2.1—Only A & R grade chemicals should be used.

X2.1 **Chrome Pickle Treatment:**

X2.1.1 **Sodium Dichromate Determination**—Determine the sodium dichromate concentration by the following procedure or other recognized analytical procedure:

X2.1.1.1 Pipet a 1-mL sample of the chrome pickle solution into a 250-mL beaker containing 150 mL of distilled/deionized water. Add 5 mL of concentrated hydrochloric acid and 5 g of potassium iodide. Mix well. Allow at least 2 min for the reaction to run to completion. Stir and titrate the free iodine with 0.1 N sodium thiosulfate solution until the yellow color is almost gone. Add several drops of starch indicator solution. Continue the titration until the purple indicator color disappears. (Warning—Do not add the starch indicator solution until the brown color is almost completely gone or inaccurate results will be obtained. The final color will range from pale green to blue in color.)

X2.1.1.2 Calculation:

\[
\text{mL} \times 0.1 \text{ N sodium thiosulfate} \times 4.9745 = \text{g/L sodium dichromate} \quad (X2.1)
\]

X2.2 **Nitric Acid Determination**—Pipet a 1-mL sample of the chrome pickle solution into a 250-mL beaker containing 50 mL of distilled water. Standardize a glass electrode equipped pH meter with a buffer solution of approximately 4.0 and immerse the glass electrode system in the solution. Stir and titrate with 0.1 N sodium hydroxide to a pH of 4.00 to 4.05.

X2.2.1 Calculation:

\[
\text{mL} \times 0.1 \text{ N sodium hydroxide} \times 4.281 = \text{g/L nitric acid} \quad (X2.2)
\]

X2.3 **Control Limits of Chrome Pickle Bath**—Depletion of the solution is indicated by paleness of color, shallowness of etch, and the slowness of action on the metal. Since insufficient exposure to air between removal from the bath and subsequent rinsing can also cause paleness, the two causes should not be confused.

X2.3.1 If the alloys being processed do not contain aluminum, the bath should be regenerated no more than once. For other alloys, it may be generated up to seven times. Regeneration should be carried out when the nitric acid content has been reduced to or near 60 mL/L of nitric acid. The nitric acid content should be reduced each time the bath is regenerated according to the following schedule:

<table>
<thead>
<tr>
<th>Run Number</th>
<th>Sodium Dichromate, g/L</th>
<th>Nitric Acid, mL/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>180</td>
<td>190</td>
</tr>
<tr>
<td>2</td>
<td>180</td>
<td>165</td>
</tr>
<tr>
<td>3</td>
<td>180</td>
<td>140</td>
</tr>
<tr>
<td>4 to 7</td>
<td>180</td>
<td>110</td>
</tr>
</tbody>
</table>

X2.3.2 If the solution is used to produce a protective coating for shipment and storage only, it may be regenerated up to 30 to 40 times, as long as a readily visible protective coating is formed.

X2.4 **Problems and Explanations**—The following operational problems may be encountered:

X2.4.1 Brown, non-adherent, powdery coatings:

X2.4.1.1 The part was in the air too long before rinsing.

X2.4.1.2 The ratio of acid concentration to sodium dichromate may be too high.

X2.4.1.3 Solution may be too hot. This may occur as the result of rapidly processing a large number of parts through a small volume of solution. Cool the solution, reduce the rate of processing, or use a larger volume of solution.

X2.4.1.4 Parts are not properly cleaned. Residual oil on the surface will cause powdery coatings.

X2.4.1.5 The solution has been regenerated too often. Excess nitrate concentrations can cause powdery coatings.

X2.4.2 Grey, nonadherent coatings on castings (Warning—This powdery coating may flash violently on impact, abrasion, or friction.):

X2.4.2.1 Use bifluoride modified treatment outlined in 7.1.3.1 instead.

X2.4.2.2 Part may have been excessively treated by being kept in the bath too long.

X2.4.2.3 If parts are to be scrapped, they should be rinsed thoroughly in cold water, dried and dipped in machine or motor oil. (For additional safety, the coating may be removed as described below before coating with oil.) If dimensional changes are not too severe for salvage, the coating may be removed by immersing the parts in 10 to 20 volume % hydrofluoric acid solution for 5 to 10 min.

X2.5 **Dichromate Treatment:**

X2.5.1 **Hydrofluoric Acid Determination**—The hydrofluoric acid is depleted very slowly in use. It should not be allowed to fall below 10 % HF as determined by titration for free acid with 1 N NaOH and phenolphthalein indicator. A solution weaker than 10 % will attack magnesium severely. A titer of 10 to 20 mL of 1 N NaOH for a 2-mL sample roughly corresponds
to 10 to 20 % by weight of HF. Pipet a 2-mL sample into approximately 100 mL of distilled water and titrate immediately. Use plastic pipet and beakers to prevent inaccurate results because of reaction of the HF with glass.

X2.5.2 Bifluoride Determination—If the acid fluoride treatment is used instead of the hydrofluoric acid treatment, control the solution by titration with sodium hydroxide. Maintain the acid fluoride content such that a 100-mL sample of the bath requires 45 to 55 mL of 1 N NaOH to produce a pink color using phenolphthalein indicator.

X2.5.3 Control of Dichromate Bath—The concentration of sodium dichromate in the bath can be determined by the method outlined in X2.1. Maintain the dichromate concentration between 120 and 180 g/L. Maintain the pH of the solution between 4.1 to 5.5 by the addition of chromic acid. Make the pH adjustment before the dichromate analysis is done.

X2.5.3.1 When treating AZ31B alloy, the pH range must be narrowed to 4.1 to 5.2. Dissolve the chromic acid in warm water to form a 10 % solution. This solution can then be used in the pH adjustment described below for making additions to the tank.

X2.5.3.2 To Make the pH Adjustment—Standardize a pH meter equipped with a glass electrode using a buffer solution of approximately 4.0. Using a convenient size measured sample, adjust the pH of the sample to 4.1 by adding measured amount of the 10 % chromic acid solution described above:

\[ \text{(mLs 10 % CrO}_3 \text{ solution/sample size)} \times \text{tank size} = \text{amount of 10 % CrO}_3 \text{ solution to be added to tank} \] (X2.3)

X2.5.3.3 Process alloys low in aluminum on the low side of the pH range to obtain a good coating.

X2.5.3.4 Problems and Explanations—The following problems might be encountered when applying the dichromate treatment:

X2.5.4 Abnormally heavy and loose powdery coatings:

X2.5.4.1 The hydrofluoric acid or bifluoride bath is too dilute.

X2.5.4.2 The pH of the dichromate bath is too low. It should not be allowed to fall below 4.1. The pH may be adjusted upward by the addition of 10 % sodium hydroxide solution. The method outlined in X2.3 may be used by substituting sodium hydroxide solution for chromic acid solution.

X2.5.4.3 Treatment of oxidized, corroded, or flux contaminated parts will result in the formation of gray to yellow streaked coatings. Clean parts as outlined in 6.1.

X2.5.4.4 Powdery coatings may form when the work contacts the tank or is in an electrical circuit with the tank through metal holding bars, baskets, or other pieces of equipment which contact the tank.

X2.5.4.5 Prolonged treatment in the dichromate bath.

X2.5.5 Failure to coat or nonuniform coatings:

X2.5.5.1 The pH of the dichromate bath is too high. This is very important when hydrofluoric acid is used as the pre-dip on low aluminum containing alloys such as AZ31B. Adjust the pH as described in X2.3. Frequent checks may be necessary depending on tank size and processing volume.

X2.5.5.2 The dichromate concentration in the bath is too low.

X2.5.5.3 Oily matter has not been completely removed resulting in a spotty coating where some areas are covered and others not. Insufficient cleaning alone may not be the fault. Properly cleaned parts may be recontaminated by an oily film floating on the fluoride or dichromate bath. This oily film could be caused by insufficient rinsing after alkaline cleaning, oil mist in the air, or oil dropping from overhead equipment or other causes.

X2.5.5.4 Previously applied chrome pickle may not have been completely removed.

X2.5.5.5 The part was fluoride treated.

X2.5.5.6 Alloy is one not suited for this type of treatment.

X2.5.5.7 Too long a hydrofluoric acid dip with alloys such as AZ31B produces a fluoride film that does not break down evenly in the normal time, producing a spotty coating. For these alloys, treatment time should be 30 s to 1 min.

X2.5.5.8 The bath was not kept boiling during the treatment period. This is particularly important when processing AZ31B alloy. Minimum bath temperature is 93°C.

X2.5.5.9 Improper rinsing after the hydrofluoric acid or soluble fluoride carried over into the bath exceeds 0.2 %. Streaked coatings will be observed before this limit is reached. A 0.2 % solution of calcium chromate can be added to the bath to precipitate the excess fluoride as calcium fluoride. If the fluoride is reduced in this way, the bath need not be discarded.

X2.6 Chromate Treatment:

X2.6.1 Chromate Salts—Control the concentration of the chromate salts as specified by the supplier. Alternatively, they may be controlled by the procedure outlined in X2.1.1. If this procedure is used however, a carefully prepared 37.5 g/L solution of the salts must first be titrated to determine the sodium dichromate content of the salts. The control point can then be corrected to this value.

X2.6.2 pH—Maintain the pH of the solution between 0.2 to 0.6 for the solution described in 7.3.1 and 0.6 to 1.0 for the solution described in 7.3.2.

X2.6.3 Wetting Agent—When the bath is in continuous operation, control the wetting agent content by adding 0.3 mL of agent for each 37.5 g of salts added to the bath. When the bath has been allowed to stand idle for a period of a week or more, and a thin foam layer does not form during processing, make a full addition of 0.3 mL/L of wetting agent.

X2.6.4 Bath Life—The bath may be replenished until satisfactory coatings are no longer obtained. Although the expected bath life will depend on the amount of dragout, the solution should not be dumped until the replenishment additions are at least equal to one and one-half times the original makeup. This is approximately equal to processing 4.2 m² of surface per litre of solution.

X2.6.4.1 Operational Problems—The following operational problems may be encountered during use of the treatment:

X2.6.5 Failure to form coatings:

X2.6.5.1 The pH of the solution is too high.

X2.6.5.2 Solution may be too cold.

X2.6.5.3 The metal is not properly cleaned. The part was not pickled before treatment in the chromate solution.
X2.6.5.4 The ratio of the acid concentration to the chromate may be too low by using the improper amount or strength of acid.

X2.6.6 Non-adherent powdery coatings:
X2.6.6.1 The part may be made of an alloy that requires a different treating solution, for example, solution specified as in 7.3.1 was used when the solution specified in 7.3.2 is required.
X2.6.6.2 The pH of the solution is too low.
X2.6.6.3 The part was not properly cleaned. The part was not pickled before treatment in the chromate solution.
X2.6.6.4 The ratio of the acid concentration to the chromate salts may be too high.
X2.6.7 Excessive smut on parts:
X2.6.7.1 Aluminum bearing alloys will develop aluminum smut if held in the treating solution for too long a period.

X2.7 Chrome-Manganese Treatment:
X2.7.1 Control—The bath composition given provides for a long working life and yet is not so concentrated as to be expensive initially or wasteful in operation from “drag out” losses. Approximately 2.5 m² of surface can be treated per litre of solution before exhaustion. The pH of the solution varies from about 4 when freshly made to about 6 when nearing exhaustion. Within these limits no control of pH is necessary. Chromating times and the appearance of the films provide an adequate control mechanism. During use a small amount of black manganese dioxide sludge is produced; it should not be discarded. An exhausted bath can be revivified by the careful, controlled addition of sulphuric acid to obtain a pH of approximately 4.1 to 4.2. Alternatively the bath can be revivified by the addition of up to 5% manganese sulphate. Sodium dichromate and magnesium sulphate deplete very slowly and further additions are rarely required except to replace “drag out” losses.

X2.7.2 Operating Problems—The bath may be contained in plastic or glass vessels, but since there is no free acid in the solution it is common to use tanks of aluminum, zinc, mild steel, or galvanized steel as desired. If metal tanks are used, it is important that the magnesium parts are not allowed to rest or otherwise be in contact with the tank. Failure to isolate the workpiece will result in the formation of thicker, powdery chromate films and a more rapid depletion of bath chemicals. The bath may however be intentionally used in a “galvanic” mode to produce a dense matte black surface for optical equipment parts, but slight dimensional losses may occur.

X2.8 Galvanic Chromate Treatment:
X2.8.1 Control—The analytical procedures for hydrofluoric acid or acid fluoride determination are the same as those used for the dichromate treatment given in X2.1 and X2.2. The sulphate-dichromate-hydroxide bath pH should be maintained between 5.6 and 6.0 with careful additions of a solution containing 5% by weight each of chromic acid and concentrated sulfuric acid.

X2.8.2 Operational Problems—The galvanic chromate treatment requires careful control during application. Treatment time, bath condition, and alloy composition affect the color of the coating. Gray, nonuniform coatings indicate unsatisfactory precleaning or baths that are depleted. A good, firm electrical contact must be made with the workpiece and care must be taken to ensure there is no contact with the tank other than by the external circuit. Racks made from monel, stainless steel, or phosphor bronze may be used to treat multiple parts. Parts must be firmly racked for the proper galvanic action to take place. If the tank is made of nonmetallic material, steel, nickel, or phosphor bronze cathode plates must be used. Nonadherent coatings are usually caused by too high a current density, too prolonged a treatment, or too low a bath pH. Poor coatings also result from the use of current densities below recommended levels.

X2.9 Semi-Bright Pickle Treatment:
X2.9.1 A reduction in chemical reaction indicates depletion of the treating solution. The pH could be 1.7 or higher and small batches of the solution should be disposed of appropriately. Larger volumes of the solution can be revivified up to twelve times by one of the following methods:

X2.9.1.1 Add fresh stock solution to replace drag-out and evaporation loss if the bath is used continuously.

X2.9.1.2 Maintain original volume if the bath is used infrequently and losses are due mainly to evaporation by adding 25% of the original amount of chemicals and sufficient water to bring the volume back to its original level.

X2.9.1.3 Analyze the solution and add chemicals as necessary to adjust the chemistry to the original bath composition.

X2.9.2 As noted under 7.5.2 lower operating temperatures reduce staining in air during transfer of the parts pickle to rinse, but longer pickling times result in maximum brightness. Short treatment times are best for polished surfaces.

X2.9.3 Brassy to dark brown stains can be caused by inadequate rinsing or by extended transfer times between pickling and cold water rinse. Should dark brown stains develop proceed as in 7.5.3.

X2.10 Phosphate Treatment:
X2.10.1 The bath composition should be controlled to maintain to following ranges:

X2.10.1.1 Ammonium phosphate 105 to 120 g/L, ammonium sulfite 15 to 30 g/L, pH 5.9 to 6.1.
Standard Specification for Metal Injection Molding (MIM), Ferrous Structural Parts

This standard is issued under the fixed designation B 883; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers ferrous metal injection molded materials fabricated by mixing elemental or prealloyed metal powders with binders, injecting into a mold, debinding, and sintering, with or without subsequent heat treatment.

1.2 This specification covers the following injection molded materials.

1.2.1 Compositions:

1.2.1.1 MIM-2200 Low-alloy steel produced from admixtures of iron powder and other alloying elements such as nickel and molybdenum.

1.2.1.2 MIM-2700 Low-alloy steel produced from admixtures of iron powder, and other alloying elements such as nickel and molybdenum.

1.2.1.3 MIM-4605 Low-alloy steel produced from admixtures of iron powder and other alloying elements such as nickel, molybdenum, and carbon.

1.2.1.4 MIM-316L Austenitic stainless steel produced from prealloyed powder or an admixture of powders.

1.2.1.5 MIM-17-4 PH Precipitation hardening stainless steel produced from prealloyed powder or an admixture of powders.

Note 1—Compositional limits and impurity elements may be different from AISI limits. Chemical composition limits are specified in 6.1 and Table 1.

1.3 Property values stated in English system units are the standard. Conversions to SI units may be approximate.

2. Referenced Documents

2.1 ASTM Standards:

B 243 Terminology of Powder Metallurgy

B 311 Test Method for Density Determination for Powder Metallurgy (P/M) Materials Containing Less than Two Percent Porosity

B 328 Test Method for Density, Oil Content and Interconnected Porosity of Sintered Metal Structural Parts and Oil Impregnated Bearings

E 8 Test Methods for Tension Testing of Metallic Materials

E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials


E 415 Test Method for Optical Emission Vacuum Spectrometric Analysis of Carbon and Low-Alloy Steel

E 1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, Oxygen, and Hydrogen in Steel and in Iron, Nickel, and Cobalt Alloys

E 1479 Practice for Describing and Specifying Induction Coupled Plasma Optical Emission Spectrometers

2.2 Other Test Methods and Standards:

MPIF Standard 35, Material Standards for Metal Injection Molded Parts

MPIF Standard 50, Method for Preparing and Evaluating Metal Injection Molded Debound and Sintered Tension Test Specimens

MPIF Standard 51, Determination of Microhardness of Powder Metallurgy Materials

3. Terminology

3.1 Definitions:

3.1.1 Definitions of powder metallurgy terms can be found in Terminology B 243. Additional descriptive information is available in the Related Material Section of Vol. 02.05 of the Annual Book of ASTM Standards.

4. Ordering Information

4.1 Orders for parts conforming to this specification may include the following:

4.1.1 ASTM Designation,

4.1.2 Alloy composition including carbon content (see 6.1 and Table 1),

4.1.3 Heat Treatment condition and hardness (see Tables 2-5),

4.1.4 Functional or mechanical property testing (see 7.1.1-7.1.3, 8.2, Tables 2-5),

4.1.5 Purchaser or purchaser’s representative desire to witness the inspection and testing of material prior to shipment (see 9.2),

4.1.6 Requirement for certification of material and a report of test results (see 11.1),

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1 This specification is under the jurisdiction of ASTM Committee B-9 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.11 on Near Full Density Powder Metallurgy Metals. Current edition approved Oct. 10, 1997. Published April 1998.

2 Annual Book of ASTM Standards, Vol 02.05.

3 Annual Book of ASTM Standards, Vol 03.01.

4 Annual Book of ASTM Standards, Vol 03.05.

5 Annual Book of ASTM Standards, Vol 03.06.

6 Available from Metal Powder Industries Federation, 105 College Road East, Princeton, NJ 08540–6692.
4.1.7 Requirement for full or partial chemical analysis (see Section 6.), and

4.1.8 Other special requirements as mutually agreed

5. Materials and Manufacture

5.1 Parts shall be made by injection molding mixtures of metal powder with binders, debinding, and sintering, with or without subsequent heat treatment. The material shall conform to the designations in 1.2.1 and meet the chemical composition specified in 6.1 and Table 1.

6. Chemical Composition

6.1 Metal injection molded material shall conform to the chemical requirements prescribed in Table 1.

6.2 Chemical analysis for the elements copper, chromium, molybdenum and nickel shall be determined in accordance with Test Method E 415 (preferred method), Test Method E 350, Test Method E 1479 or other such method as shall be agreed upon between buyer and seller. Analysis of the element carbon shall be determined in accordance with Test Method E 1019, via optical emission spectroscopy, or other method agreed upon between the purchaser and seller.

7. Mechanical Property Requirements

7.1 Mechanical Properties:

<table>
<thead>
<tr>
<th>Material Designation</th>
<th>Minimum Mandatory Values</th>
<th>Typical Values</th>
<th>Typical Values</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Tensile Properties</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ultimate</td>
<td>Yield</td>
<td>Elongation</td>
</tr>
<tr>
<td></td>
<td>Strength</td>
<td>Strength</td>
<td>in 1 in.</td>
</tr>
<tr>
<td>MIM-2200</td>
<td>255</td>
<td>110</td>
<td>20</td>
</tr>
<tr>
<td>MIM-2700</td>
<td>380</td>
<td>205</td>
<td>20</td>
</tr>
<tr>
<td>MIM-4605</td>
<td>380</td>
<td>170</td>
<td>11</td>
</tr>
<tr>
<td>MIM-4605-HTC</td>
<td>1480</td>
<td>1310</td>
<td>-</td>
</tr>
</tbody>
</table>


Density determined in accordance with Test Methods B 311 or B 328. Apparent hardness determined in accordance with Test Methods E 18. Tensile properties determined on test bars prepared in accordance with MPIF Standard 50 and tested in accordance with Test Method E 8.

5. Converted from HK 100 Microhardness, MPIF Standard 51, Appendix B.

6. These data were measured on test bars tempered for 1 hour at 177°C.

TABLE 1 Chemical Requirements For Metal Injection Molded Parts

<table>
<thead>
<tr>
<th>Material Designation</th>
<th>Fe</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>C</th>
<th>Cu</th>
<th>Nb + Ta</th>
<th>OTHER</th>
</tr>
</thead>
<tbody>
<tr>
<td>MIM-2200</td>
<td>Min.</td>
<td>Bal.</td>
<td>1.5</td>
<td>-</td>
<td>0.0</td>
<td>0.0</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Max.</td>
<td>Bal.</td>
<td>2.5</td>
<td>-</td>
<td>0.5</td>
<td>0.1</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>MIM-2700</td>
<td>Min.</td>
<td>Bal.</td>
<td>6.5</td>
<td>-</td>
<td>0.0</td>
<td>0.0</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Max.</td>
<td>Bal.</td>
<td>8.5</td>
<td>-</td>
<td>0.5</td>
<td>0.1</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>MIM-4605</td>
<td>Min.</td>
<td>Bal.</td>
<td>1.5</td>
<td>-</td>
<td>0.2</td>
<td>0.4</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Max.</td>
<td>Bal.</td>
<td>2.5</td>
<td>-</td>
<td>0.5</td>
<td>0.6</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>MIM-316L</td>
<td>Min.</td>
<td>Bal.</td>
<td>10.0</td>
<td>16.0</td>
<td>2.0</td>
<td>0.00</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Max.</td>
<td>Bal.</td>
<td>14.0</td>
<td>18.0</td>
<td>3.0</td>
<td>0.03</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>MIM-17-4PH</td>
<td>Min.</td>
<td>Bal.</td>
<td>3.0</td>
<td>15.5</td>
<td>-</td>
<td>0.00</td>
<td>3.0</td>
<td>0.15</td>
</tr>
<tr>
<td></td>
<td>Max.</td>
<td>Bal.</td>
<td>5.0</td>
<td>17.5</td>
<td>-</td>
<td>0.07</td>
<td>5.0</td>
<td>0.45</td>
</tr>
</tbody>
</table>

TABLE 2 Mandatory and Typical Mechanical Properties of Metal Injection Molded Low-Alloy Steels

<table>
<thead>
<tr>
<th>Material Designation</th>
<th>Minimum Mandatory Values</th>
<th>Typical Values</th>
<th>Typical Values</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Tensile Properties</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ultimate</td>
<td>Yield</td>
<td>Elongation</td>
</tr>
<tr>
<td></td>
<td>Strength</td>
<td>Strength</td>
<td>in 25.4 mm</td>
</tr>
<tr>
<td>MIM-2200</td>
<td>255</td>
<td>110</td>
<td>20</td>
</tr>
<tr>
<td>MIM-2700</td>
<td>380</td>
<td>205</td>
<td>20</td>
</tr>
<tr>
<td>MIM-4605</td>
<td>380</td>
<td>170</td>
<td>11</td>
</tr>
<tr>
<td>MIM-4605-HTC</td>
<td>1480</td>
<td>1310</td>
<td>-</td>
</tr>
</tbody>
</table>


Density determined in accordance with Test Methods B 311 or B 328. Apparent hardness determined in accordance with Test Methods E 18. Tensile properties determined on test bars prepared in accordance with MPIF Standard 50 and tested in accordance with Test Method E 8.

5. Converted from HK 100 Microhardness, MPIF Standard 51, Appendix B.

6. These data were measured on test bars tempered for 1 hour at 177°C.
TABLE 4 Mandatory and Typical Mechanical Properties of Metal Injection Molded Stainless Steels (a)

<table>
<thead>
<tr>
<th>Material Designation</th>
<th>Minimum Mandatory Values</th>
<th>Typical Values</th>
<th>Typical Values</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Tensile Properties</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ultimate Strength</td>
<td>Yield Strength</td>
<td>Elongation % in 1 in.</td>
</tr>
<tr>
<td>MIM-316L</td>
<td>65</td>
<td>20</td>
<td>40</td>
</tr>
<tr>
<td>MIM-17-4 PH</td>
<td>115</td>
<td>94</td>
<td>4</td>
</tr>
<tr>
<td>MIM-17-4 PH-HT (c)</td>
<td>155</td>
<td>140</td>
<td>4</td>
</tr>
</tbody>
</table>


Density determined in accordance with Test Methods B 311 or B 328. Apparent hardness determined in accordance with Test Methods E 18. Tensile properties determined on test bars prepared in accordance with MPIF Standard 50 and tested in accordance with Test Methods E 8.

SI Units:

<table>
<thead>
<tr>
<th>Material Designation</th>
<th>Minimum Mandatory Values</th>
<th>Typical Values</th>
<th>Typical Values</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Tensile Properties</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ultimate Strength MPa</td>
<td>Yield Strength MPa</td>
<td>Elongation % in 25.4 mm</td>
</tr>
<tr>
<td>MIM-316L</td>
<td>450</td>
<td>140</td>
<td>4</td>
</tr>
<tr>
<td>MIM-17-4 PH</td>
<td>795</td>
<td>645</td>
<td>4</td>
</tr>
<tr>
<td>MIM-17-4 PH-HT (c)</td>
<td>1070</td>
<td>965</td>
<td>4</td>
</tr>
</tbody>
</table>

(a) Reprinted by permission from MPIF Standard 35, "Materials Standards for Metal Injection Molded Parts", 1993/1994 Metal Powder Industries Federation, 105 College Road East, Princeton, NJ 08540-6692. S.I. values converted from English units in Table 3.

Density determined in accordance with B 311 or B 328. Apparent hardness determined in accordance with ASTM E 18. Tensile properties determined on test bars prepared in accordance with MPIF Standard 50 and tested in accordance with ASTM E 8.

SI Units:

7.1.1 The preferred method of verifying the acceptable performance of a finished part is a qualification test to be performed on an actual part. The specific test should be determined following consideration of the function of the part, and should be agreed upon between manufacturer and purchaser.

7.1.2 Mandatory and typical mechanical properties of materials covered by this specification are shown in Tables 2-5.

7.1.3 The tensile properties of MIM materials should be measured using test specimens prepared and evaluated in accordance with MPIF Standard 50.

7.2 Mechanical Property Test Methods:

7.2.1 Tensile Test Method—When requested in the purchase order, tensile specimens shall be specially injection molded per MPIF Standard 50, sintered, and heat treated if necessary, along with production parts. Tensile specimens shall be tested in accordance with Test Method E 8. Yield strength shall be determined by the 0.2 % offset method. MPIF Standard 50 governs the manufacture of the test bars, but the testing procedure is governed by Test Method E 8.

8. Sampling

8.1 Lot—Unless otherwise specified, a lot is a quantity of product produced under similar conditions so that the product within the lot is expected to be homogeneous in all significant attributes and submitted for inspection at one time.

8.2 Testing—The manufacturer and purchaser shall mutually agree upon the number of specimens to represent the lot for qualification, chemical or mechanical property testing.

9. Inspection

9.1 Inspection of the parts supplied under this specification shall be the responsibility of the manufacturer or a mutually agreed upon third party.

9.2 If the purchaser desires that a representative witness the inspection and testing of the material prior to shipment, such a requirement shall be part of the purchase order.

10. Rejection

10.1 Parts that fail to conform to the requirements of this specification may be rejected. Rejection should be reported to the manufacturer or supplier promptly and in writing.

11. Certification

11.1 When specified in the purchase order, a manufacturer’s certification shall be furnished to the purchaser that the parts were manufactured, samples tested and inspected in accordance with this specification and found to meet its requirements. When specified in the purchase order, a report of the test results shall be furnished.

12. Keywords

12.1 mechanical properties; metal injection molding (MIM); metal injection molded parts; metal injection molded steels; metal powders; MIM; PIM; powder injection molding; sintered steels; stainless steels
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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 100 Barr Harbor Drive, West Conshohocken, PA 19428.
Standard Test Method for Determination of Magnetic Saturation (Ms) of Cemented Carbides

This standard is issued under the fixed designation B 886; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope *

1.1 This test method covers the determination of magnetic saturation (Ms) of cemented carbide powder materials and sintered products using magnetic saturation induction test instrumentation.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   A 340 Terminology of Symbols and Definitions Relating to Magnetic Testing
   B 243 Terminology of Powder Metallurgy
   E 1316 Terminology for Nondestructive Examinations

3. Terminology

3.1 Definitions:
   3.1.1 For definition of terms used in this procedure refer to Terminology A 340, Terminology B 243, and Terminology E 1316.
   3.1.2 dc—direct current.

4. Summary of Test Method

4.1 A test specimen is statically positioned in the magnetic field generated by d-c coils or a permanent magnet and sensing coils of the test apparatus. The specimen is magnetized to saturation by the field and translated linearly that the intrinsic magnetic moment of the specimen’s binder phase induces a d-c current to flow in the sensing coils of the test apparatus. The induced current is proportional to the amount of magnetic binder phase present in the test specimen. Measurement of the induced current permits calculation of the intrinsic magnetic saturation of the test sample.

5. Significance and Use

5.1 This test method allows the non-destructive measurement of the magnetic fraction of the binder phase in cemented carbide powder materials and sintered product, and may be used as an indirect measure of the carbon level in the material or product.

5.2 Measurement of magnetic saturation provides a comparison of the relative fraction of magnetic binder phase that is, cobalt, nickel, or iron, present in the material and can be used for acceptance of product to specification.

5.3 Measurement of magnetic saturation can be used as a measure of the quality of powder material.

6. Interferences

6.1 No direct absolute measurement of magnetic saturation can be made. The measurement is a relative comparison of standard reference materials.

6.2 Measurement of magnetic saturation is a non-destructive “bulk” measurement which is averaged over the test specimen volume. The technique cannot be used to infer anything regarding the relative distribution of the binder phase within the test sample.

6.3 Measurement of magnetic saturation is affected by test sample size and shape, especially aspect ratio, and the composition of the binder phase, that is, a mixture of ferromagnetic elements of varying intrinsic magnetic moments

6.4 Small test samples or test samples containing relatively low concentrations of the binder phase may be immeasurable in that the low concentration prohibits detection by the field sensing coils of the specific apparatus employed.

7. Apparatus

7.1 Instrumentation capable of inducing magnetic saturation and then sensing the inherent intrinsic magnetic moment of the magnetic fraction of the binder phase.

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*A Summary of Changes section appears at the end of this standard.

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7.2 Balance having a capacity of 200 g and a sensitivity of 0.001 g.

8. Standards

8.1 No certified cemented carbide standards, powder materials or sintered product, are available for magnetic saturation measurement. Most common practice is the development of (internal) reference materials representative of the test samples being evaluated.

8.2 Pure ferromagnetic materials such as cobalt (Co), iron (Fe), or Nickel (Ni), and iron oxide, that is, Fe₃O₄, with known magnetic moments are recommended for calibration of the instrumentation.

9. Test Specimen

9.1 Test specimens may be in the form of sintered solids, unsintered powder compacts, or encapsulated powders of a size suitable to fit into the specimen holder of the apparatus. The long axis of the specimen should be perpendicular to the magnetic field of the test apparatus.

10. Procedure

10.1 For commercial instrumentation, refer to the equipment manufacturers operating manual and follow operating instructions.

10.2 Obtain the mass of each test specimen.

10.3 Position the test sample in the center of the magnetic field/sensing coils.

10.4 Saturate the test specimen.

10.5 Remove/translate the test specimen in the sensing coils.

10.6 Read and record the value of the intrinsic magnetic saturation from the test apparatus. Measurement units are Tesla-m³ per kilogram (emu per gram (csg)).

10.7 Relative percent of magnetic saturation of the test sample may be calculated and reported. Consideration must be given to the composition of the binder phase with respect to ferromagnetic components, and the presence of other metallic constituents, for example, chromium (Cr) in the powder material or sintered product, that would affect the binder phase composition or magnetic moment of the constituents.

10.8 Repeat measurements can be made to obtain an average value and range of values for test specimens of asymmetric shape. Orientation of the test sample should be changed within the test apparatus sample chamber for each repeat measurement.

11. Report

11.1 Report magnetic saturation as Tesla-m³ per kilogram (emu per gram), or as relative percent, (that is, magnetic portion of the binder phase/total mass of binder phase in test sample).

12. Precision and Bias

12.1 Precision—Measurement of magnetic saturation is a relative comparison against well characterized reference materials of similar composition and mass (see 8.1). No statement about precision can be made due to the effects of test specimen size and shape, variations in the binder phase composition that are within material or product specification, and the effect of variations of the carbon content of the test samples (see 6.3).

12.2 Bias—Measurement of magnetic saturation is a relative comparison against well characterized reference materials of similar composition and mass (see 8.1). No statement about bias can be made due to the affects of test specimen size and shape, variations in the binder phase composition that are within material or product specification, and the affect of variations of the carbon content of the test samples (see 6.3).

13. Keywords

13.1 cemented carbide; magnetic saturation (Ms)

SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue (B 886 – 98) that may impact the use of this standard.

(1) The use of a permanent magnet to induce saturation was added.

(2) The word “or” was added in Section 5.2. It has been determined that mixtures of nickel, cobalt, and iron in sintered carbides will produces alloys of various amounts whose intrinsic magnet moment is different than the sum of the individual binder materials.

(3) The units were changed to SI units as the standard unit in Sections 10.6 and 11.

(4) The reference to vanadium affecting the magnetic moment of the binder material was removed, because vanadium does not affect magnetic saturation determinations.
Standard Test Method for Determination of Coercivity (Hcs) of Cemented Carbides

This test method covers the determination of magnetization coercivity (Hcs) of cemented carbide materials and products using coercive force instrumentation. It is patterned after ISO 3326.

1. Scope

1.1 This test method covers the determination of magnetization coercivity (Hcs) of cemented carbide materials and products using coercive force instrumentation. It is patterned after ISO 3326.

1.2 The values in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
A 340 Terminology of Symbols and Definitions Relating to Magnetic Testing
B 243 Terminology of Powder Metallurgy

2.2 ISO Standard:
ISO 3326 Hardmetals - Determination of (the Magnetization) Coercivity

3. Terminology

3.1 Definitions:

3.1.1 For definition of terms used in this procedure refer to Terminology A 340 and Terminology B 243.

3.1.2 dc—direct current

4. Summary of Test Method

4.1 A test sample is positioned in the dc magnetic field of the test apparatus and magnetized to technical saturation. The magnetic field polarity is then reversed and the test sample is demagnetized by increasing the energy of the reversed magnetic field until the test sample reaches zero magnetism. The coercive force (Hc) is the magnetizing force required to return the saturated magnetic induction to zero.

5. Significance and Use

5.1 Measurement of coercivity provides a relative comparison of carbide grain size, binder content, and possibly carbon deficiency for a given graded carbide material or product, and may be employed as a non-destructive measurement indicating deviation from a specified norm.

5.2 This test method allows the non-destructive estimate of average carbide grain size in sintered cemented carbide hardmetals. It is appropriate for a wide range of compositions and tungsten carbide (WC) WC grain sizes, and can be used for acceptance of material or product to specification.

6. Interferences

6.1 Hcs measurement is a non-destructive “bulk” measurement that is averaged over the specimen volume. Bi-modal grain size distributions will give approximately the same Hc value as would be obtained from a normal grain size distribution about the same mean value.

6.2 Large test specimens must be sized to fit within the magnetic field coil spacing available for the apparatus employed.

6.3 Small test specimens may be immeasurable if their size prohibits detection by the magnetic field coils for the apparatus employed.

6.4 Specimen shape, that is, symmetry and aspect ratio, influence Hc measurement values and repeatability of results. Test specimens should be positioned with their long axis in the direction of the magnetic field. Asymmetrically shaped test specimens should be tested in several positions, the measurement values recorded, and the average value reported.

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2 Annual Book of ASTM Standards, Vol 03.04.

3 Annual Book of ASTM Standards, Vol 02.05.

7. Apparatus

7.1 Instrumentation capable of the dc magnetization of appropriately sized test samples to technical saturation and accurate measurement of the energy required to restore the magnetic induction to zero.

8. Procedure

8.1 For commercial instrumentation, refer to the equipment operators manual and follow the manufacturer’s operating instructions.

8.2 Position the test sample in the center of the magnetic field. The test sample should be positioned with its long axis in the direction of the magnetic field (see 6.4).

8.3 Magnetize the test sample to technical saturation.

8.4 Reverse the magnetic field polarity and demagnetize the test sample to zero.

8.5 Record the $H_c$ measurement, that is, energy required to demagnetize the test sample.

8.6 Replicate measurement of the same test sample shall be made, reversing the polarity of the saturation and demagnetizing magnet fields, where possible.

8.7 For asymmetric sample shapes, repeat measurements shall be made by repositioning the specimen in the d-c magnetic field of the instrumentation with consideration being given to the shape, that is, symmetry of the test sample, and to its aspect ratio, that is, length versus width.

9. Report

9.1 Report the following information:
9.1.1 Test sample identification,
9.1.2 Average $H_c$ coercivity (amp per meter (A/m) or oersteds), and
9.1.3 Range of measured $H_c$ values, especially for replicate measurements of asymmetric sample shapes.

10. Precision and Bias

10.1 Precision—Measurement of coercive force is a relative comparison against well characterized reference materials (see Section 8). No statement about precision can be made due to the effects of test specimen shape, variations in the binder content that are within material or product specification, and the presence of carbon deficiency.

10.2 Bias—Measurement of coercive force is a relative comparison against well characterized reference materials (see Section 8). No statement about bias can be made due to the effects of test specimen shape, variations in the binder content that are within material or product specification, and the presence of carbon deficiency.

10.3 No certified cemented carbide standards are available for coercive force measurement. Most common practice is the development of (internal) reference materials representative of the product(s) being evaluated.

11. Keywords

11.1 cemented carbide; coercive force; coercivity; magnetization

SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue (B 887 – 98) that may impact the use of this standard.

(1) Section 1.2 was added to the Scope to indicated the use of SI units in the standard. Current Section 1.2 was renumbered to 1.3.

(2) The misspelling of asymmetrically was corrected in Section 6.4.

(3) Section 9.1.2 was removed because the reporting of mass has no bearing on the value obtained for coercivity testing.

(4) Section 9.1.3 was renumbered to 9.1.2. The units were changed to reflect SI units as the acceptable standard.

(5) Section 9.1.4 was renumbered to 9.1.3.
Standard Test Method for Determination of Metallic Constituents of Tungsten Alloys and Tungsten Hardmetals by X-Ray Fluorescence Spectrometry

This standard is issued under the fixed designation B 890; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a procedure for the determination of the concentration, generally reported as mass percent, of the metallic constituents of tungsten-based alloys and hardmetals utilizing wavelength dispersive X-ray fluorescence spectrometry (XRF). This test method incorporates the preparation of standards using reagent grade metallic oxides, lithium-borate compounds, and fusion techniques. This test method details techniques for preparing representative specimens of both powder and sintered tungsten-based material. This test method is accurate for a wide range of compositions, and can be used for acceptance of material to grade specifications.

1.2 This test method is applicable to mixtures of tungsten or tungsten carbide with additions of refractory metal carbides and binder metals. Table 1 lists the most common elemental constituents and their concentration range. Note that many of these occur as metallic carbides.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
E 1361 Guide for Correction of Interelement Effects in X-Ray Spectrometric Analysis

2.2 ISO Standard:
ISO-4503(E) Hardmetal—Determination of contents of metallic elements by X-ray fluorescence - Fusion method

2.3 Handbook of Chemistry and Physics, 67th ed

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology E 135.

4. Summary of Test Method

4.1 A suite of standards which closely match the chemical content of the material to be analyzed are prepared using reagent grade metallic oxides. Test samples are oxidized in a high-temperature furnace open to air. Fused glass specimens are prepared for these standards and for the test samples to be analyzed. These specimens of oxidized tungsten or tungsten carbide alloys are irradiated with an energetic primary X-ray beam. The intensity of the resultant secondary X-rays, characteristic in energy, for each elemental constituent is measured by means of a suitable detector or combination of detectors after diffraction by a Bragg spectrometer. The concentration of each constituent element is calculated by comparison with standard samples which closely match the chemical content of the analyzed material. The calculation may be manual, incorporate

<table>
<thead>
<tr>
<th>Table 1 Elemental Constituents and Concentration Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Element</td>
</tr>
<tr>
<td>---------</td>
</tr>
<tr>
<td>Chromium (Cr)</td>
</tr>
<tr>
<td>Cobalt (Co)</td>
</tr>
<tr>
<td>Hafnium (Hf)</td>
</tr>
<tr>
<td>Iron (Fe)</td>
</tr>
<tr>
<td>Molybdenum (Mo)</td>
</tr>
<tr>
<td>Nickel (Ni)</td>
</tr>
<tr>
<td>Niobium (Nb)</td>
</tr>
<tr>
<td>Tantalum (Ta)</td>
</tr>
<tr>
<td>Titanium (Ti)</td>
</tr>
<tr>
<td>Vanadium (V)</td>
</tr>
</tbody>
</table>

ISO-4503(E) Hardmetal—Determination of contents of metallic elements by X-ray fluorescence - Fusion method

4 Available from American National Standards Institute, 11 West 42nd St., 13th Floor, New York, NY 10036.
a calibration curve, or be performed by a computer program which incorporates correction routines for X-ray absorption and enhancement effects (see Guide E 1361).

5. Significance and Use

5.1 This test method allows the determination of the chemical composition of powdered and sintered tungsten-based hardmetals. This test method is not applicable to material which will not oxidize readily at high temperatures in air, such as tungsten/copper or tungsten/silver alloys.

5.2 This test method specified lithium-borate compounds for the glass fusion material. However, numerous other choices are available. These include other lithium-borate compounds, sodium carbonate and borate mixtures, and others. The methodology specified here is still applicable as long as the same fusion mixture is used for both standards and specimens.

6. Interferences

6.1 Errors in XRF-determined compositional values may be encountered due to X-ray enhancement and absorption effects dependent on the elements present and the X-ray line being measured for a specific element. This effect can be reduced by determination of correction factors using appropriate standards and interelement correction routines, manual or computerized.

6.2 Accuracy and precision of the analytical results obtained from molybdenum-containing samples may be rendered unreliable due to the sublimation and evaporation of molybdenum from the material during the oxidation step in specimen preparation.

6.3 Incorporation of the fusion method of specimen preparation will:

6.3.1 Reduce the deleterious influence of particle size effects experienced when analyzing powder materials by varying particle size.

6.3.2 Reduce inhomogeneities within a sample.

6.3.3 Improve penetration of X rays.

6.3.4 Reduce interelement interferences by tungsten on all other elements.

7. Apparatus

7.1 X-Ray Fluorescence Wavelength Spectrometer

7.2 Fluxer—An automated high-temperature mixing device capable of melting, mixing, and pouring a molten liquid specimen into a proper casting dish, is highly preferred

7.3 Analytical Balance, readability of 0.00001 g

7.4 Toploading Balance, readability of 0.001 g

7.5 Ordinary Laboratory Apparatus

7.6 One Pt - 5 % Au Casting Dish (minimum)

7.7 One Pt - 5 % Au Crucible (minimum)

7.8 Platinum Tipped Tongs

7.9 Weighing Paper

7.10 Chemical Spoon and Scoopula

7.11 Ceramic Combustion Boat

7.12 High Temperature Tube or Muffle Furnace, open to the atmosphere

7.13 Self-adhering Stickers, ¼ by 1 in.

7.14 High-Temperature marking pen

7.15 Ceramic Mortar and Pestle

7.16 Miniature Mixer, optional

8. Reagents and Materials

8.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specification of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 Di-lithiumtetraborate (Li₂B₄O₇): Lithiummetaborate (LiBO₂), 66 + 34.

8.3 Lithium Bromide (LiBr).

8.4 Metallic Oxide Powder, highest oxidation state for elements of interest; that is Co₃O₄, Cr₂O₃, Fe₃O₄, HfO₂, MoO₃, Nb₂O₅, NiO, Ta₂O₅, TiO₂, V₂O₅, and WO₃.

Warning—Several of the metallic oxides used in this test method are highly toxic and possibly carcinogenic, such as Cr₂O₃, NiO, or V₂O₅. Extreme care should be used at all times when handling this material (especially V₂O₅). All mixing of standards should be performed in a fume hood. All of the lithium compounds are water-soluble and therefore able to be absorbed into the body by inhalation and possibly by absorption through the skin. This material should be weighed in a fume hood.

8.5 Nitric Acid (HNO₃).

9. Specimen Preparation

9.1 Prepare specimens of the material to be analyzed by oxidizing, weighing, and fusing starting powders, chips, or crushed sintered hard metal samples.

9.2 Place 3 to 5 g of powdered specimen in a labeled ceramic combustion boat. If a sintered sample is to be analyzed, then the sample must be crushed or pulverized into small pieces or chips must be produced by machining prior to placement in the combustion boat.

9.3 Oxidize the specimen in the heat zone of a high-temperature tube or muffle furnace open to the atmosphere at 825 ± 25°C. All specimens must be oxidized.

9.4 When the specimen has been completely oxidized (4 to 6 h), remove from the furnace and allow to cool.

Note 1—Complete oxidation of a sintered magnetic tungsten hard metal sample can be checked by testing the cool oxidized chips with a magnet. If any of the chips are still magnetic, recrush the sample and place back in the furnace for further oxidation.

9.5 Pour the specimen onto a clean sheet of paper or into a clean mortar and gently crush with a pestle.

9.6 Transfer the specimen to a labeled specimen vial.

9.7 In a fume hood, weigh out 15,000 ± 0.001 g of the dilithium tetraborate; lithiummetaborate mixture and 0.200 ± 0.001 g of LiBr and transfer to a clean sample vial. This mixture will be referred to as the “fusion mixture.” Seal and store until needed.

9.8 The lithiummetaborate mixture will be referred to as the “fusion mixture.” Seal and store until needed.

*Reagent Chemicals, American Chemical Society Specification, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analytical Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopoeia and National Formulary, U.S. Pharmaceutical Convention, Inc. (USPC), Rockvale, MD.
NOTE 2—Other fusion materials can be used. See 5.2.

9.8 In a fume hood, transfer the fusion mixture to a platinum crucible immediately prior to weighing of the oxidized sample material.

9.9 Weigh out 1.0000 ± 0.00005 g of oxidized specimen and transfer to the platinum crucible. Mix gently with the fusion mixture.

NOTE 3—If there is not enough sample to make a standard fusion, proportionate amounts of oxidized test sample and fusion mixture can be utilized to prepare a specimen recognizing that larger fractional errors may be incurred in the analysis. This should only be used when absolutely necessary.

9.10 Using the fluxer, melt the specimen at 1300 ± 100°C and cast into a heated platinum casting dish.

9.10.1 Warning—The process of making glass fusions exposes personnel to high-temperature liquids. Extreme care should be exercised while preparing these samples. These high temperatures also cause some volatilization of the lithium compounds. The fluxer should have an exhaust hood to remove these gases from the facility. The lithium compounds used in this procedure are hygroscopic. Material open to the atmosphere for an extended period of time will absorb moisture. Exposure of this material to subsequent high heat will cause rapid formation of steam and may cause spattering of the molten glass onto the instrument and possibly the operator.

9.11 While the fused specimen is cooling, remove the crucible from the instrument with the platinum-tipped tongs and cool under a stream of water.

9.12 Place the crucible in a 1000-mL beaker which has a 10-volume percent solution of nitric acid. Put the beaker on a hot plate and warm the solution. The crucible should be clean in approximately 30 min. Remove the crucible from the acid bath with tongs and rinse with water. Dry the crucible and store.

9.13 When the fused specimen is cool, remove from the casting dish by gripping the dish firmly with tongs, turning the dish over, and gently tapping against a clean paper. The dish and fused specimen should cleanly separate. Label the fused specimen with a self-adhering tag.

NOTE 4—Any evidence of wetting between the specimen and the platinum crucible or casting dish is an indication that the specimen has reacted with these vessels and is not a valid representative sample.

9.14 If the fusion crystallizes or fractures on cooling, crush the fusion and recast. If the fused specimen cannot be removed from the platinum casting dish with very light tapping, dissolve the specimen from the dish using a warm 10-volume percent nitric acid solution. Prepare a new specimen in accordance with 9.7-9.10.

Caution—Excessive prying or tapping of the crystallized specimen while it is in the dish will damage the platinum ware.

10. Standardization of Spectrometer and Analysis

10.1 Based on the X-ray spectrometer configuration and instrument manufacturer’s operating instructions, determine the instrument operating parameters to provide optimum spectral analysis for each element being analyzed in a given matrix. Table 2 provides the approximate X-ray peak positions (Bragg angle - 2θ) and crystals recommended for each of the elements of interest.

10.2 If required, normalize the X-ray spectrometer operating parameters to obtain the appropriate secondary x-ray intensities from the reference standards utilized.

10.3 Measure X-ray intensities on a sufficient number of fused standards to establish a calibration curve (intensity versus concentration of analyte) for each element of interest. This should only be used when absolutely necessary.

10.4 Calibration curves may be established manually, or corrections for interelement effects may be calculated using XRF vendor-supplied computer software.

NOTE 5—The number of standards sufficient to establish a calibration curve is dependent on the range of concentrations to be analyzed for each element. In all cases, a minimum of six standards is required.

10.5 Calibration curves may be established manually, or corrections for interelement effects may be calculated using XRF vendor-supplied computer software.

11.1 Obtain X-ray intensity data from the fused test specimens.

11.2 Calculate relative concentrations utilizing appropriate calibration curves and absorption and enhancement correction routines, if available.

12. Report

12.1 Report the results of the analysis as mass percent of the metallic or carbide constituent. Report average values of replicate determinations, either measurements or samples, if performed, along with the concentration range. The correction routine employed to determine final concentration values should also be specified by the party completing the analysis, if required.

12.2 The parties involved may require reporting of the actual X-ray spectrometer operating parameters employed for each element of interest. These typically include:

12.2.1 Specimen form,
12.2.2 X-ray source type,
12.2.3 X-ray tube operating conditions, kV and mA,
12.2.4 X-ray line analyzed,
12.2.5 Diffracting crystal,
12.2.6 Type of detector, and
12.2.7 X-ray path whether vacuum, air, or inert gas.
relative concentration of the analyte. The repeatability standard deviation for minor constituents has been determined to be \( \pm 3.35\% \) of the relative concentration of the analyte. The 95\% repeatability limits for major constituents is 0.7\% of the relative concentration of the analyte. The reproducibility is being determined.

13.2 Bias—No information can be presented on the bias in this test method because no material having an accepted reference value is available.

14. Keywords

14.1 absorption and enhancement effects; fusion; interelement effects; tungsten alloys; tungsten carbides; tungsten hardmetals; X-ray fluorescence spectrometry

ANNEX

(Mandatory Information)

A1. PREPARATION OF FUSED MATCHED STANDARDS

A1.1 Determine the composition of the reference standard required for calibration of the spectrometer. Using the conversion factors listed in Table A1.1, convert the mass percent of metallic carbides or elements to their oxide mass percent for each of the reference standards. A minimum of six standards with matrices which cover the concentration range of the material to be measured is required for accurate determination of constituents. A reference standard may also be required for periodic monitoring of the X-ray spectrometer to correct for instrument drift. This test method should contain all elements of interest.

A1.2 Using an analytical balance, weigh out each of the required metallic oxides to \( \pm 0.00005 \) g and combine into one container.

A1.3 Mix this material using a miniature laboratory mixer or a mortar and pestle to obtain a homogeneous mixture of components.

A1.4 Make three fusion specimens of each standard in accordance with 9.7-9.10.

A1.5 Crush all three specimens of each standard and blend together to form a homogenous mixture.

A1.6 Weigh out approximately 16 g of this mixture and recast.

A1.7 Label each fusion sample with a suitable sticker.

NOTE A1.1—Maintain standards in a desiccator while not in use. If devitrification of the fusion occurs, crush and recast in accordance with 9.10.

---

**TABLE A1.1 Conversion Factors for Carbides, Oxides, and Elements**

<table>
<thead>
<tr>
<th>Material</th>
<th>Density</th>
<th>Molecular Mass</th>
<th>Conversion Carbid(\rightarrow) Element</th>
<th>Conversion Element(\rightarrow) Oxide</th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>19.35</td>
<td>183.85</td>
<td>WC%(\times(0.938676))</td>
<td>W%(\times(1.261073))</td>
</tr>
<tr>
<td>WC</td>
<td>15.63</td>
<td>195.88</td>
<td>WC%(\times(0.938676))</td>
<td>Co%(\times(1.361990))</td>
</tr>
<tr>
<td>WO(_{3})</td>
<td>7.16</td>
<td>231.85</td>
<td>W%(\times(0.938676))</td>
<td>W%(\times(1.261073))</td>
</tr>
<tr>
<td>Co</td>
<td>8.85</td>
<td>58.93</td>
<td>Co%(\times(1.361990))</td>
<td>W%(\times(0.938676))</td>
</tr>
<tr>
<td>Co(<em>{3})O(</em>{4})</td>
<td>6.07</td>
<td>240.80</td>
<td>Co%(\times(1.361990))</td>
<td>W%(\times(0.938676))</td>
</tr>
<tr>
<td>Ti</td>
<td>4.50</td>
<td>47.90</td>
<td>Ti%(\times(0.799519))</td>
<td>Ti%(\times(1.667641))</td>
</tr>
<tr>
<td>TiC</td>
<td>4.93</td>
<td>59.89</td>
<td>Ti%(\times(0.799519))</td>
<td>Ti%(\times(1.667641))</td>
</tr>
<tr>
<td>TiO(_{2})</td>
<td>4.26</td>
<td>79.88</td>
<td>Ti%(\times(0.799519))</td>
<td>Ti%(\times(1.667641))</td>
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<tr>
<td>Ta</td>
<td>16.60</td>
<td>180.95</td>
<td>Ta%(\times(1.221042))</td>
<td>Ta%(\times(1.221042))</td>
</tr>
<tr>
<td>TaC</td>
<td>14.53</td>
<td>192.96</td>
<td>Ta%(\times(1.221042))</td>
<td>Ta%(\times(1.221042))</td>
</tr>
<tr>
<td>TaO(_{5})</td>
<td>8.20</td>
<td>441.89</td>
<td>Ta%(\times(1.221042))</td>
<td>Ta%(\times(1.221042))</td>
</tr>
<tr>
<td>Nb</td>
<td>8.57</td>
<td>92.91</td>
<td>Nb%(\times(0.885520))</td>
<td>Nb%(\times(1.403526))</td>
</tr>
<tr>
<td>NbO(_{3})</td>
<td>7.85</td>
<td>104.92</td>
<td>Nb%(\times(0.885520))</td>
<td>Nb%(\times(1.403526))</td>
</tr>
<tr>
<td>Ni</td>
<td>8.90</td>
<td>58.93</td>
<td>Ni%(\times(1.272402))</td>
<td>Ni%(\times(1.272402))</td>
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<td>NiO(_{5})</td>
<td>6.67</td>
<td>74.69</td>
<td>Ni%(\times(1.272402))</td>
<td>Ni%(\times(1.272402))</td>
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<tr>
<td>Fe</td>
<td>7.86</td>
<td>55.85</td>
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<td>Fe%(\times(1.381990))</td>
</tr>
<tr>
<td>FeO(_{4})</td>
<td>5.18</td>
<td>231.54</td>
<td>Fe%(\times(1.381990))</td>
<td>Fe%(\times(1.381990))</td>
</tr>
<tr>
<td>Cr</td>
<td>7.19</td>
<td>52.00</td>
<td>Cr%(\times(1.460000))</td>
<td>Cr%(\times(1.460000))</td>
</tr>
<tr>
<td>Cr(<em>{2})O(</em>{3})</td>
<td>6.68</td>
<td>180.01</td>
<td>Cr(<em>{2})C(</em>{2}%(\times(0.866552))</td>
<td>Cr%(\times(1.460000))</td>
</tr>
<tr>
<td>Cr(<em>{2})O(</em>{3})</td>
<td>5.21</td>
<td>151.99</td>
<td>Cr(<em>{2})C(</em>{2}%(\times(0.866552))</td>
<td>Cr%(\times(1.460000))</td>
</tr>
<tr>
<td>Hf</td>
<td>13.31</td>
<td>178.49</td>
<td>Hf%(\times(1.179282))</td>
<td>Hf%(\times(1.179282))</td>
</tr>
<tr>
<td>HfC</td>
<td>12.20</td>
<td>190.50</td>
<td>HfC%(\times(0.936951))</td>
<td>HfC%(\times(0.936951))</td>
</tr>
<tr>
<td>HfO(_{2})</td>
<td>9.68</td>
<td>210.49</td>
<td>HfC%(\times(0.936951))</td>
<td>HfC%(\times(0.936951))</td>
</tr>
<tr>
<td>V</td>
<td>5.96</td>
<td>50.94</td>
<td>V%(\times(1.785190))</td>
<td>V%(\times(1.785190))</td>
</tr>
<tr>
<td>VC</td>
<td>5.77</td>
<td>62.95</td>
<td>V%(\times(1.785190))</td>
<td>V%(\times(1.785190))</td>
</tr>
<tr>
<td>V(<em>{2})O(</em>{5})</td>
<td>3.36</td>
<td>181.88</td>
<td>V%(\times(1.785190))</td>
<td>V%(\times(1.785190))</td>
</tr>
<tr>
<td>Mo</td>
<td>10.20</td>
<td>95.94</td>
<td>Mo%(\times(0.888740))</td>
<td>Mo%(\times(0.888740))</td>
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<tr>
<td>MoC</td>
<td>8.90</td>
<td>203.89</td>
<td>MoC%(\times(0.894109))</td>
<td>MoC%(\times(0.894109))</td>
</tr>
<tr>
<td>MoO(_{3})</td>
<td>8.20</td>
<td>107.95</td>
<td>MoC%(\times(0.894109))</td>
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<td>MoO(_{3})</td>
<td>4.69</td>
<td>143.94</td>
<td>MoC%(\times(0.894109))</td>
<td>MoC%(\times(0.894109))</td>
</tr>
</tbody>
</table>

Specification for
Hard-Coat Anodizing of Magnesium for Engineering
Applications¹

This standard is issued under the fixed designation B 893; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers requirements for electrolytically formed oxide coatings on magnesium and magnesium alloy parts where appearance, abrasion resistance, and protection against corrosion are important.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 117 Practice for Operating Salt Spray (Fog) Apparatus²
B 244 Test Method for Measurement of Thickness of Anodic Coatings on Aluminum and of Other Nonconductive Coatings on Nonmagnetic Basis Metals with Eddy-Current Instruments³
B 322 Practice for Cleaning Metals Prior to Electroplating³
B 374 Terminology Relating to Electroplating³
B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section³
B 537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure³
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings³
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings³
B 762 Test Method of Variables Sampling of Metallic and Inorganic Coatings³
D 3951 Practice for Commercial Packaging⁴

D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser⁵

2.2 ISO Standard:
ISO 2080 Electroplating and Related Processes - Vocabu-

lary⁵

3. Terminology

3.1 Definitions—For definitions of terms relating to this specification see Terminology B 374.

3.1.1 anodizing—an electrolytic oxidation process in which the surface of a metal, when anodic, is converted to a coating having desirable protective or functional properties.

3.1.2 hard coat—in anodizing magnesium, an anodic oxide coating on magnesium with a higher apparent density and thickness, and a greater resistance to wear than the base metal.

4. Classification

4.1 Coating Designation—Thickness.

4.1.1 Minimum Thickness of 20 µm.

4.2 TYPE - Post Treatments.

4.2.1 TYPE A—no post treatment.

4.2.2 TYPE B—Purchaser Specified.

5. Ordering Information (to be supplied by the purchaser to the producer.)

5.1 Alloy Designation—When ordering articles anodized in accordance with this specification, the purchaser shall state, the alloy designation number.

5.2 Appearance—Unless otherwise specified by the purchaser, an off-white color shall be acceptable for TYPE A coating. The purchaser shall specify the color and surface appearance required for TYPE B coatings. All coatings shall be uniform in color and free from stains. Alternatively, samples showing the required finish, or range of finishes, shall be supplied or approved by the purchaser. When required, the basis material may be subjected to such mechanical polishing as may be required to yield the desired final surface characteristics.

¹ This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.07 on Chromate Conversion Coatings.


² Annual Book of ASTM Standards, Vol. 03.02.
³ Annual Book of ASTM Standards, Vol. 02.05.
⁵ Annual Book of ASTM Standards, Vol. 06.01.
5.3 Significant Surface—The areas of the article covered by the coating, for which the coating is essential for service or appearance or both.

5.3.1 Contact Marks—Contact marks will occur. The Purchaser shall specify where contact marks are unacceptable.

5.4 Tolerances—Dimensional build-up is approximately one-half of anodic film thickness. The order document shall include any coating thickness tolerances and shall not exceed any applicable drawing dimensions.

5.5 The purchaser shall provide the number for this standard TYPE.

5.6 The purchaser shall state any special post treatments. (see 4.2 and 5.2)

5.7 Test Methods—The purchaser shall state the test method(s) by which the coated article will be evaluated (see Section 8).

5.8 Sampling Plan—see Section 9.

5.9 Any requirement for certification (see Section 11).

5.10 Any requirement for packaging (see Section 12).

6. Materials and Process

6.1 Process

6.1.1 Basis Metal—This standard does not specify requirements for the surface condition of the basis metal before anodizing, but agreement should preferably be reached between the purchaser and the producer that the surface condition of the basis metal is satisfactory.

6.1.2 Surface Preparation—Preparatory procedures and cleaning of the basis material may be necessary, see Practice B 322.

6.1.3 Hard-Coating—Following the preparatory operations, the articles are introduced into the solution for a period of time at the current density and temperature required to produce the hard-coated surface.

6.2 Post Treatments:

6.2.1 Final Rinsing—Rinsing subsequent to anodizing is necessary to remove all traces of the electrolyte that may affect the appearance and performance of the part. Deionized or distilled water may be used to avoid water spots.

6.2.2 Post Treatment—Surface sealers or topcoats may be specified to reduce friction, add color, or further increase performance of the article.

7. Requirements

7.1 Acceptance:

7.1.1 Visual Defects—The significant surfaces of the article to be hardcoated shall be free of clearly visible defects such as pits, roughness, striations, or discoloration when examined with normal or corrected to 20/20 eyesight at a distance of approximately 0.5 meter.

7.1.2 Thickness

7.1.2.1 Coating Thickness for SC20—The anodic film thickness shall be a minimum of 20 um and not exceed 30 um on the significant surface.

7.1.2.2 Test—Hard-coating thickness shall be evaluated by one or more of the following test methods in Section 8.

7.2 Qualification Tests—The process shall be evaluated monthly or more frequently if required by the purchaser using the following test methods on panels that are of the same alloy of the parts coated with TYPE A hard coat.

7.2.1 Corrosion Test—Use method described in 8.1

7.2.2 Abrasion Resistance Test—Use the Taber Abraser test method in Annex A1 of this specification.

8. Test Methods

8.1 Corrosion Test—Panel(s) shall be subjected to a 5 percent salt spray (fog) test in accordance with Practice B 117 for 336 h. The panel(s) shall be prepared and evaluated in accordance with Practice B 537. A Protective Rating of 6 or less is considered a failure.

8.2 Abrasion Resistance Test—Test panels in accordance with Annex A1. The acceptance criteria shall be as follows.

8.2.1 Abrasive Wheel No. CS-10—A wear index of more than 7 mg/1000 cycles or less than 10 000 wear cycles shall be considered a failure.

8.2.2 Abrasive Wheel No. CS-17—A wear index of more than 20 mg/1000 cycles or less than 5 000 wear cycles shall be considered a failure.

8.3 Thickness Tests:

8.3.1 Eddy-Current Method (Test Method B 244)

8.3.2 Microscopical Cross Section Method (Test Method B 487).

9. Sampling

9.1 The purchaser and producer are urged to employ statistical process control in the coating process. Properly performed, statistical process control will assure coated products of satisfactory quality and will reduce the amount of acceptance inspection. The sampling plan used for the inspection of the quality coated article shall be agreed upon between the purchaser and producer.

9.1.1 When a collection of coated articles (inspection lot, see 8.2) is examined for compliance with the requirements placed on the articles, a relatively small number of the articles (sample) is selected at random and is inspected. The inspection lot is then classified as complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria for compliance are determined by the application of statistics. The procedure is known as sampling inspection. Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for sampling inspection of coatings.

9.1.2 Test Method B 602 contains four sampling plans, three for use with tests that are non-destructive and one when they are destructive. Method B 602 provides a default plan if one is not specified.
9.1.3 Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. Guide B 697 provides a default plan if one is not specified.

9.1.4 Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The test must yield a numeric value and certain statistical requirements must be met. Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. Method B 762 provides a default plan if one is not specified.

9.2 An inspection lot shall be defined as a collection of coated articles that are the same kind, that have been produced to the same specification, that have been coated by a single supplier at one time or approximately the same time, under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

10. Rejection and Hearing

10.1 Parts that fail to conform to the requirements of this specification may be rejected. Rejection should be reported to the producer or supplier promptly, and in writing. In case of dissatisfaction with test results, the producer or supplier may make a claim for a rehearing.

11. Certification

11.1 When specified in the purchase order or contract, the purchaser shall be furnished certification that samples representing each lot have been tested or inspected as directed in this specification and that the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

12. Packaging

12.1 If packaging requirements are necessary under this specification, they shall be in accordance with Designation D 3951.

13. Keywords

13.1 anodizing; hard coat; magnesium

ANNEX

(Mandatory Information)

A1. TABER ABRASER WEAR TEST METHOD

A1.1 Scope

A1.1.1 This test method will evaluate the resistance of the coating to abrasive wear. The test is performed by coating a special specimen with the coating of interest, mechanically rotating the panel under an abrasive wheel and measuring the loss of material. The test is generally performed for 5000 cycles with CS-17 or 10,000 cycles with a CS-10 but can be extended to as high as 25,000 cycles.

Note A1.1—Variations in the results of this method have been attributed to humidity in the laboratory and storage conditions of the wheels. Care should be taken to control the humidity of the wheels between tests.

A1.1.2 The results are variable between tests and therefore three (3) coated test specimens should be tested for 5000 or 10,000 cycles each depending on the type of abrasive wheel. The results should be averaged and the abrasion wear reported as the weight loss in mg/1000 cycles (Taber Wear Index).

Note A1.2—The outer surface of the coating may contain roughness or in the case of anodizing may be less dense which will cause a greater loss in the first 1000 cycles than the remaining cycles. By not reporting the first 1000 cycles a more representative value for the bulk coating is obtained. For these applications, however, the outer density and wear debris in the first 1000 cycles is important.

A1.2 Apparatus

A1.2.1 Taber Abraser Wear Testing Unit—This unit must be capable of applying a 1000 g load on the wheel and have an operating vacuum for removal of wear debris while in test.

A1.2.2 Abrasion Wheels—Use the CS10 or CS17 (Resilient Rubber) Taber Wheels. To resurface the wheels use the CS11 discs from Taber.

Note A1.3—The hardness of the wheels can change with time and can effect the reproducibility of the results. (see Test Method D 4060) Redressing the wheels as well as using new wheels and controlling the humidity will improve the reproducibility. Round Robin tests between laboratories has produced a bias of 50% with these factors in control while the intra laboratory bias is 20%.

A1.2.3 Specimens—Test Specimens shall be made from magnesium plate 4 in. × 4 in. × 0.250 in. (100 mm × 100 mm × 2 mm to 6.35 mm) with a 0.250 in. (6.35 mm) hole in the center.

A1.2.4 Analytical Balance—Scale shall be capable of measuring to 150 g ± 0.1 mg

A1.3 Procedure

A1.3.1 Coat Specimens—Coat a set of three (3) specimens to the thickness of 20 to 30 µm.

A1.3.2 Testing Specimens—Perform the following test on each of the three specimens.

A1.3.2.1 Step 1—Weigh Specimens—Cool the specimen to ambient temperature and weigh to the nearest 0.1 mg.

A1.3.2.2 Step 2—Dress Wheel—The wheel shall be dressed using a CS11 disc for 50 cycles.

A1.3.2.3 Step 3, Wear Specimens, 5,000 cycles for CS-17 or 10,000 cycles for a CS-10. Test the specimens by loading the wheel to 1000 g and wearing the specimen for the required cycles.

A1.3.2.4 Step 4, Weigh Specimen—Cool the specimen to ambient temperature and weigh to the nearest 0.1 mg.
Standard Test Methods for Evaluating the Corrosion Resistance of Powder Metallurgy (P/M) Stainless Steel Parts/Specimens by Immersion in a Sodium Chloride Solution

This standard is issued under the fixed designation B 895; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover a procedure for evaluating the ability of sintered P/M stainless steel parts/specimens to resist corrosion when immersed in a sodium chloride (NaCl) solution.

1.2 Corrosion resistance is evaluated by one of two methods. In Method 1, the stainless steel parts/specimens are examined periodically and the time to the first appearance of staining or rust is used to indicate the end point. In Method 2, continued exposure to the sodium chloride solution is used to monitor the extent of corrosion as a function of time.

1.3 This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
A 380 Practice for Cleaning and Descaling Stainless Steel Parts, Equipment and Systems
B 243 Terminology of Powder Metallurgy
B 528 Test Method for Transverse Rupture Strength of Sintered Metal Powder Specimens
D 1193 Specification for Reagent Water
G 1 Practice for Preparing, Cleaning and Evaluating Corrosion Test Specimens
G 48 Test Method for Pitting and Crevice Corrosion Resistance of Stainless Steel and Related Alloys By the Use of Ferric Chloride Solution

3. Terminology

3.1 Definitions—Useful definitions of terms for metal powders and powder metallurgy are found in Terminology B 243.

4. Summary of Test Method

4.1 Method 1 is recommended for evaluating the corrosion resistance of stainless steel powder metallurgy parts/specimens and to verify that proper materials and processing conditions were used.

4.1.1 In this method, parts/specimens are immersed in 5 % (by mass) NaCl solution and examined periodically until the first appearance of staining or rust. A part or specimen is considered to have reached the end point when the first sign of corrosion occurs.

4.2 Method 2 is recommended for evaluating the processing variables used in producing parts/specimens.

4.2.1 In this method, parts/specimens are exposed further to the NaCl solution and periodically rated as either A, B, C, or D (A-no corrosion; D-high or extreme corrosion) by comparison with Fig. 1, a photograph of corroded specimens which serves as a standard. Method 2 has been found useful in alloy screening and process optimization studies.

5. Significance and Use

5.1 The ability of sintered powder metallurgy stainless steel parts/specimens to resist corrosion when immersed in sodium chloride solution is important to their end use. Causes of unacceptable corrosion may be incorrect alloy, contamination of the parts by iron or some other corrosion-promoting material or improper sintering of the parts (for example, undesirable carbide and nitride formations caused by poor lubricant burnoff or improper sintering atmosphere).

5.2 This standard may be part of a purchase agreement between the P/M parts producer (seller) and the user of the parts (purchaser) (Method 1). It may also be used to optimize part or specimen production parameters (Method 2).

6. Apparatus

6.1 Sealable Glass or Plastic Jars, of suitable capacity for specimens to be completely covered by the NaCl solution.

6.2 Glass Beads (4 mm is recommended).

6.3 Glass Stirring Rods.

6.4 Tongs (Stainless steel or plastic, nonmetallic plated).

7. Reagents

7.1 A sodium chloride solution consisting of 5 ± 0.1 % (by
mass) NaCl shall be prepared using distilled or deionized water conforming to Specification D 1193 (Type 4) and ACS reagent grade NaCl solution. The 5 % NaCl solution shall be prepared no less than 16 h before beginning the corrosion testing.

7.2 Concentrated HCl.
7.3 Distilled or deionized water.

8. Test Specimen

8.1 Usually test parts are sintered parts, but they may also be standard transverse rupture bars as defined in Test Method B 528. A minimum of five parts/specimens shall be used for each test.
8.1.1 The density of the parts or specimens as well as any post sintering treatments, (that is, coining, repressing, machining, etc.) shall be stated. Parts or specimens shall be free of oil, dirt, grease and fingerprints. If they have been cleaned, the cleaning method shall be stated. Refer to Practices A 380 and G 1 for recommended cleaning practices.
8.1.2 The use of tongs or gloves, or both, to prevent contamination in handling is suggested.

9. Preparation of Apparatus

9.1 Soak previously used jars and glass beads in concentrated HCl for at least 12 h to remove rust stains; rinse with distilled or deionized water, then rinse again and allow to dry.
9.1.1 Place the glass beads in the bottom of the beaker. Use a sufficient number of beads to keep the test specimen off the bottom of the jar.

10. Procedure

10.1 Method 1:
10.1.1 Place one part or specimen per jar on top of the glass beads. Add the NaCl solution to each jar so that the volume of solution, in millilitres, is at least five times the mass of the specimen in grams. The distance from the surface of the part/specimen to the top of the solution should be at least 25 mm. The ratio of the volume of air to the volume of solution in a jar is recommended to be about 1:2 to 1:3. Remove air bubbles attached to the specimen surface and glass beads by swirling the solution or moving the specimen with a glass stirring rod. Close the jars. Record the date and the time of the start of the test. Store the immersed test specimens at a temperature of 21 to 24°C.
10.1.2 Examine the parts/specimens after \( \frac{1}{2} \), 1, 2, 4, and 8 h and at 24 h intervals from the onset of the test. Thereafter, the interval may be lengthened as time progresses until the first appearance of rust or stain. The corrosion life of a part or specimen is the time of the previous examination, that is, the last examination taken before the observation of stain or rust. Do not include corrosion which appears at the interface between a part/specimen and the glass beads. This is considered to be crevice corrosion and may be evaluated by Test Method G 48.

10.2 Method 2:
10.2.1 Method 2 follows the procedure of Method 1 through 10.1.1. It continues with periodic examination as in 10.1.2,
except that testing continues beyond the first appearance of stain or rust. In Method 2, for each examination, the parts/specimens are ranked for the degree of staining or corrosion according to the following:

A—The part/specimen is free from stain or rust.
B—The first sign of stain or rust appears or up to 1 % of surface is covered by stain or rust.
C—More than 1 and up to 25 % of surface is covered by stain or rust.
D—More than 25 % of surface is covered by stain or rust.

10.2.1.1 Photographs exhibiting these conditions are shown in Fig. 1.
10.2.1.2 Do not include corrosion which appears at the interface between the test specimens and the glass beads. This is considered to be crevice corrosion and is not the subject of this test method. As a part/specimen shows increasing amounts of rust, it may become necessary to use a pair of tongs to remove it from the solution so as to distinguish between loose and adherent rust. Rinsing should not be used because loose rust may be washed off.

10.2.2 Record the results of the timed observation at the intervals used. Table 1 shows a typical example of a data collection table.

10.2.3 Plot the percent of parts/specimens having an A, B or C class rating for suitable time intervals on a logarithmic time scale.

10.2.4 Draw curves to fit the data. A typical plot is shown in Fig. 2.

11. Report

11.1 Method 1:

11.1.1 Report the individual corrosion lives in hours as determined in 10.1.2 and calculate the arithmetic average. Acceptance criteria shall be mutually agreed upon between purchaser and seller.

11.1.2 Density of parts/specimens as well as any post-sintering treatments and cleaning methods (if cleaned) shall be recorded.

11.2 Method 2:

11.2.1 Report the time when 50 % of the parts/specimens have an A, B, or C class rating.

11.2.2 Photographs of tested parts, indicating the test conditions and the number of hours tested shall be provided at the customers request.

11.2.3 Density of parts or specimens as well as any post-sintering treatments and cleaning methods (if cleaned) shall be recorded.

12. Precision and Bias

12.1 No repeatability and reproducibility data or information on any bias inherent in these test methods are available at this time.

13. Keywords

13.1 corrosion; powder metallurgy (P/M) parts; stainless steel

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**TABLE 1 Example of Corrosion Rating Chart for a Set of Ten Replicate Specimens of Sintered 316L Stainless Steel**

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</table>

% Holding 'A' Rating

| 100 | 100 | 100 | 100 | 100 | 100 | 90  | 70  | 50  | 10  | 0   |

% Holding 'B' Rating

| 100 | 90  | 60  | 10  | 0   |

% Holding 'C' Rating

| 100 | 90  | 80  | 70  | 40  | 10  |

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*aSee Section 10.1.1 for definition of ratings.*
FIG. 2 Plot of Percentage of Replicate Specimens with a Given Rating Versus Immersion Time

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Standard Specification for Autocatalytic Nickel over Autocatalytic Copper for Electromagnetic Interference Shielding

This standard is issued under the fixed designation B 904; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

This specification presents the requirements for multilayer coatings of autocatalytic nickel-phosphorus over autocatalytic copper on metallic and polymeric substrates. The coating system is intended to provide electromagnetic interference (EMI) protection properties or electrostatic discharge (ESD) protection to parts fabricated from either polymeric or metallic materials.

2. Referenced Documents

2.1 ASTM Standards:
A 919 Terminology Relating to Heat Treatment of Metals
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 252 Guide for Preparation of Zinc Alloy Die Castings for Electroplating and Conversion Coatings
B 253 Guide for Preparation of Aluminum Alloys for Electroplating
B 320 Practice for Preparation of Iron Castings for Electroplating
B 322 Practice for Cleaning Metals Prior to Electroplating
B 374 Terminology Related to Electroplating
B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
B 523 Specification for the Appearance of Electroplated Plastic Surfaces
B 532 Guide for Preparation of Plastics Materials for Electroplating
B 727 Practice for Preparation of Plastics Materials for Electroplating
B 733 Specification for Autocatalytic Nickel-Phosphorus Coatings on Metals
B 568 Test Method for Coating Thickness by X-Ray Spectrometry
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
B 733 Specification for Autocatalytic Nickel-Phosphorus Coatings on Metals
D 3330/D 3330M Test Methods for Peel Adhesion of Pressure-Sensitive Tape at 180° Angle
D 3359 Test Methods for Measuring Adhesion by Tape Test
D 4935 Test Method for Measuring the Electromagnetic Shielding Effectiveness of Planar Materials

2.2 Military Standard:
MIL-STD-461 Electromagnetic Emission and Susceptibility Requirements for the Control of Electromagnetic Interference

3. Terminology

3.1 Definitions—Many of the terms used in this specification can be found in Terminologies A 919 or B 374.

3.2 Definitions of Terms Specific to This Standard:
3.2.1 significant surfaces, n—these surfaces are classified as primary, secondary, nonsignificant, and coating-free surfaces.
3.2.1.1 coating-free areas, adj—areas specified on part drawings or suitably marked samples.
3.2.1.2 nonsignificant surfaces, adj—all holes, recesses, and other areas where a controlled deposit cannot be obtained under normal coating conditions and that cannot be touched with a 20-mm diameter ball shall be considered nonsignificant surfaces unless otherwise specified on part drawings or suitably marked samples.
3.2.1.3 primary significant surface, adj—all mating surfaces and those other surfaces specified on part drawings or suitably marked samples.
3.2.1.4 secondary significant surfaces, adj—all surfaces,
other than primary significant surfaces, that can be touched with a 20-mm diameter ball shall be considered secondary significant surfaces unless otherwise specified on part drawings or suitably marked samples.

4. Classification

4.1 This classification system provides for the following:

4.1.1 Types of coating based on thickness and testing requirements, and
4.1.2 Grades of coating based on alloy composition.

4.2 Coating Type:

4.2.1 The coating type indicates the type of application and tests to be used in determining the acceptance of the coating.

4.2.2 Coating Type Definitions:

4.2.2.1 Type 1—Coatings intended to shield devices for FCC/VDE Class A requirements.
4.2.2.2 Type 2—Coatings intended to shield devices for FCC/VDE Class B service for MIL-STD-461 requirements.
4.2.2.3 Type 3—Coatings intended to shield devices for FCC/VDE Class B service in harsh environments.
4.2.2.4 Type 4—Coatings intended to shield devices for FCC/VDE Class A requirements.

4.2.3 The description of Types 1, 2, 3, and 4 is summarized in Table 1.

4.3 Coating Grade:

4.3.1 The coating grade is based upon phosphorus content.

NOTE 1—The coating grade indicates the relative contact impedance and the relative corrosion resistance of the nickel-phosphorus coating and tests to be used in determining the acceptance of the coating.

4.3.2 Coating Grade Definitions:

4.3.3 Grade 1 coatings have a phosphorus content between 1 and 5 

NOTE 2—Low phosphorus coatings exhibit low electrical contact impedance. High phosphorus contents exhibit somewhat higher contact impedance, however, the coating is more corrosion resistant.

4.3.4 Grade 2 coatings have a phosphorus content between 6 and 11 

NOTE 3—The adhesion and resistance to blistering are improved on some polymeric substrates by an initial flash deposit of autocatalytic nickel.

5. Ordering Information

5.1 To avoid misunderstanding between contractual parties, purchase orders or contracts for autocatalytic nickel over autocatalytic copper coatings under this specification should include the designation, issue date, and the following information:

5.1.1 Type of substrate.
5.1.1.1 Metallic substrates should state the composition and metallurgical condition. Assemblies of dissimilar materials should be identified.
5.1.1.2 Polymeric substrates should state the polymer type and should be of a plating grade.
5.1.2 Classification of the deposit by type and grade.
5.1.3 Primary significant surfaces and coating-free surfaces must be indicated on drawings.
5.1.4 Any special requirements.
5.1.5 Test methods for coating adhesion, thickness, porosity.
5.1.6 Sampling program.

6. Surface Preparation

6.1 Surface Contamination—Surfaces of polymeric parts must be free of all mold release agents, dirt, oil, grease, and contamination detrimental to the final finish. Surfaces of metallic parts must be free of all scale, oxidation, and contamination detrimental to the final finish. A clean surface is essential to the adhesion and electrical conductivity requirements of the subsequent coated part.

6.2 Cleaning, Conditioning, and Activating—Any adequate method of cleaning, conditioning, and activating is acceptable provided the coated parts meet the inspection requirements and are free of distortion. Examples of adequate methods of cleaning can be found in 10.2.

6.2.1 Base Material Suitability—The parts to be coated shall be inspected by the coater prior to any processing to determine their suitability for coating. Unsuitable parts shall be returned to the fabricator or molder.

6.3 Mechanical Roughening—Mechanical roughening of polymer surfaces, to promote adhesion, may only be used when specified on the part drawing.

7. In-Process Storage and Handling

7.1 Following cleaning, conditioning, and activating, all parts shall be immediately coated with copper and then nickel-phosphorus to the thickness specified in Table 1. The parts processing cycle shall be a continuous operation without any interruption.

7.2 Handling—The parts shall be suitably racked so as to prevent gas entrapment and to avoid physical handling of the primary significant surfaces.

7.3 Drying—Following coating, the parts may be dried with warm air currents. Drying temperature shall not exceed the heat distortion temperature of the substrate. Wetting agents may be used to enhance water shedding provided they do not interfere with subsequent paint adhesion.

7.4 Storage—Following drying, all parts shall be stored in a clean dry area, protected from corrosive fumes and humidity prior to packaging and shipment.

8. Inspection

8.1 Process Qualification—All nickel-phosphorus over copper coatings shall be produced from processes qualified in accordance with the requirements in Section 9.
8.2 Nickel-Phosphorus Over Copper Coating—The nickel-phosphorus coating shall meet the requirements of Specification B 733.

8.2.1 Appearance—The nickel-phosphorus over copper coating shall be smooth, semi-bright, adherent, and free from defects that will impair the corrosion resistance, electrical conductivity or electromagnetic shielding effectiveness properties of the coating, see ASTM B 532 for polymeric parts and Specification B 733 for metallic parts.

8.2.2 Blisters and Unplated Areas

8.2.2.1 Blisters—The parts shall be examined visually for 10 to 15 s or as necessary to adequately examine the entire plated surface at a distance of 600 to 900 mm (arms length) for evidence of blisters. Visually means 20/20 vision or corrected to 2/20. Parts exhibiting blisters shall be rejected.

8.2.2.2 Unplated Areas—Voids, skips, and other unplated areas, visible to the unaided eye, exposing the substrate shall be limited to the sizes and numbers shown in Table 2. Voids and skips exposing copper are not permitted.

8.2.3 Thickness—The thickness test shall be performed on the primary and secondary significant surfaces of the finished part. Thickness shall be as specified in Table 1. For parts acceptance the combined thickness of deposit and tolerance shall be held at least 25 and no more than 200 mm apart.

8.2.3.2 Restrictions—The following test methods are suitable for measuring total thickness of nickel-phosphorus and copper coating, see Practice B 554. See 8.2.3.2 for restrictions.

Coulometric Method—See Test Method B 504
X-Ray Method—See Test Method B 568
Beta Backscatter Method—See Test Method B 567
(a) Coulometric Method—This semi-destructive method is suitable for the measurement of individual layers in the range of 0.25 to 100 µm.
(b) X-Ray Method—This nondestructive method is suitable for the measurement of individual layers in the range of 0.25 to 65 µm and shall be the referee method.
(c) Beta Backscatter Method—This nondestructive method is suitable for measuring total coating thickness between 0.1 and 100 µm.

8.2.3.2 Restriction—Electronic thickness testers utilizing the Eddy-current principle are not suitable for this specification and shall not be used.

8.2.4 Adhesion—The coatings shall not peel or separate from the base material when subjected to the tape test (see 9.4.1).

8.2.5 Electrical Continuity—The coating shall form a continuous electrical path across the significant and nonsignificant surfaces.

8.2.5.1 Electrical Criteria—The DC resistance of the coating system, between all points of the primary significant surfaces shall not exceed 0.1 Ω unless otherwise specified on the part drawings. The measurement shall be made with an ohmmeter, having a sensitivity of at least 20 000 Ω/V, and a measuring voltage of 9 ± 3 V DC and a 2 A load.

8.2.5.2 Electrical Measurement Procedure—The DC resistance of the coating system, shall be measured on a test sample prepared by assembling two coated parts together, clamping them with 2 M4, class 4.8 threaded fasteners assembled with two flat washers and a mating nut torqued to 1.5–2.0 NM. The measuring probes shall each be pressed firmly into the opposing faces of the assembly so as to make intimate contact with the coating. Alternatively, when it is not practical to assemble two parts together for this test, a single part may be used. In that event, press one probe firmly into the surface of the part so that it is in intimate contact with the coating. The second probe shall be held in contact with the part surface, in a position and with a pressure approximating that of writing with a pencil. This probe shall have a radius of 1 mm maximum. The probes shall be held at least 25 and no more than 200 mm apart.

8.3 Sampling—A suitable sampling plan may be selected from those in Test Method B 602. Guidance in selecting a suitable sampling plan will be found in Guide B 697.

9. Requirements for Process Qualification

9.1 Process Selection—Commercial processes are available that meet the requirements of this specification.

9.2 Coating Composition—The composition of the nickel-phosphorus coating shall be 3 to 11 % phosphorus and the remainder nickel. The composition of the copper coating shall be 99 % copper minimum.

9.3 Electrical Integrity—Parts or coupons shall be subject to 20 cycles of the cyclic temperature-humidity test and then to the requirements of 8.2.5.1 and 8.2.5.2.

9.4 Adhesion—The coatings shall not peel or separate from the base material when subjected to the following tests.

9.4.1 Tape Test:

9.4.1.1 Apply a piece of pressure sensitive filament tape, approximately 25 mm wide and 75 mm long, onto the coated surface, pressing it firmly into place. Remove the tape, within 5 minutes, with a continuous, smooth and rapid pull at an angle of approximately 90°.

9.4.2 The filament tape shall be 25 ± 1 mm wide semitransparent pressure-sensitive tape with an adhesion strength of 44.6± 2.8 g/mm width when tested in accordance with Test Method D 3330/D 3330M. The adhesion shall not change more than ± 6.5 % of its mean value within 12 months.

9.4.2.1 Pull/Fail Criteria—Removal of any metal coating shall constitute test failure.

9.4.3 Adhesion-Cross Hatch—A series of six parallel cuts approximately 25 mm long and 3 mm apart are made through the coating by means of a sharp knife. Six similar cuts then are superimposed perpendicular to the original. All cuts must penetrate to the base material.

9.4.3.1 Procedure—Press a strip of pressure-sensitive filament tape approximately 25 mm wide and 75 mm long onto the

<table>
<thead>
<tr>
<th>TABLE 2 Allowable Unplated Areas</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area</td>
</tr>
<tr>
<td>Mating surface areas</td>
</tr>
<tr>
<td>Nonmating area</td>
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</tbody>
</table>
area formed by the cuts in the coating. Gently rub the tape into place and then remove the tape at an angle of 90° to the coated surface with a rapid pull. Examine the part and the tape for any coating removal.

9.4.3.2 Pass/Fail Criteria—Removal of more than 20% (estimated) metal removal in any 3 of the 25 squares shall constitute test failure.

9.5 Thermal Shock Resistance—The thermal shock resistance of the coating system shall be evaluated by subjecting coated test coupons to a thermal shock test. A second set of test coupons shall be subjected to the cross hatch adhesion test (see 9.4.3).

9.5.1 Pass/Fail Criteria—Coupons subjected to the thermal shock test shall be stabilized for 30 minutes at room temperature and then examined at 10X magnification. Any evidence of chips, cracks, or delaminations shall constitute test failure. Coupons subjected to the cross hatch adhesion test shall be examined visually for coating removal. More than 50% coating removal in any 3 of the 25 squares shall constitute test failure.

9.6 Thermal Cycling Test—Test specimens shall be subjected to Test Method B 553, service condition 2 for 3 cycles.

9.7 Number of Samples—The number of samples tested for each test shall be selected from the sampling plans of Test Method B 602.

9.8 Shielding Effectiveness—Parts or coupons shall meet the shielding effectiveness requirements shown in Table 1 after being subjected to 20 cycles of the cyclic temperature-humidity test when tested to Test Method D 4935.

10. Surface Preparation Recommendations

10.1 Preparation of Metal Surfaces—The following practices describe preparation methods that have been determined to be adequate: B 183, B 242, B 252, B 253, B 320, B 322.

10.2 Preparation of Polymeric Surfaces—Practice B 727 describes a preparation method that has been determined to be adequate.

11. Platable Polymeric Materials

11.1 Plating grades of the following polymeric types can be plated to this specification:

11.1.1 Polycarbonate;
11.1.2 Modified Polyphenylene Oxide;
11.1.3 Acetal;
11.1.4 Polysulfone;
11.1.5 Acrylonitrile-Butadiene-Styrene;
11.1.6 Polyphenolene ether;
11.1.7 Polystyrene;
11.1.8 Nylon;
11.1.9 Polyester; and
11.1.10 Styrene-Malic-Anhydride.

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Standard Test Methods for Assessing the Adhesion of Metallic and Inorganic Coatings by the Mechanized Tape Test

1. Scope

1.1 These test methods describe procedures for assessing the adhesion of metallic and inorganic coatings and other thin films to metallic and nonmetallic substrates. Assessment is made by applying pressure-sensitive tape to a coated surface and then utilizing a mechanical device to remove the tape at a regulated, uniform rate and constant angle while simultaneously recording the removal force.

1.2 Four methods are described. Methods A1 and A2 are intended primarily for use on parts. Methods B1 and B2 are intended primarily for use in laboratory evaluations. Methods B1 and B2 are not recommended for testing coatings and films on polymer substrates.

1.3 These test methods may be used to establish whether the adhesion of a coating to a substrate is within a required range (between a quantified low and a quantified high level). Determination of actual adhesive forces requires more sophisticated methods of measurement. In multilayer systems adhesion failure may occur between intermediate coating layers so that the adhesion of the total coating system to the substrate may not necessarily be determined.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 183 Practice for Preparation of Low-Carbon Steel for Electroplating
B 242 Practice for Preparation of High-Carbon Steel for Electroplating
B 252 Guide for Preparation of Zinc Alloy Die Castings for Electroplating and Conversion Coating
B 253 Guide for Preparation of Aluminum Alloys for Electroplating
B 254 Practice for Preparation of and Electroplating on Stainless Steel
B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coating
B 319 Guide for Preparation of Lead and Lead Alloys for Electroplating
B 320 Practice for Preparation of Iron Castings for Electroplating
B 343 Practice for Preparation of Preparation of Nickel for Electroplating with Nickel
B 480 Guide for Preparation of Magnesium and Magnesium Alloys for Electroplating
B 481 Practice for Preparation of Titanium and Titanium Alloys for Electroplating
B 482 Practice for Preparation of Tungsten and Tungsten Alloys for Electroplating
B 537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure
B 538 Method of FACT (Ford Anodized Aluminum Corrosion) Test
B 558 Practice for Preparation of Nickel Alloys for Electroplating
B 629 Practice for Preparation of Molybdenum and Molybdenum Alloys for Electroplating
B 630 Practice for Preparation of Chromium for Electroplating with Chromium
B 727 Practice for Preparation of Plastics Materials for Electroplating
D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting
D 1731 Practices for Preparation of Hot-Dip Aluminum Surfaces for Painting
D 1732 Practices for Preparation of Magnesium Alloy Surfaces for Painting
D 2370 Test Method for Tensile Properties of Organic Coatings
D 3330/D 3330M Test Methods for Peel Adhesion of Pressure-Sensitive Tape at 180° Angle
D 3359 Test Methods for Measuring Adhesion by Tape Test

3. Summary of Test Method

3.1 Pressure-sensitive tape is adhered to the surface of the
coating and then removed utilizing a motorized mechanical device that peels the tape at a constantly maintained angle and controlled rate of peel. A digital recording force gage is used to record the maximum peel force.

Note 1—All due care must be taken to ensure that test specimens are handled and stored such that they are not subjected to conditions that will cause deleterious effects. These conditions include but are not limited to handling without the use of gloves, storing in areas that accumulate dust, areas of high humidity or where the sample may be subjected to fumes or vapors that might condense on the sample.

3.2 Methods A1 and A2:
3.2.1 In these methods, which are nondestructive, the measurement area used is the unbroken coating surface with peel angles of 90° and 180° respectively.

3.2.2 Adhesion is assessed in terms of “passed,” if the coating does not detach, or “failed,” if the coating detaches within the specified range of peel forces as recorded during the test.

3.3 Methods B1 and B2:
3.3.1 In these methods, which are destructive, the measurement area used is a broken coating surface created by scoring a lattice pattern through the coating to the substrate and peeling at angles of 90° and 180° respectively.

3.3.2 Adhesion is assessed qualitatively on the 0 to 5 scale.

4. Significance and Use
4.1 If a coating is to fulfill its function of protecting or imparting unique properties to the surface of a substrate, it must adhere to the substrate for the expected service life. Because surface preparation (or lack of it) has a drastic effect on adhesion of coatings, a test method for evaluating adhesion to different surface treatments or of different coatings to the same treatment is of considerable use to the industry.

4.2 The limitations of all adhesion methods, and the specific limitation of this method to lower levels of adhesion (see 1.3) should be recognized before using it. These test methods are mechanized adaptations of Test Methods D 3359; therefore, the intra- and inter-laboratory precision of these test methods are similar to Test Methods D 3359 and to other widely-accepted tests for coated substrates, for example, Test Method D 2370, but this is partly the result of it being insensitive to all but large differences in adhesion. The pass-fail scale of 0 to 5 for Method B1 and B2 was selected deliberately to avoid a false impression of being sensitive.

5. Apparatus and Materials
5.1 Peel Test Fixture—The fixture shall consist of a frame to which the specimen is rigidly clamped, and a moveable beam by which the tape is pulled off under a constant peel angle of 90° (Method A1 and B1) or 180° (Method A2 and B2). The peel rate should be controllable between 20 mm/s and 200 mm/s for Method A1 and B1 and between 14 mm/s and 140 mm/s for Methods A2 and B2. A recording force gage is fitted between the tape grip and the movable beam (see Fig. 1).

5.2 Pressure-Sensitive Tape—Unless otherwise specified in the document referencing this test, the tape shall be 25 mm wide, semitransparent, pressure-sensitive tape with an adhesion strength of 43 ± 5.6 g/mm or N/100 mm width when tested in accordance with D 3330/D 3330M. The adhesion shall not change by more than + 6.5 % of its mean value within 12 months. The backing of the tape may consist of fiber-reinforced cellulose acetate, unplasticized poly (vinyl chloride), or polyester film. When results obtained in different laboratories do not agree it is recommended that the test be repeated using tape from the same batch.

5.3 Roller—The roller, which is hand operated, consists of a steel roller 85 ± 2.5 mm in diameter and 45 ± 1.5 mm in width, covered with rubber approximately 6 mm in thickness, having a Shore scale A durometer hardness of 80 ± 5. The surface of the roller shall be a true cylinder void of any concave or convex deviations. The mass of the roller shall be 2040 ± 45 g.

Note 2—A standardized roller is used in place of the pencil eraser of Test Methods D 3359 because of the variety of rubber and abrasives formulations used to make pencil erasers. A further consideration was the extended range of localized pressures that could be exerted by the pencil and eraser.

TEST METHOD A
NONDESTRUCTIVE, PARTS TAPE TEST

6. Test Specimen
6.1 Parts—This test normally is performed on parts. Any requirements for test specimens will be found in the document specifying their use.

Note 3—When specified by the document referencing this test, the coated parts shall be subjected to a preliminary exposure, such as water immersion, salt spray, or humidity, before conducting the tape test.

7. Procedure
7.1 Test Area—Select a flat area, on a significant surface, free of blemishes and minor surface imperfections. Ensure that the surface is clean and dry. Extremes in temperature or relative humidity may affect the adhesion of the tape or the coating.

7.2 Tape Section—Remove two complete laps of the pressure-sensitive tape from the roll and discard. Remove an additional length at a steady (that is, not jerked) rate and cut a piece off at least 100 mm long plus an additional length equal to the space between the test surface and the grip on the fixture for pulling the tape.

7.3 Tape Placement—Carefully place the tape on the area of coating to be tested and lightly press and smooth the tape into place, taking care to prevent any entrapment of air bubbles between the tape and the coating. Once the tape is in place, roll the tape firmly; once in each lengthwise direction (see 5.3). The color under the semi-transparent tape is a useful indication of when good contact has been made.

7.4 Time, Rate, and Angle:
7.4.1 Within 90 ± 30 s of the tape application, fasten the specimen onto the fixture, placing the free end of the tape in the pulling grip. The apparatus should be set to maintain the normal 90° peel angle, Method A1 (see Notes 4 and 5).

7.4.2 Pull the tape at a constant rate until only about 20 mm of tape remains in contact with the coating. The residual

6 Available from the Pressure-Sensitive Tape Council (PSTC), 104 Wilmot Rd., Suite 201, Deerfield, IL 60015.
adhering tape can be removed manually after assessing adhesion on the area peeled off. This final removing of the residual tape is not a constituent of the adhesion test.

**NOTE 4**—As far as possible, preference should be given to Method A1 (90° peel angle) due to the stress-strain behavior of the tape. If the coating to be tested is not approachable for 90° testing, for example, at the wall side inside a housing, Method A2 (180° peel angle) may be applied.

**NOTE 5**—At a peel angle of 90°, the peel rate of the tape from the coating surface is equal to the pull rate, that is, the velocity by which the free end of the tape is moved in the loading direction, whereas in a peel angle of 180°, the peel rate of the tape is only half the pull rate.

### 7.5 Peel Force Control

Read the maximum value of peel force recorded from the force registration gauge. If the force value recorded is greater than 2.5 N ± the force value specified in the document referencing this test, the test shall be repeated. Use a correspondingly lower or higher rate of peel until the maximum value is within ± 2.5 N.

**NOTE 6**—For metallic coatings on polymeric substrates, the typical force range is:

- 15 N – 25 N for Method A1
- 20 N – 35 N for Method A2

### 7.6 Evaluation

Inspect both the area of coating and the tape for evidence of flaking or detachment. Repeat the test at another area of the part. Perform sufficient tests to ensure that the adhesion evaluation is representative of the whole surface.

### 8. Report

8.1 The test report shall contain the following:

- 8.1.1 Method (A1 or A2),
- 8.1.2 Type of coating,
- 8.1.3 Any intermediate layers,
- 8.1.4 Substrate material,
- 8.1.5 Type of tape used,
- 8.1.6 Peel angle,
- 8.1.7 Rate of peel,
- 8.1.8 Any environmental exposure, and
- 8.1.9 Any failure at intermediate layers.

8.2 Report the test results as:

\[
\frac{p}{F_{\text{max}}} \quad (1)
\]

or

\[
\frac{f}{F_{\text{max}}} \quad (2)
\]

where:

- \(p\) = passed (no coating detachment),
- \(f\) = failed (coating detachment), and
- \(F_{\text{max}}\) = maximum registered peel force.

### 9. Precision of Test Method A

9.1 An interlaboratory study of this test method reviewed tape peel measurements on various coatings under several peel
conditions. The results showed that maximum peel force of the tape depended significantly on the type of coating, the type of tape, the peel rate and the peel angle. There was an insignificant relationship to the pressure used for adhering the tape to the coating.

9.2 The results of coating adhesion assessments obtained on different coatings should only be compared when the maximum deviation recorded, does not exceed 2.5 N.

9.3 The results of coating adhesion assessments obtained with Method A1 should not be compared with the results obtained with Method A2.

TEST METHOD B
DESTRUCTIVE, CROSS CUT TAPE TEST
FOR LABORATORY INVESTIGATION

10. Apparatus and Materials

10.1 In addition to the apparatus and materials in 5.1 the following also are required:

10.1.1 Cutting Tool—A sharp razor blade, scalpel, knife or other cutting device having a cutting edge angle between 15 and 30° that will make either a single cut or several cuts at once. It is of particular importance that the cutting edge be in good condition.

10.1.2 Cutting Guide—If cuts are made manually (as opposed to a mechanical apparatus) a steel or other hard metal straight edge or template should be used to ensure straight cuts.

10.1.3 Rule—A tempered steel rule graduated in 0.5 mm units is required for measuring individual cuts.

10.1.4 Illumination—A light source is recommended for determining whether the cuts have been made through the film to the substrate.

10.1.5 Magnifying Glass—An illuminated magnifier to be used while making individual cuts and examining the test area.

11. Test Specimen

11.1 Test panels shall be approximately 75 by 150 mm and at least 0.4 mm thick. Apply the coating and preparation procedure to be tested to the panels of the composition and surface conditions on which it is desired to determine adhesion.

NOTE 7—Information on test panels and surface preparation methods are given in Practices B 183, B 242, B 254, B 281, B 320, B 343, B 481, B 482, B 537, B 629, B 630, B 727, D 1730, D 1731, and D 1732; Guides B 252, B 253, and B 480; and Method B 538.

12. Procedure

12.1 Test Area—Select an area free of blemishes and minor surface imperfections. Assure that the surface is clean and dry. Extremes in temperature or relative humidity may affect the adhesion of the tape or the coating.

12.2 Scribing the First Series of Lines—Place the panel on a firm base and under the illuminated magnifier make parallel cuts as follows:

12.2.1 Space the cuts 4 mm apart and make six cuts (see Note 8). Make all cuts about 20 mm long. Cut through the film to the substrate in one steady motion using just sufficient pressure on the cutting tool to have the cutting edge reach the substrate. When making successive single cuts with the aid of a guide, place the guide on the uncut area. After making the six cuts, brush the film lightly with a soft brush or tissue to remove any detached flakes or ribbons of coating.

NOTE 8—Scribed lines in metallic coatings often result in vertical displacements from the coating surface. These displacements interfere with the proper adhesion of the tape and the circumscribed areas particularly when closer than 4 mm apart. This can result in questionable adhesion passing this test.

12.3 Scribing the Second Series of Lines—Prior to scribings the second series of lines examine the cutting edge and, if necessary, remove any flat spots or wire-edge by abrading lightly on a fine oil stone. Cut the second series of six lines spaced 4 mm apart at 90° to and centered on the first series of lines (see Note 8). Brush the area as before and inspect the incisions for reflection of light from the substrate. If the substrate has not been reached make another grid in a different location.

12.4 Tape, see 7.2.

12.5 Tape Placement—Carefully place the tape over the cross-hatch area of the coated test panel and lightly press and smooth the tape into place, taking care to prevent any entrapment of air bubbles between the tape and the coating. Once the tape is in place, roll the tape firmly once in each lengthwise direction see 5.3). The color under the semitransparent tape is a useful indication of when good contact has been made.

12.6 Time, Rate, and Angle—Within 90 ± 30 s of the tape application, fasten the test panel onto the fixture placing the free end of the tape in the pulling grip. The apparatus should be set to maintain the normal 90° peel angle, Method B1 (see Notes 4 and 8). Pull the tape at a constant rate until only about 20 mm of tape remains in contact with the coating. The residual adhering tape can be removed manually after assessing adhesion on the area peeled off. This final removing of the residual tape is not a constituent of the adhesion test.

NOTE 9—As far as possible, preference should be given to Method B1 (90° peel angle) due to the stress-strain behavior of the tape. If the coating to be tested is not approachable for 90° testing Method B2 (180° peel angle) may be applied.

12.7 Peel Force Control, see 7.5.

12.8 Evaluation—Using the illuminated magnifier, inspect the grid area for removal of coating from the substrate or from an intermediate coating. Rate the adhesion in accordance with the following scale illustrated in Fig. 2 and described as follows:

- 0 Flaking and detachment worse than Grade 1.
- 1 The coating has flaked along the edges of cuts in large ribbons and whole squares have detached. The area affected is 35 to 65 % of the lattice.
- 2 The coating has flaked along the edges and on parts of the squares. The area affected is 15 to 35 % of the lattice.
- 3 Small flakes of the coating are detached along edges and at intersections of cuts. The area affected is 5 to 15 % of the lattice.
- 4 Small flakes of the coating are detached at intersections; less than 5 % of the area is affected.
- 5 The edges of the cuts are completely smooth; none of the squares of the lattice are detached.

Flaking and detachment worse than Grade 1.

Repeat the test in two other locations on each test panel.

13. Report

13.1 The test report shall contain the following information:

13.1.1 Method (B1 or B2),

5 The edges of the cuts are completely smooth; none of the squares of the lattice are detached.
4 Small flakes of the coating are detached at intersections; less than 5 % of the area is affected.
3 Small flakes of the coating are detached along edges and at intersections of cuts. The area affected is 5 to 15 % of the lattice.
2 The coating has flaked along the edges and on parts of the squares. The area affected is 15 to 35 % of the lattice.
1 The coating has flaked along the edges of cuts in large ribbons and whole squares have detached. The area affected is 35 to 65 % of the lattice.
0 Flaking and detachment worse than Grade 1.
13.1.2 Type of coating,
13.1.3 Any intermediate layers,
13.1.4 Substrate material,
13.1.5 Type of tape used,
13.1.6 Peel angle,
13.1.7 Rate of peel,
13.1.8 Any environmental exposure, and
13.1.9 Any failure at intermediate layers.

13.2 Report the test results as follows:

\[
\frac{C}{F_{\text{max}}} = \text{Classification number according to Fig. 2, and maximum registered peel force.}
\]

**14. Precision of Method B**

14.1 The results of coating adhesion assessments obtained on different coatings should only be compared when the maximum deviation recorded, does not exceed \( \pm 2.5 \text{ N} \).

14.2 The results of coating adhesion assessments obtained with Method B1 should not be compared with the results obtained with Method B2.
Standard Specification for Passivation of Stainless Steels Using Electropolishing

This standard is issued under the fixed designation B 912; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers the passivation of stainless steel alloys in the 200 (UNS2XXXX), 300 (UNS3XXXX), and 400 (UNS4XXXX) series, and the precipitation-hardened alloys, using electropolishing procedures.

NOTE 1—Surface passivation occurs simultaneously with electropolishing under proper operating conditions. The quality of passivation will depend on the type of stainless steel, the formulation of the electropolishing solution, and the conditions of operation. Free iron on the surface of the stainless steel is removed resulting in improved corrosion resistance. Surface smoothing obtained by electropolishing will also improve corrosion resistance. Electropolishing will also remove heat tint and oxide scale.

1.2 This specification may involve hazardous materials, operations, and equipment. This specification does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
A 380 Practice for Cleaning and Descaling Stainless Steel Parts, Equipment, and Systems
A 967 Specification for Chemical Passivation Treatments for Stainless Steel Parts
B 117 Practice for Operating Salt Spray (Fog) Apparatus
B 322 Practice for Cleaning Metals Prior to Electroplating
B 374 Terminology Relating to Electroplating
B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
D 3951 Practice for Commercial Packaging

2.2 ISO Standards:
ISO 2080 Electroplating and Related Processes—Vocabulary
ISO 4519 Electrodeposited Metallic Coatings and Related Finishes—Sampling Procedures for Inspection by Attributes
ISO 9227 Corrosion Tests in Artificial Atmospheres—Salt Spray Tests
ISO/DIS 15730 Metallic and Other Inorganic Coatings—Electropolishing as a Means of Smoothing and Passivating Stainless Steels
ISO/DIS 16348 Metallic and Other Inorganic Coatings—Definitions and Conventions Concerning Appearance

3. Terminology

3.1 Definition of terms in this specification can be found in Terminology B 374 and ISO 2080.

3.2 Definitions:
3.2.1 passivation, n—The rendering of a stainless steel surface into a lower state of chemical reactivity.
3.2.1.1 Discussion—Passivated surfaces are characterized by the absence of free iron, as defined by Practice A 380.
3.2.2 electropolishing, n—the electrochemical process in which the article(s) to be passivated are treated anodically in a suitable acid medium.

4. Ordering Information

4.1 When ordering articles to be electropolished in conformance with this standard, the purchaser shall state the following:

4.1.1 Alloy Designation—When ordering articles passivated in accordance with this specification, the purchaser shall state, in addition to the ASTM designation number, the date of issue, the alloy designation number, and the testing method(s) by which the article will be evaluated (see 5.3).

4.1.2 Appearance—The purchaser shall specify the appearance required, for example, bright or dull. Unless otherwise specified by the purchaser, a bright luster shall be acceptable. Alternatively, samples showing the required finish, or range of finish, shall be supplied or approved by the purchaser. When required, the basis material may be subjected, before electropolishing, to such mechanical polishing as may be required to yield the desired final surface characteristics.

4.1.3 Contact Marks—Visible contact marks may occur. The location of electrical contact marks shall be agreed upon

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1 This specification is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.07 on Chromate Conversion Coatings.


2 Annual Book of ASTM Standards, Vol 01.03.

3 Annual Book of ASTM Standards, Vol 03.02.

4 Annual Book of ASTM Standards, Vol 02.05.


6 Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

7 This standard is not a requirement but can be referenced for additional information.
between purchaser and supplier.

4.1.4 **Metal Removal**—Some metal is removed from the surface of the article during electropolishing, typically 5 to 10 um. As much as 50 µm may be removed for additional smoothing. The ordering document shall include the maximum amount of metal to be removed.

4.1.5 Any requirement for certification—See Section 9.

4.1.6 Any requirement for packaging—See Section 10.

5. **Product Requirements**

5.1 **Visual Defects**—When specified, the significant surfaces of the article to be passivated by electropolishing shall be free of clearly visible defects such as pits, roughness, striations, or discoloration when examined with 20/20 eyesight at a distance of approximately 0.5 m.

**Note** 2—Defects in the surface of the basis material such as scratches, porosity, inclusions, and so forth, may adversely affect the appearance and performance of the article. Visible examination shall include wearing correctional glasses if the inspector normally wears them.

5.2 **Process**:

5.2.1 **Surface Preparation**—Preparatory procedures and cleaning of the basis material may be necessary; see Practices A 380 and B 322.

5.2.2 **Electropolishing**—Following the preparatory operations, the articles are introduced into the electropolishing solution for a period of time at the current density and temperature required to produce the passive surface and required surface finish, if any.

**Note** 3—A typical electropolishing solution and operating conditions suitable for many stainless steel alloys is shown in Appendix X1. Proprietary electropolishing solutions are available offering special features such as low sludging, better bright throwing power, longer life, or better performance with specific stainless steel alloys.

**Note** 4—Intricately shaped articles may not receive the same degree of passivation in recessed area as a result of low-current densities. Increasing time or overall current density, or both, or the use of auxiliary cathodes, may be used to improve electropolishing in these areas and to pass subsequent passivation tests.

5.2.3 **Post Dip**—Articles withdrawn from the electropolishing solution will have a residual film that may adversely affect the appearance or performance of the product. The preferred method of removing this film is by rinsing the articles in a room-temperature solution of 10 to 30 % v/v nitric acid (specific gravity 1.42, 70 % w/w).

5.2.3.1 Where local conditions prevent the use of nitric acid (nitrates) for film removal, other options may be used as long as the articles meet the requirements of 5.3.

**Note** 5—A 60-g/L solution of citric acid has been used for film removal; however, note that this procedure may pose waste treatment difficulties. The use of other mineral acids, such as sulfuric or hydrochloric acids, is not recommended as the passive film may be compromised. Neutralization procedures such as immersion in alkaline solutions should not be used as they can have a tendency to “set” the residual film and detract from appearance and performance.

5.2.4 **Final Rinsing**—Rinsing subsequent to passivation is necessary to remove all traces of acidified water that may affect the appearance and performance of the passive part. Deionized or distilled water may be used to avoid water spots.

5.3 **Passivation Testing**:

5.3.1 Passivation by electropolishing shall be evaluated by one or more of the following test methods (see Section 6 for test procedures):

5.3.1.1 **Water immersion test**,

5.3.1.2 **Humidity test**,  

5.3.1.3 **Salt spray test**,  

5.3.1.4 **Copper sulfate test**, and  

5.3.1.5 Modified “ferroxyl” test for free iron.

6. **Test Procedures**

6.1 **Water Immersion Test** (Specification A 967)—The article(s) shall be alternately immersed in distilled water for 1 h, then allowed to dry for 1 h for twelve wet-dry cycles (24 h total). Failure is indicated by the presence of red rust or staining as a result of the presence of free iron on the surface.

6.2 **Humidity Test** (Practice A 380, Specification A 967)—The article(s) shall be subjected to 100 % humidity at 38 ± 2°C in a suitable humidity cabinet for a period of 24 h. Failure is indicated by the presence of red rust or staining as a result of the presence of free iron on the surface.

6.3 **Salt Spray (Fog) Testing** (Practice B 117 (ISO 9227))—The article(s) shall be subjected to the prescribed test for a minimum of 2 h in an accredited cabinet. Failure is indicated by the presence of red rust or staining as a result of the presence of free iron on the surface.

6.4 **Copper Sulfate Test** (Practice A 380, Specification A 967)—See Appendix X2. The article is swabbed with an acidified solution of copper sulfate. Failure is indicated by the presence of a copper-colored deposit or copper-colored spots, or both. This test is not to be used on martensitic 400 series stainless steels.

6.5 Modified “Ferroxyl” Test (Practice A 380, Specification A 967)—See Appendix X3. The article is swabbed with a solution of potassium ferricyanide. Failure is indicated by the presence of a dark blue color within 30 s.

7. **Sampling Requirements**

7.1 Test Method B 602 (ISO 4519) can be applied to finishes such as electropolished surfaces.

7.2 A random sample of the size required by Test Method B 602 shall be selected from the inspection lot (see 7.3). The articles in the lot shall be inspected for conformance to the requirements of this specification and the lot shall be classified as conforming or not conforming to each requirement according to the criteria of the sampling plans in Test Method B 602.

**Note** 6—Test Method B 602 contains four sampling plans for the original inspection of coated articles. Three are to be used when the test methods are nondestructive; that is, the test method does not make the articles nonconforming. The fourth plan is used when the test method is destructive. If it is not clear whether the test is destructive or nondestructive, the purchaser should identify which test methods are destructive and which are nondestructive. In some instances, both destructive and nondestructive test methods may exist for the testing of the conformance of a finish to a particular requirement. The purchaser shall state which is to be used.

7.3 An inspection lot shall be defined as a collection of articles that are of the same kind, that have been produced to the same specifications, that have been finished by a single supplier at one time, or at approximately the same time, under
essentially identical conditions, and that are submitted for acceptance or rejection as a group.

7.4 If separate test specimens are used to represent the articles in a test, the specimens shall be of the nature, size, and number, and be processed as required in the test methods of this specification. Unless a need can be demonstrated, separately prepared specimens shall not be used in place of production articles for nondestructive tests and visual examination. For destructive tests, separately prepared specimens may be used.

8. Rejection and Hearing

8.1 Parts that fail to conform to the requirements of this specification may be rejected. Rejection should be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with test results, the producer or supplier may make a claim for a rehearing.

9. Certification

9.1 When specified in the purchase order or contract, the purchaser shall be furnished certification that samples representing each lot have been either tested or inspected as directed in this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results shall be furnished.

10. Packaging

10.1 If packaging requirements are necessary under this specification, they shall be in accordance with Practice D 3951.

11. Test Report

11.1 The test report shall include the following information:
11.1.1 A reference to this standard,
11.1.2 A reference to the test method(s) used (see 5.3.1),
11.1.3 The location(s) of the test area(s) on each specimen,
11.1.4 The quantity of specimens tested,
11.1.5 The name of the operator and the testing laboratory,
11.1.6 The date on which the test(s) was (were) performed,
11.1.7 Any circumstances or conditions thought likely to affect the results or their validity,
11.1.8 Any deviation from the test method specified, and
11.1.9 The result(s) of the test(s) (see section 6).

12. Keywords

12.1 electropolishing; passivation; stainless steel

APPENDIXES

(Nonmandatory Information)

X1. TYPICAL ELECTROPOLISHING SOLUTION

11.1 Electropolishing Materials

<table>
<thead>
<tr>
<th>Material</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sulfuric acid, 96 %/w</td>
<td>50 % v/v</td>
</tr>
<tr>
<td>Orthophosphoric acid, 85 %/w</td>
<td>50 % v/v</td>
</tr>
<tr>
<td>Current density</td>
<td>15 amp/dm² minimum</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature</td>
<td>75°C</td>
</tr>
<tr>
<td>Time, typical</td>
<td>2 to 4 min</td>
</tr>
<tr>
<td>Cathodes</td>
<td>stainless steel, copper, lead</td>
</tr>
</tbody>
</table>

X2. COPPER SULFATE TEST (FOR FREE IRON)

X2.1 Materials—Copper sulfate test solution:

<table>
<thead>
<tr>
<th>Material</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Distilled water</td>
<td>100 mL</td>
</tr>
<tr>
<td>Sulfuric acid, 96 %/w</td>
<td>0.4 mL</td>
</tr>
<tr>
<td>Copper sulfate, ACS₅H₂O₅CuSO₄</td>
<td>1.6 g</td>
</tr>
</tbody>
</table>

(dissolve all ingredients)

X2.2 Procedure—Using a cotton swab, apply the test solution to a clean area of the passivated surface to be tested, keeping the surface wet for a period of 6 min. The formation of a copper-colored deposit or copper-colored spots or both indicates failure. Parts used for testing should be discarded or reprocessed.

X2.3 Storage—The test solution shall be made up fresh every two weeks.

X3. MODIFIED “FERROXYL” TEST (FOR FREE IRON)

X3.1 Materials—Potassium ferricyanide test solution:

<table>
<thead>
<tr>
<th>Material</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Distilled water</td>
<td>70 mL</td>
</tr>
<tr>
<td>Potassium ferricyanide, ACS₅Fe(CN)₆</td>
<td>1.0 g</td>
</tr>
<tr>
<td>Nitric acid, Reagent Grade</td>
<td>30 mL</td>
</tr>
<tr>
<td>sp. gr. 1.415 to 1.420</td>
<td></td>
</tr>
</tbody>
</table>

(dissolve all ingredients)

X3.2 Procedure—Using a cotton swab, apply the test solution to a clean area of the passivated surface to be tested. Parts used for testing should be discarded or reprocessed.

X3.3 Storage—The test solution should be made fresh daily.
1. Scope

1.1 This test method covers the procedure for the qualitative and quantitative evaluation of static heat effects on porcelain enamel coatings.

1.2 This test method is adaptable to various temperatures and times, since the requirements in the porcelain enameling industry differ between manufacturers.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

D 523 Test Method for Specular Gloss

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 blistering, n—a defect caused by gas evolution consisting of a bubble that protrudes on the surface of the glass.

3.1.2 color and gloss change, n—this is determined by the color and gloss delta values calculated between each heat test cycle.

3.1.3 copper heads/stickers, n—protrusions of iron oxide crystals permeated from the steel substrate or conglomerated metal components in the enamel.

3.1.4 crazing, n—a defect appearing as one or more fine cracks in the porcelain enamel from thermal contraction and expansion in the glass.

3.1.5 edge burn off, n—this may occur in the first 24 to 72 h and appears as a thin dull discolored gray line around the entire edge of the test plate.

3.1.6 enamel breakdown, n—the point at which the glass composite has disintegrated. Microscopic examination will show loss of enamel bubble structure and devitrification of the enamel. This stage reveals metallic type elements derived from the glass composition and the iron oxide from the steel substrate that has completely permeated the glass.

3.1.7 hazy appearance/scumming, adj—the glass will develop an opaque film that exhibits low gloss on the surface of the porcelain enamel.

3.1.8 metalizing, n—enamel on test plates will have a reflective copper color from condensed metals in the enamel that may increase with the deterioration of the glass during the heat test cycle.

4. Significance and Use

4.1 This test method is intended for testing the porcelain enamel finish on oven parts of self-cleaning ranges.

4.2 The numerical values and visual evaluation derived by this test method are used to measure differences in heat resistant characteristics between enamel formulas intended to meet oven manufacturer specifications.

5. Apparatus

5.1 Furnace, capable of holding a constant temperature of 920°F (493°C) minimum.

5.2 Standing Bracket, approximately 10 by 10 by 6 in. (25.4 by 25.4 by 15.24 cm). Dimensions may vary according to test plate size.

5.3 Two Heavy-Gauge Steel Rods, approximately 12 in. (30.48 cm) long and 1⁄8 in. (0.32 cm) in diameter.

5.4 Standard Size 3⁄8–16 or 3⁄8–24 (0.96-cm) Nuts, used for spacers between heat test plates.

5.5 Spectrocolorimeter, capable of reading color by reflection of enameled plates on reflectance 45°/0° and L*, a*, b* color space. Equipment available for making color readings is an instrument that reads Spectrocolorimeter L, a, b scale such as Macbeth Color Eye or a Hunter Lab ColorQUEST.

5.6 Gloss Meter, capable of reading 60° ASTM gloss.
measurements (see Test Method D 523).  

6.  Procedure  

6.1  Punched holes are located ¼ in. (0.63 cm) down from the top and ½ in. (1.2 cm) from the right and left sides on a 5⅞ by 5¼-in. (13.65 by 13.65-cm) uncoated metal plate.  

6.2  The metal plate is processed in the enamel formula that is to be subjected to static heat testing.  

6.3  Before placing the test plate on heat test, initial color (L, a, b) and gloss (60°) readings are recorded, dated, and established as 0 h.  

6.4  The enameled plate should be clean and free of any fingerprints or smudges. To clean a soiled plate, a lint-free paper towel dampened with alcohol is rubbed back and forth on the surface until plate is clean. To prevent further smudging, handle test plates on the edges.  

6.5  A steel rod is passed through each hole of the test plate. The test plates are suspended across the top of the standing bracket by the steel rods.  

6.6  If more than one test plate is required, a spacer is placed on the rods between each test plate. This allows a ¼-in. (0.63-cm) space between each test plate. Approximately 26 heat test plates can be suspended from the two steel rods.  

6.7  A cover plate is placed on both ends of each row. The cover plate should be an enameled plate the same size as the heat test plates (previous heat test plates no longer needed for cover plate should be an enameled plate the same size as the heat test plates can be suspended from the two steel rods.  

6.8  The enameled plate should be clean and free of any fingerprints or smudges. To clean a soiled plate, a lint-free paper towel dampened with alcohol is rubbed back and forth on the surface until plate is clean. To prevent further smudging, handle test plates on the edges.  

6.9  A steel rod is passed through each hole of the test plate. The test plates are suspended across the top of the standing bracket by the steel rods.  

6.10  The color and gloss readings of test plates are dated and recorded after every 24 h, stand at room temperature and evaluated for the amount of change in color, gloss, and physical appearance.  

6.11  Test plates that complete the 360 h are examined visually. Deltas of color and gloss readings from 0 to 360 h are recorded.  

6.12  Heat test plates that do not display any extreme physical breakdown are returned to the 920°F (493°C) oven to continue the heat test cycle.  

6.13  Test plates are removed every 24 h from the oven, set to room temperature, and visually examined. Color and gloss readings are recorded, dated, and the total number of hours of exposure is recorded.  

6.14  Test plates that fail because of excessive color and gloss change or show extreme enamel breakdown as described in 6.11 are removed from the test and recorded as to what time failure has occurred.  

6.15  Test plates that complete the 360 h are examined visually. Deltas of color and gloss readings from 0 to 360 h are recorded.  

7.  Rating Test Specimens  

7.1  A full heat test life cycle begins when the enameled plate(s) is placed in the preheated furnace and ends after the 360-h time frame.  

7.2  The plate(s) shall be rated for the number of cycles it has passed without failure. For example, enamel that failed at 168 h would be unfavorable compared to enamel that did not fail until 336 h. Heat test plates are rated by the number of hours accumulated in the furnace before failure occurs.  

7.3  For comparison purposes, sample plates processed in standard enamel would have to be heat tested at the same time to complete the rating of the test enamel from 0 to 360 h.  

7.4  Visual examination of an enameled test plate after a 360-h heat cycle would be rated according to the calculation of total color change. (DE)_, where its value is equal to or less than the (DE) calculated for a standard enamel, which might be a current production enamel, over the full 360-h heat cycle.  

7.5  Change in gloss over full heat cycle must be comparable to the standard enamel.  

8.  Report  

8.1  The following is a list of data to be recorded:  

8.1.1  Amount of hours test plate has accumulated in the furnace.  

8.1.2  Color readings on the L, a, b scales.  

8.1.3  Gloss readings on the 60° gloss meter.  

8.1.4  Changes in gloss, color, and DE values are recorded.  

8.1.5  Written comments on any visual enamel breakdown during the test cycle.  

9.  Precision and Bias  

9.1  No justifiable statements can be made regarding the precision and bias of this test method. This test is subject to a
wide margin of subjective visual rating and enamel batch formulation. Since base metal composition, fabrication, enamel formulation, and processing will give rise to variables in the heat resistance of porcelain-enamedled plates, each test plate should be considered separately.

10. Keywords

10.1 porcelain enamel coatings; self-cleaning oven coating; static heat
Standard Test Method for Adherence of Porcelain Enamel Coatings to Sheet Metal

This standard is issued under the fixed designation B 916; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 The scope of this test method is:

1.1.1 To determine quantitatively the amount of enamel remaining after the porcelain enamel coating specimen has been deformed;

1.1.2 To standardize the deformation parameters for testing adherence of porcelain enamel to sheet metal; and,

1.1.3 To provide a quantitative adherence rating scale for comparison to reference standards.

1.2 This adherence test method is applicable to porcelain enamel coatings on substrates from 18 to 24 gage (0.0478 to 0.025 in. or from 1.214 to 0.654 mm).

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Significance and Use

2.1 The measurement of adherence in porcelain enamel systems, such as ground coat, ground coat/cover coat, and direct-on cover-coat enamels, is an important procedure for monitoring process variables in metal preparation, enamel application thickness, steel selection, and enamel selection, as well as assuring the ultimate quality of the finished product.

3. Apparatus

3.1 Drop Weight Deformation Device, see Figs. 1 and 2.

3.2 The drop weight deformation device as shown in Fig. 1 shall impart a single impact blow from a hemispherical indenter to a coated specimen supported over a sharp-edged bottomless die. The area of the deformation and the concomitant damage to the coating is largely influenced by the size of the hemispherical indenter; the die opening size and edge sharpness; and, the energy of the blow at impact.

3.3 A satisfactory degree of deformation and damage are obtained if the conditions in 3.2 use the following parameters: 0.50-in. (12.7-mm) diameter hemispherical indenter, 0.75-in. (19.1-mm) diameter bottomless die (lower plunger guide in Fig. 1) with sharp edge, and 80-in./lb (9.03-J) energy at impact.

3.4 Deformation Devices, of other designs may be used provided that the parameters in 3.3 are used.

3.5 Adherence Reference Standards, see 7.1.

4. Test Specimens

4.1 The test specimens shall be flat enameled panels not less in size than 4 in.² (101.6 mm²). Larger sizes and shapes may be used provided that a flat surface can be held firmly against and parallel to the lower plunger guide during deformation.

5. Safety Precautions

5.1 During the procedure in 6.1, fine enameled chips may be released with some force from the steel surface; therefore, it is recommended that personnel in the immediate vicinity of the test wear safety goggles.

6. Procedure

6.1 Specimen Deformation—The hammer weight of the suggested drop weight device is 5 lb (2268 g), which requires a 16-in. (406.4-mm) drop distance to yield the specified 80-in./lb (9.03-J) energy at impact. The hammer weight of the drop weight device may range from 2 to 8 lb (907 to 3629 g). If the hammer weight is within this range, but not 5 lb, calculate the drop distance to yield 80 in./lb of energy at impact. The center of the area to be deformed shall be at least 1½ in. (38.1 mm) from the edge of the specimen. If two or more deformations are made on a single specimen, place them at least 3 in. (76.2 mm) between centers and average and treat the measurements on them as a single measurement. Raise and secure the hammer to obtain the required drop distance. Hold the specimen firmly against and parallel to the top surface of the die with the enameled side up and release the hammer.

6.2 The specified drop weight device is provided with a spring to limit the indenter to a single impact blow against the specimen. If the device to be used is not designed in this way, use another means or procedure to avoid a restrike of the indenter.

6.3 A visual examination of the impact area is then made. Evaluation of the degree of adherence obtained is judged by the amount of glass retained in the impact area.
7. Rating of Adherence

7.1 Examine the impacted area and compare to the following reference standards (see Figs. 3 and 4):

0 = No adherence—only shiny metal showing, and enamel may be flaking off beyond the impact area.
1 = Poor adherence—very little glass adhering, less than 10 %, and only at the outer edges of the impact area.
2 = Fair adherence—glass particles adhering in at least 25 to 30 % of the impact area.
3 = Good adherence—glass particles adhering in at least 50 to 60 % of the impact area.
4 = Very good adherence—glass particles adhering in at least 70 to 80 % of the impact area.
5 = Excellent adherence—glass particles covering almost all of the impact area—at least 90 %.

7.2 Report adherence rating, estimated to the nearest 0.5.

8. Referee Method

8.1 Since adherence depends on factors other than the enamel, that is, steel, firing, and metal preparation, in cases of disputed results, a direct comparison should be made between the test enamel known to have acceptable adherence by coating one-half of a panel with each of the enamels and comparing results as in 7.1.

9. Precision and Bias

9.1 The precision and bias of this test method is being established.

10. Keywords

10.1 adherence; porcelain enamel coatings; sheet metal
FIG. 2 Drop Weight Adherence Tester

- Pin for holding weight at top held in by spring
- 3/4 DIA.
- 5 lb. weight
- 1/4 holes 1 in. on centers
- Pin for holding weight
- Plunger 5/8 DIA., 2 3/4 lg., drilled in end 1/4 in. deep for 1/2 in. ball and then pounded over to hold ball. Spring keeps plunger up when weight is not on it.
- Press fit in top plate
- Test specimen
- 1/2 in. ball
- Hole drilled 3/16 for plunger with 1/8 RAD. on top.
- Press fit in base
Note: See Section 7 for guide to numbers.

FIG. 3 Two Coat-One Fire Bond Classification
FIG. 4 One Coat-Ground Coat Bond Classification

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Standard Specification for Non-hexavalent Chromium Conversion Coatings on Aluminum and Aluminum Alloys

This standard is issued under the fixed designation B 921; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers the requirements relating to rinsed and non-rinsed non-hexavalent chromium conversion coatings on aluminum and aluminum alloys intended to give protection against corrosion and as a base for other coatings.

1.2 Aluminum and aluminum alloys are conversion coated in order to retard corrosion; as a base for organic films including paints, plastics, and adhesives; and as a protective coating having a low electrical contact impedance.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory requirements prior to use.

2. Referenced Documents

2.1 ASTM Standards:
- B 117 Practice for Operating Salt Spray (Fog) Apparatus
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
- D 1730 Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting
- D 3359 Test Methods for Measuring Adhesion by Tape Test
- ISO Standards:
  - ISO 2409 Paint and Varnishes—Cross-Cut Test
  - ISO 3768 Metallic Coatings—Neutral Salt Spray Test (NSS Test)
  - ISO 4519 Electrodeposited Metallic Coatings and Related Finishes—Sampling Procedures for Inspection by Attributes
- Federal Standard:
  - Fed. Std. No. 141 Paints, Varnish, Lacquer, and Related Materials; Methods of Inspection

2.4 Military Specification:
- MIL-C-5541 Chemical Films for Aluminum and Aluminum Alloys

3. Terminology

3.1 Definitions:
- rinsed—conversion coatings that are rinsed in water prior to drying.

3.1.1 Discussion—This type of coating is typically applied to extruded aluminum fabricated parts and castings.

4. Classification

4.1 A class one conversion coating provides for maximum salt fog corrosion protection to bare metal. A class two conversion coating is designed to give painted salt fog corrosion resistance and some bare salt fog corrosion resistance. A class three conversion coating provides little salt fog corrosion resistance, but low electrical contact resistance (see MIL-C-5541). Class four and five conversion coatings only provide for painted salt fog corrosion resistance.

4.2 The finishes are divided into four classes; their most important characteristics are listed in Table 1.

5. Surfaces Preparation

5.1 The surfaces of the parts to be conversion coated shall be clean and free of any oxidation, scale, or soils such as metal turnings, grinding dust, oil, grease, lubricants, hand-sweat, or any other contamination detrimental to the conversion coating process. The parts shall therefore, as far as necessary, be...
cleaned in a non-hexavalent chromium cleaner before conversion coating and, if necessary, be pickled.

6. Methods of Application of Conversation Coatings

6.1 Metallic material other than aluminum shall not be treated with the parts to be conversion coated.

6.2 Conversion coatings are normally applied by dipping: the coating may also be applied by inundation, spraying, roller coating, or by wipe-on techniques. The application method used shall be taken from the operating instructions for the conversion coating process employed. Conversion coating solutions are usually acidic and may contain various salts that may be varied to affect the appearance and hardness of the film. The type of conversion coating depends on the composition of the solution and may also be affected by the pH, temperature, duration of the treatment, and the nature and surface condition of the alloy being treated.

6.3 Those coatings receiving a final water rinse. If the coating is meant to be a basis for additional coatings, the detail shall be subject to a rinse in deionized water with a conductivity less than 100 µS/cm. Hot water shall be used only if allowed by the manufacturer of the conversion coating in question. The drying of the coating shall be carried out in accordance with the specifications of the manufacturer of the conversion or conversion coating in question.

6.4 Any additional subsequent treatments will depend upon the direction of the manufacture of the conversion or conversion coating in question.

7. Coating Requirements

7.1 Electrical Resistance—When specified, the electrical resistance shall be in accordance with MIL-C-5541.

<table>
<thead>
<tr>
<th>Class</th>
<th>Corrosion Protection</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Maximum corrosion resistance generally used as a final finish</td>
</tr>
<tr>
<td>2</td>
<td>Moderate corrosion resistance, used as a paint base and for bonding to rubber</td>
</tr>
<tr>
<td>3</td>
<td>Decorative, slight corrosion resistance, low electrical contact resistance</td>
</tr>
<tr>
<td>4</td>
<td>Moderate corrosion resistance, used as a paint base and for bonding to rubber</td>
</tr>
</tbody>
</table>

TABLE 1 Classification of Non-hexavalent Chromium Conversation Coatings

<table>
<thead>
<tr>
<th>Coating Class</th>
<th>Non-Heat-Treatable Wrought Alloys</th>
<th>Heat-Treatable Alloys with a Nominal Silicon Content &lt;1%</th>
<th>Cast Alloys with a Nominal Silicon Content &gt;1%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>500</td>
<td>336</td>
<td>48</td>
</tr>
<tr>
<td>2</td>
<td>250</td>
<td>168</td>
<td>24</td>
</tr>
<tr>
<td>3</td>
<td>168</td>
<td>120</td>
<td>12</td>
</tr>
<tr>
<td>4</td>
<td>500</td>
<td>336</td>
<td>48</td>
</tr>
</tbody>
</table>

*TABLE 2 Relative Corrosion Resistance*

7.2 Adhesion—The coatings shall be adherent and non-powdery. A practical evaluation of the adhesion can be made by measuring the adhesion of a secondary organic film applied to the conversion coating in question. When specified, the conversion coating shall pass the organic coating adhesion test in Test Method D 3359 or the equivalent ISO 2409.

7.2.1 Class 4 coatings intended for use under MIL-C-5541 shall have their adhesion evaluated by Method 6301 of Fed. Std. No. 141.

7.2.2 Additional treatments applied under MIL-C-5541 can be found in Practices D 1730, Methods 5, 6, and 7.

7.3 Corrosion Resistance—When subjected to the neutral salt spray test specified in Test Method B 117 or in the equivalent ISO 3768, three separate test specimens of the coating shall withstand exposure for the hours shown in Table 2 without giving evidence, to the unaided eye, of more than a total of 8 isolated spots or pits. None shall be larger than 1 mm in diameter. Each individual test specimen shall not have more than 5 isolated spots or pits, none larger than 1 mm in diameter on their respective surfaces. Spots within 10 mm of the edges of the panels are not counted.

7.4 Test Specimens and Samplings—Unless otherwise specified, the sampling plans of Test Method B 602 or the equivalent ISO 4519 shall be used to test the coatings.

7.4.1 Test specimens shall be of the same alloy and surface condition as the articles represented. Test specimens shall be 150 by 100 mm.

8. Keywords

8.1 conversion coating; non-hexavalent chromium conversion coating; non-rinsed; rinsed
Standard Test Method for
Metal Powder Specific Surface Area by Physical Adsorption

This standard is issued under the fixed designation B 922; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers determination of surface area of metal powders. The test method specifies general procedures that are applicable to many commercial physical adsorption instruments. The method provides specific sample outgassing procedures for listed materials. It includes additional general outgassing instructions for other metals. The multipoint equation of Brunauer, Emmett and Teller (BET)\(^2\), along with the single point approximation of the BET equation, forms the basis for all calculations.

1.2 This test method does not include all existing procedures appropriate for outgassing metallic materials. The procedures included provided acceptable results for samples analyzed during interlaboratory testing. The investigator shall determine the appropriateness of listed procedures.

1.3 This method uses SI units as standard. State all numerical values in terms of SI units unless specific instrumentation software reports surface area using alternate units. In this case, present both reported and equivalent SI units in the final written report. Many instruments report surface area as m\(^2\)/g, instead of using correct SI units (m\(^2\)/kg).

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 215 Practices for Sampling Finished Lots of Metal Powders\(^3\)
B 243 Definitions of Terms Used in Powder Metallurgy\(^3\)
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method\(^4\)

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3. Terminology

3.1 Definitions—Refer to Terminology B 243 for additional terms specific to metal powders.
3.2 Definitions of Terms Specific to This Standard:
3.2.1 adsorbate, \(n\)—material that has been retained by the process of adsorption.
3.2.2 adsorbent, \(n\)—any solid having the ability to concentrate or collect significant quantities of other substances on its surface.
3.2.3 adsorption, \(n\)—a process in which fluid molecules are concentrated or collected on a surface by chemical or physical forces, or both.
3.2.4 adsorptive, \(n\)—any substance available for adsorption.
3.2.5 outgassing, \(n\)—the evolution of gas from a material in a vacuum or inert gas flow, at or above ambient temperature.
3.2.6 physical adsorption (van der Waals adsorption), \(n\)—the binding of an adsorbate to the surface of a solid by forces whose energy levels approximate those of condensation.
3.2.7 surface area, \(n\)—the total area of the surface of a powder or solid including both external and accessible internal surfaces (from voids, cracks, open porosity, and fissures). The area may be calculated by the BET (Brunauer, Emmett, and Teller) equation from gas adsorption data obtained under specific conditions. It is useful to express this value as the specific surface area, for example, surface area per unit mass of sample (m\(^2\)/kg).
3.2.8 surface area (BET), \(n\)—the total surface area of a solid calculated by the BET (Brunauer, Emmett, Teller) equation, from nitrogen adsorption or desorption data obtained under specific conditions.
3.2.9 surface area, specific, \(n\)—the area, per unit mass of a granular or powdered or formed porous solid, of all external plus internal surfaces that are accessible to a penetrating gas or liquid.

4. Summary of Test Method

4.1 An appropriately sized sample (to provide at least the minimum surface area required for reliable results for the instrument used) is outgassed under appropriate conditions prior to analysis.

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\(^1\) This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.03 on Refractory Metal Powders.


\(^3\) Annual Book of ASTM Standards, Vol 02.05.


4.2 Multipoint BET Analyses only—Volume of gas adsorbed, or desorbed, is determined as cm$^3$ corrected to standard temperature and pressure (STP) for a minimum of four relative pressures within the linear BET transformation range of the physical adsorption, or desorption, isotherm characteristic of the metal. The linear range is that which results in a least squares correlation coefficient of 0.9999 or greater for the relationship between BET transformation and relative pressure. Typically, the linear range includes relative pressures between 0.05 and 0.30.

4.3 Single Point BET Analyses only—Volume of gas adsorbed, or desorbed, is determined as cm$^3$ corrected to standard temperature and pressure (STP) at the highest known relative pressure within the linear BET transformation range of the physical adsorption, or desorption, isotherm. Typically, a relative pressure of 0.30 is used. (It may be necessary to first perform a multipoint analysis of the material to determine the optimum single point relative pressure.)

4.4 The sample is weighed to nearest 0.1 mg after analysis. It is important to use an analytical balance to determine the sample mass. The physical adsorption instrument measures the total amount of gas adsorbed onto, or desorbed from, the sample under analysis. The sample mass is then used to normalize the measured adsorption results. Any error in the sample mass will affect the final BET surface area.

4.5 Calculations are based on the BET equation, as required by the instrument being used for the determination. The cross sectional area for the adsorbate is taken to be 0.162 nm$^2$ if nitrogen is used as the adsorptive. Use the appropriate value recommended by the instrument manufacturer for adsorbives other than nitrogen. Report this cross sectional area with the BET surface area results.

5. Significance and Use

5.1 Both suppliers and users of metals can benefit from knowledge of the surface area of these materials. Results of many intermediate and final processing steps are controlled by, or related to, specific surface area of the metal. The performance of many sintered or cast metal structures may be predicted from the specific surface area of the starting metal powder, or all or a portion of the finished piece.

6. Interferences

6.1 This test method can be used to determine the internal and external surface area of a powder or solid only after these surfaces have been cleaned of any physically adsorbed molecules. Such adsorbed species, for example water or volatile organic compounds, prevent physical adsorption of the gas probe molecules used to measure surface area. Therefore, it is necessary to remove these adsorbed contaminants prior to surface area analysis. Generally, such outgassing is performed by evacuating or flushing the sample. Outgassing can be accelerated by using elevated temperatures, provided no irreversible sample changes occur. Typical minimum vacuum levels attained are $10^{-1}$ Pa. Typical flushing gases are helium, nitrogen, or a mixture of the two. Outgassing is complete when duplicate surface area analyses produce results within expected instrument repeatability limits, when a constant residual vapor pressure is maintained upon isolation from the vacuum source, or when flushing gas composition is unaffected while passing over the sample.

7. Apparatus

7.1 Commercial instruments are available from several manufacturers for the measurement of specific surface area by physical adsorption. Some are automated versions of the classical vacuum apparatus. Others make use of balanced adsorption technology. Additionally, commercial instruments are available which measure physical adsorption based on the dynamic flow method.

7.2 Analytical Balance, capable of weighing to the nearest 0.1 mg.

8. Reagents and Materials

8.1 Liquid Nitrogen.

8.2 Nitrogen, 99.999 mole percent, with the sum of O$_2$, argon, CO$_2$, hydrocarbons (as CH$_4$), and H$_2$O totaling less than 10 parts per million; dry and oil-free; cylinder, or other source of purified nitrogen.

8.3 Helium, 99.999 mole percent, with the sum of N$_2$, O$_2$, argon, CO$_2$, hydrocarbons (as CH$_4$), and H$_2$O totaling less than 10 parts per million; dry and oil-free; cylinder, or other source of purified helium, if needed for determination of void space above sample.

8.4 Blended Nitrogen and Helium, dry and oil-free; cylinder, or other source of blended gases. The actual composition of the blend must be known. For use with dynamic flow instruments only.

9. Hazards

9.1 Precautions applying to the use of liquid nitrogen and compressed gases should be observed.

10. Sampling, Test Specimens, and Test Units

10.1 It is important that the test portion being analyzed represent the larger bulk sample from which it is taken. The bulk sample should be homogenized before any sampling takes place. Best results are obtained when a flowing bulk material is temporarily diverted into a collector for an appropriate time. It is better to sample the entire flow for a short time than to sample a portion of the flow for a longer time. Collecting several small aliquants and combining them improves the reliability of the sampling process. Rotating rifflers are available commercially which satisfy these sampling requirements. Refer to Practices B 215 for information on the use of a chute sample splitter.

11. Calibration and Standardization

11.1 Follow manufacturer’s instructions for calibration and operational verification of the instrument.

12. Outgassing

12.1 Weigh (to nearest 0.1 mg) a clean, empty sample tube, along with stopper or seal. Record the empty tube mass.

12.2 Add test portion to empty sample tube. Sample quantity should be sufficient to satisfy minimum surface area as required by manufacturer.
12.3 Attach prepared sample tube to outgassing port of instrument. Secure heating mantle or oven around sample tube at the time appropriate for sample.

12.4 Initiate outgassing program according to manufacturer’s instructions. Program mantle or oven for initial outgassing temperature. Increase temperature as appropriate for the sample. Allow sample to continue to outgas until prescribed vacuum level or detector signal is achieved and/or for prescribed outgassing time. Samples analyzed during the interlaboratory study were heated for 2 h at 200°C.

12.5 Remove heating mantle or oven from sample tube. Allow sample tube to cool to ambient temperature. Remove and seal sample tube according to manufacturer’s instructions.

12.6 Weigh sample tube (to nearest 0.1 mg) to obtain sample and tube mass. Record mass. Subtract empty sample tube mass determined in 12.1 to obtain outgassed sample mass. Record calculated mass.

13. Procedure

13.1 Attach appropriately prepared sample holder to analysis port according to manufacturer’s instructions. Include any required hardware.

13.1.1 Use nitrogen as adsorptive for all tests. Use blended nitrogen and helium with dynamic flow instruments. Use pure nitrogen with volumetric instruments.

13.1.2 Use helium to determine sample holder void space with volumetric instruments as necessary.

13.1.3 Use liquid nitrogen as cryogen for all tests.

13.2 Automated Instruments only—select, or input, desired analysis and report parameters.

13.2.1 Multipoint BET Analyses only—use at least four analysis points in the linear BET transformation range of the isotherm characteristic of the sample. If necessary, input the outgassed sample mass. (The final mass should be determined and entered after the analysis.)

13.2.2 Single Point BET Analyses only—use highest relative pressure known to be in the linear BET transformation range of the isotherm. If necessary, input the outgassed sample mass. (The final mass should be determined and entered after the analysis.)

13.3 Dynamic Flow Instruments only—collect data points as volume of gas desorbed versus relative pressure.

13.3.1 Multipoint BET Analyses only—use at least four analysis points in the linear BET transformation range of the isotherm characteristic of the sample.

13.3.2 Single Point BET Analyses only—use highest relative pressure known to be in the linear BET transformation range of the isotherm.

13.4 Perform analysis using the specified conditions according to instrument manufacturers’ instructions.

13.5 When the analysis has finished and the sample has warmed to room temperature, remove and seal the sample tube.

Dry tube and weigh (to nearest 0.1 mg). Record the final tube and sample mass. Subtract the empty tube mass recorded in 12.1 to obtain the final sample mass. Record final sample mass.

13.6 Automated Instruments only—edit the file containing sample information to include the final sample mass. Generate final sample report.

14. Calculations

14.1 Automated Instruments only—software automatically calculates results for the chosen reports using the final mass input in 13.6.

14.2 Dynamic Flow Instruments only—follow manufacturer’s instructions for multipoint, or single point, calculations. Use the final sample mass determined in 13.5 when calculating the specific surface area.

15. Report

15.1 Report the following information:

15.1.1 Complete sample identification.

15.1.2 Collected isotherm point(s) as volume adsorbed, or desorbed, versus relative pressure. Note whether adsorption or desorption isotherm is used. Note any units used other than standard.

15.1.3 Analysis gas used (with cross sectional area if other than nitrogen).

15.1.4 BET specific surface area. Note any units used other than standard.

15.1.5 Final sample mass. Note any units used other than standard.

15.1.6 Sample outgassing method, including total time and outgassing temperature(s).

16. Precision and Bias

16.1 An interlaboratory study is underway, conducted according to Practice E 691. The study includes iron, tungsten, nickel, cobalt, molybdenum, chromium carbide, and tungsten carbide powders. Expected precision will be determined for these materials. No statement is given for other metal powders.

16.2 Precision—The repeatability standard deviation and reproducibility standard deviation will be determined upon completion of the interlaboratory study.

16.3 Bias—No information can be presented on the bias of the procedure in this test method for measuring specific surface area because no metal powder having an accepted reference value is available.

17. Keywords

17.1 BET surface area; metal powders; multipoint surface area; outgassing; physical adsorption; refractory metal powders; single point surface area; specific surface area; surface area
1. Scope

1.1 This test method covers determination of skeletal density of metal powders. The test method specifies general procedures that are applicable to many commercial pycnometry instruments. The method provides specific sample outgassing procedures for listed materials. It includes additional general outgassing instructions for other metals. The ideal gas law forms the basis for all calculations.

1.2 This test method does not include all existing procedures appropriate for outgassing metal materials. The included procedures provided acceptable results for samples analyzed during an interlaboratory study. The investigator shall determine the appropriateness of listed procedures.

1.3 This method uses SI units as standard according to Practice E 380. State all numerical values in terms of SI units unless specific instrumentation software reports volume and/or density using alternate units. In this case, present both reported and equivalent SI units in the final written report. Many instruments report skeletal density as g/cm³ instead of using correct SI units (kg/m³).

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 215 Practices for Sampling Finished Lots of Metal Powders

2.43 Definitions of Terms Used in Powder Metallurgy

E 380 Practice for Use of the International System of Units

(SI)³

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁴

3. Terminology

3.1 Definitions—Refer to Terminology B 243 for additional definitions relating to metal powders.

3.2 Definitions: Definitions of Terms Specific to This Standard:

3.2.1 density, n— the mass per unit volume of a material.

3.2.2 density, skeletal, n — the ratio of mass of discrete pieces of solid material to the sum of the volumes of the solid material in the pieces and closed (or blind) pores within the pieces.

3.2.3 outgassing, n — the evolution of gas from a material in a vacuum or inert gas flow, at or above ambient temperature.

3.2.4 skeletal volume, n — the sum of the volumes: the solid material in the pieces and closed (or blind) pores within the pieces.

4. Summary of Test Method

4.1 An appropriately sized sample (to provide at least the minimum skeletal volume required for reliable results for the instrument or apparatus used) is outgassed under appropriate conditions prior to analysis.

4.2 The sample is weighed to nearest 0.1 mg. It is important to use an analytical balance to determine the sample mass. The pycnometer measures the total displaced skeletal volume of the sample under analysis. The sample mass is then used to calculate the skeletal density of the metal. Any error in the sample mass will affect the calculated density. Some cleaning of the sample surface may take place inside the pycnometer. Therefore, it is best to reweigh the sample after analysis and use the final mass when calculating skeletal density.

4.3 Sample skeletal volume is determined a minimum of five times. Skeletal volume average and standard deviation are calculated using standard statistical methods.

4.4 Calculations are based on the ideal gas law, as required by the instrument being used for the determination. The assumption of ideal behavior is accepted as valid at analytical temperatures and pressures. For instruments designed with two pressure chambers, one a sample compartment, and the other a gas expansion chamber, the equation for sample volume calculation takes the form:
\[
V_{\text{sample}} = V_{\text{cell}} - V_{\exp} \left( \frac{P_2}{P_1 - P_2} \right)
\]

where:
- \( V_{\text{sample}} \) = calculated sample volume,
- \( V_{\text{cell}} \) = calibrated sample compartment volume,
- \( V_{\exp} \) = calibrated expansion volume,
- \( P_1 \) = measured gas pressure when only \( V_{\text{cell}} \) is filled with analysis gas, and
- \( P_2 \) = measured gas pressure after expansion of the analysis gas into \( V_{\exp} \).

5. Significance and Use
5.1 Both suppliers and users of metals can benefit from knowledge of the skeletal density of these materials. Results of many intermediate and final processing steps are controlled by or related to skeletal density of the metal. In addition, the performance of many sintered or cast metal structures may be predicted from the skeletal density of the starting metal powder, for all or a portion of the finished piece.

6. Interferences
6.1 This test method can be used to determine the skeletal volume of a powder or solid only after the open pores have been emptied of any physically adsorbed molecules. Such adsorbed species (for example, water or volatile organic compounds) prevent entry of the gas probe molecules into the open porosity of the sample. Therefore, it is necessary to remove these adsorbed contaminants prior to pycnometry analysis. Generally, such outgassing is performed by evacuating or flushing the sample. Outgassing can be accelerated by using elevated temperatures, provided no irreversible sample changes occur. Typical minimum vacuum levels attained are 10\(^{-1}\) Pa. Typical flushing gases are those used for analysis. Outgassing is complete when duplicate skeletal volume analyses produce results within expected instrument repeatability limits. Some commercial instruments include capabilities for automated evacuation, or flushing of the sample, or both. Elevated temperatures should not be used when outgassing samples inside the pycnometer.
6.2 This test method can be used to determine the volume of a sample whose pores have been deliberately filled with a second phase. In this case, removal of the second phase should be avoided. Vacuum degassing or flushing of the sample is not necessary in this case.

7. Apparatus
7.1 Commercial instruments are available from several manufacturers for the measurement of skeletal volume by gas pycnometry. Some instruments perform calculations of skeletal volume, or density, or both, upon completion of the analysis. Others require manual calculation of skeletal volume and density.
7.2 Analytical Balance, capable of weighing to 0.1 mg.

8. Reagents and Materials
8.1 Helium, 99.999 mole percent, with the sum of \( \text{N}_2, \text{O}_2, \text{argon}, \text{CO}_2, \text{hydrocarbons} \) (as \( \text{CH}_4 \)), and \( \text{H}_2\text{O} \) total less than 10 parts per million; dry and oil-free; cylinder, or other source of purified helium.

8.2 Nitrogen, 99.999 mole percent, with the sum of \( \text{O}_2, \text{N}_2, \text{CO}_2, \) hydrocarbons (as \( \text{CH}_4 \)), and \( \text{H}_2\text{O} \) totaling less than 10 parts per million; dry and oil-free; cylinder, or other source of purified nitrogen.
8.3 Other High Purity Gas, dry and oil-free; cylinder, or other source of gas, if other gas is to be used as the analysis or flushing gas. The actual composition of the gas shall be known.

9. Hazards
9.1 Precautions applying to the use of compressed gases should be observed.

10. Sampling, Test Specimens, and Test Units
10.1 No specific instructions are given. However, it is important that the test portion being analyzed represent the larger bulk sample from which it is taken. The bulk sample should be homogenized before any sampling takes place. Best results are obtained when flowing bulk material is temporarily diverted into a collector for an appropriate time. It is better to sample the entire flow for a short time than to sample a portion of the flow for a longer time. Collecting several small test portions and combining them improves the reliability of the sampling process. Rotating rifflers are available which satisfy these requirements. Refer to Practices B 215 for information on the use of a chute sample splitter.

11. Calibration and Standardization
11.1 Follow manufacturer’s instructions for calibration and operational verification of the instrument.

12. Conditioning
12.1 Weigh (to nearest 0.1 mg) a clean, empty sample holder. Record the empty sample holder mass.
12.2 Add sample aliquant to empty sample holder. Sample quantity should be sufficient to satisfy minimum skeletal volume as required by manufacturer. Weigh (to nearest 0.1 mg) and record sample and sample holder mass.
12.3 Sample outgassing may be performed inside the pycnometer. If so proceed to the Procedure section of this test method. Otherwise, follow the remaining steps in this section for external outgassing.
12.3.1 Place prepared sample holder in outgassing device.
12.3.2 Program outgassing device for initial outgassing temperature. Increase temperature as appropriate for the sample. Allow sample to continue to outgas until prescribed vacuum level is achieved, or for prescribed outgassing time, or both.
12.3.3 The metal powders analyzed during the interlaboratory study were prepared inside the instruments by purging with analysis gas. Had preliminary outgassing been desired, a temperature of 200°C applied for 1 h would have been used.
12.3.4 Reduce temperature of outgassing device to ambient. Remove sample holder.
12.3.5 Weigh sample holder (to nearest 0.1 mg) to obtain sample and sample holder mass. Record mass. Subtract empty sample holder mass determined in 12.1 to obtain outgassed sample mass. Record calculated mass.
13. Procedure

13.1 Place filled sample holder in pycnometer. Close sample chamber.

13.2 Use helium, nitrogen, or other high purity gas for analysis and flushing gas.

13.3 Automated Instruments only—select, or input, desired analysis and report parameters. Include outgassing parameters if sample preparation is performed as a part of the sample analysis. If necessary, input the outgassed sample mass. (The final mass should be determined and entered after the analysis.) Determine skeletal volume a minimum of five times.

13.4 Manually Operated Instruments only—collect five sets of analysis data according to manufacturer’s recommended procedure for maximum accuracy and precision.

13.5 When the analysis has finished, remove the sample holder. Weigh holder (to nearest 0.1 mg). Record the final sample holder and sample mass. Subtract the empty sample holder mass recorded in 12.1 to obtain the final sample mass. Record final sample mass.

13.6 Automated Instruments only, input the final sample mass. Generate final sample report.

14. Calculations

14.1 Automated Instruments only, software automatically calculates results for the chosen reports using the final mass input in 13.5.

14.2 Manually Operated Instruments only, calculate skeletal volume using collected data according to manufacturer’s instructions. Use final sample mass from 13.4 to calculate skeletal densities. Calculated average and standard deviation for skeletal volume and density as described in Practice E 691.

15. Report

15.1 Report the following information:

15.1.1 Complete sample identification.

15.1.2 Measured skeletal volumes and statistics. Note any units used other than standard.

15.1.3 Skeletal density determined. Note any units used other than standard.

15.1.4 Final sample mass. Note any units used other than standard.

15.1.5 Analysis gas used.

15.1.6 Sample outgassing method, including total time and outgassing temperature(s).

16. Precision and Bias

16.1 An interlaboratory study is underway, conducted according to Practice E 691. The study includes iron, tungsten, nickel, cobalt, molybdenum, chromium carbide, and tungsten carbide powders. Expected precision will be determined for these materials. No statement is given for other metal powders.

16.2 Precision—The repeatability standard deviation and reproducibility standard deviation will be determined upon completion of the interlaboratory study.

16.3 Bias—No information can be presented on the bias of the procedure in this test method for measuring skeletal density because no metal powder having an accepted reference value is available.

17. Keywords

17.1 density; metal powders; outgassing; pycnometry; refractory metal powders; skeletal density; skeletal volume
1. Scope

1.1 These standard practices cover the specifications for those uniaxially compacted test specimens that are used in ASTM standards, the procedures for producing and preparing these test specimens, and reference the applicable standards.

1.2 Basic tool design and engineering information regarding the tooling that is required to compact the test specimens and machining blanks are contained in the annexes.

1.3 This standard is intended to be a comprehensive one-source document that can be referenced by ASTM test methods that utilize P/M test specimens and in ASTM P/M material specifications that contain the engineering data obtained from these test specimens.

1.4 These practices are not applicable to metal powder test specimens that are produced by other processes such as cold isostatic pressing (CIP), hot isostatic pressing (HIP), powder forging (P/F) or metal injection molding (MIM). They do not pertain to cemented carbide materials.

1.5 Detailed information on P/M presses, compacting tooling and sintering furnaces, their design, manufacture and use are not within the scope of these practices.

1.6 Test specimen and die cavity dimensions shown in inch-pound units are to be regarded as standard and are applicable to the referenced ASTM test methods and material specifications. Values in SI units are shown in parentheses and result from conversion in accordance with IEEE/ASTM Standard SI-10. They may be approximate and are only for information.

1.7 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

A 34/A 34M Practice for Sampling and Procurement Testing of Magnetic Materials
A 596/A 596M Test Method for Direct-Current Magnetic Properties of Materials Using the Ballistic Method and Ring Specimens
A 927/A 927M Test Method for Alternating-Current Magnetic Properties of Torroidal Core Specimens Using the Voltmeter-Ammeter-Wattmeter Method
B 215 Practices for Sampling Finished Lots of Metal Powder
B 243 Terminology of Powder Metallurgy
B 312 Test Method for Green Strength for Compacted Metal Powder Specimens
B 328 Test Method for Density, Oil Content, and Interconnected Porosity of Sintered Metal Structural Parts and Oil-Impregnated Bearings
B 331 Test Method for Compressibility of Metal Powders in Uniaxial Compaction
B 438/B 438M Specification for Sintered Bronze Bearings (Oil-Impregnated)
B 439 Specification for Iron-Base Sintered Bearings (Oil-Impregnated)
B 528 Test Method for Transverse Rupture Strength of Metal Powder Specimens
B 595 Specification for Sintered Aluminum Structural Parts
B 610 Test Method for Measuring Dimensional Changes of
Metal Powder Specimens
B 612 Specification for Iron Bronze Sintered Bearings (Oil-Impregnated)
B 715 Specification for Sintered Copper Structural Parts for Electrical Conductivity Applications
B 782 Specification for Iron Graphite Sintered Bearings (Oil-Impregnated)
B 783 Specification for Materials for Ferrous Powder Metallurgy (P/M) Structural Parts
B 823 Specification for Materials for Nonferrous Powder Metallurgy (P/M) Structural Parts
B 853 Specification for Powder Metallurgy (P/M) Boron Stainless Steel Structural Components
E 8 Test Methods for Tension Testing of Metallic Materials
E 9 Test Methods of Compression Testing of Metallic Materials at Room Temperature
E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials
E 23 Test Methods for Notched Bar Impact Testing of Metallic Materials
E 228 Test Method for Linear Thermal Expansion of Solid Materials with a Vitreous Silica Dilatometer
E 1876 Test Method for Dynamic Young’s Modulus, Shear Modulus and Poisson’s Ratio by Impulse Excitation of Vibration

IEEE/ASTM Standard:
SI-10 Standard for Use of the International System of Units (SI): The Modern Metric System

MPIF Standard:
Standard 56 Method for Determination of Rotating Beam Fatigue Endurance Limit in Powder Metallurgy Materials

3. Terminology

3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243. Additional descriptive information is available in the Related Materials section of Vol 02.05 of the Annual Book of ASTM Standards.

4. Summary of Practice

4.1 These practices describe the production, by pressing and sintering metal powders, and the preparation, by machining sintered blanks, of test specimens used to measure properties of metal powders and sintered materials.

5. Significance and Use

5.1 Test specimens are used to determine the engineering properties of P/M materials, for example, tensile strength, ductility, impact energy, etc.; property data that are essential to the successful use of P/M material standards. Processing P/M test specimens under production conditions is the most efficient method by which to obtain reliable P/M material property data since in most cases it is impractical or impossible to cut test bars from sintered parts.

5.2 The performance characteristics of metal powders, for example, compressibility, green strength and dimensional changes associated with processing are evaluated using P/M test specimens under controlled conditions. The data obtained are important to both metal powder producers and P/M parts manufacturers.

5.3 P/M test specimens play a significant role in industrial quality assurance programs. They are used to compare properties of a new lot of metal powder with an established lot in an acceptance test and are used in the part manufacturing process to establish and adjust production variables.

5.4 In those instances where it is required to present equivalent property data for a production lot of P/M parts, standard test specimens compacted from the production powder mix to the same green density can be processed with the production P/M parts and then tested to obtain this information.

5.5 Material property testing performed for industrial or academic research and development projects uses standard P/M test specimens so the test results obtained can be compared with previous work or published data.

5.6 Powder metallurgy test specimens may have multiple uses. The dimensions and tolerances given in this standard are nominal in many cases. The user is cautioned to make certain that the dimensions of the test specimen are in agreement with the requirements of the specific test method to be used.

6. Powder Metallurgy Test Specimens

POWDER COMPRESSIBILITY TESTING

6.1 Cylindrical Powder Compressibility Test Specimen:

6.1.1 Description and Use—This solid cylindrical test specimen, see Fig. 1, is produced by compacting a test portion of powder mix in laboratory powder metallurgy tooling similar to that shown in Fig. A1.1 in the Annex. An alternative test specimen for measuring powder compressibility is the transverse rupture test specimen. These test specimens are not...
sintered. The compressibility of the metal powder mix or a compressibility curve showing the green density as a function of compacting pressure is determined according to the procedures in Test Method B 331.

6.1.2 Applicable ASTM Standards:
6.1.2.1 See Test Method B 331.

TRANSVERSE RUPTURE, DIMENSIONAL CHANGE AND GREEN STRENGTH TESTING

6.2 Transverse Rupture Strength Test Specimen:
6.2.1 Description and Use—The pressed-to-size transverse rupture test specimen, Fig. 2, is produced by compacting metal powder in tooling similar to that shown in Fig A1.2. This rectangular test specimen has multiple uses in P/M. Primarily, it is designed to determine the transverse rupture strength of sintered or heat treated compacts by breaking the test specimen as a simple beam in three-point loading following Test Method B 528. But, it is also used to measure the dimensional changes of metal powder mixes due to pressing and sintering or other processing steps according to Test Method B 610, and it is used in both a 0.250 and 0.500 in. (6.35 and 12.70 mm) thick version to determine green strength using the procedure in Test Method B 312.

6.2.2 Applicable ASTM Standards:
6.2.2.1 See the following Test Methods: B 312, B 331, B 528, B 610, E 18, and E 1876.

6.2.2.2 See the following P/M Material Specifications: A 811, A 839/A 839M, A 904, B 783, and B 823.

RADIAL CRUSHING TESTING

6.3 Radial Crushing Strength Test Specimen:
6.3.1 Description and Use—The radial crushing strength test specimen shown in Fig. 3 is compacted to size in tooling (Fig. A2.3) suitable for the production of a thin-walled hollow cylinder within the range of the dimensions listed. The testing procedure involves the application of a compressive force perpendicular to the central axis of the test cylinder and calculating the radial crushing strength from the breaking load and test specimen dimensions. Radial crushing strength is the material property that is used to quantify the mechanical strength of sintered metal bearings, (oil impregnated).

6.3.1.1 This test specimen is widely used in a quality control test to determine the sintered material strength of powder mixes that are to be used for the production of any metal powder product because it is a quick, easy test and gives reliable and reproducible results. Laboratories testing powder mixes intended for the manufacture of porous bearings have recognized that breaking an unsintered test specimen by diametrical loading will give a green strength value that is relevant in production.

6.3.2 Applicable ASTM Standards:
6.3.2.1 See the following P/M Bearing Specifications: B 438/B 438M, B 439, B 612, and B 782.

<table>
<thead>
<tr>
<th>Dimensions</th>
</tr>
</thead>
<tbody>
<tr>
<td>in.</td>
</tr>
<tr>
<td>W—Width</td>
</tr>
<tr>
<td>L—Length</td>
</tr>
<tr>
<td>R—Corner radius</td>
</tr>
<tr>
<td>T—Thickness (thin)</td>
</tr>
<tr>
<td>T—Thickness (thick)</td>
</tr>
</tbody>
</table>

Note—Thickness shall be parallel within 0.005 in. (0.13 mm).

FIG. 2 P/M Transverse Rupture Strength Test Specimen

<table>
<thead>
<tr>
<th>Dimensions</th>
</tr>
</thead>
<tbody>
<tr>
<td>in.</td>
</tr>
<tr>
<td>D—Outside diameter</td>
</tr>
<tr>
<td>d—Inside diameter</td>
</tr>
<tr>
<td>T—Thickness</td>
</tr>
</tbody>
</table>

Note—Wall thickness (D-d) shall be less than D/3.

FIG. 3 P/M Radial Crushing Strength Test Specimen
TENSION TESTING

6.4 Flat Unmachined Tension Test Specimen:

6.4.1 Description and Use—The unmachined flat tension test specimen shown in Fig. 4 is commonly referred to in the industry as “the dogbone.” It is compacted directly to size and shape using tooling similar to that shown in Fig. A2.4 in the Annex. This test specimen has been designed to have a convenient 1.00 in.² (645.2 mm²) pressing area to simplify compacting calculations.

6.4.1.1 It is intended for determining the tensile properties and ductility of P/M materials that have not been heat treated (not quenched and tempered nor sinter-hardened). The testing procedures for this unmachined P/M test specimen can be found in Test Method E 8.

6.4.1.2 The flat tension test specimen is not normally used with heat treated P/M materials because it may produce unreliable test results and it has a tendency to slip in the grips. Slippage can be prevented by the use of hydraulic grips, but the square corner design of the flat specimen will give rise to stress concentrations that may result in scattered test values. The machined 190-Round tension test specimen, Fig. 5, is recommended for use with heat treated P/M materials.

6.4.2 Applicable ASTM Standards:

6.4.2.1 See Test Methods E 8.

6.4.2.2 See the following P/M Material Specifications: A 811, A 839/A 839M, A 904, B 715, B 783, B 823, and B 853.

6.5 Machined 190-Round Tension Test Specimen:

6.5.1 Description and Use—The 190-Round tension test specimen may be prepared by machining a sintered Izod test specimen blank, to the shape and dimensions shown in Fig. 5. The gage section shall be free of nicks, scratches, and toolmarks. Polish longitudinally with 00 emery paper and finish with crocus cloth. The section shall be concentric within 0.001 in. (0.03 mm) T.I.R.

6.5.2 Applicable ASTM Standards:

6.5.2.1 See Test Methods E 8.

6.5.2.2 See the following P/M Material Specifications: B 595, B 783, and B 817.

COMPRESSION TESTING

6.6 Machined Compression Test Specimen:

6.6.1 Description and Use—This test specimen, shown in Fig. 6, is usually prepared by machining a sintered Izod test specimen blank. It can also be prepared from large, 1.005 in. (26.7 mm) thick sintered blanks which are sawed vertically into smaller pieces for turning into test specimens that are to be tested in the compacting direction. This compression test cylinder is not pressed to size because of its excessive length to diameter ratio.

6.6.1.1 The compressive strength of P/M materials is measured by use of an extensometer clamped to the gage length during the test following the procedures in Test Method E 9. The stress at 0.1 % or 0.2 % permanent offset is usually reported. When reporting the results, it is important that the
relationship between the original compacting direction and the testing direction be clearly noted.

6.6.2 Applicable ASTM Standards:

6.6.2.1 See Test Method E 9.

6.6.2.2 See the following P/M Material Specifications: B 783 and B 823.

IMPACT ENERGY TESTING

6.7 Izod Impact Test Specimen:

6.7.1 Description and Use—This P/M impact test specimen, shown in Fig. 7, is produced by compacting and sintering to the shape and dimensions of the standard Izod test bar. Typical tooling is shown in Fig. A2.5.

6.7.1.1 The standard industry practice for P/M material specifications is to report Izod impact energy as unnotched impact energy. It is determined in an Izod (cantilever-beam) impact test using a single-blown pendulum-type impact machine. The striking direction is 90 degrees to the original compacting direction. (If for other reasons, the Izod test specimen is to be tested in a notched condition, then refer to Test Method E 23 for specifications of notch types and testing procedures for notched bars.)

6.8 Charpy Impact Test Specimen:

6.8.1 Description and Use—This P/M test specimen, shown in Fig. 8, is produced by compacting and sintering to the shape and dimensions of the standard Charpy test bar. Typical tooling is shown in Fig. A2.6. It can also be prepared by shortening a sintered Izod test bar.

6.8.1.1 The standard industry practice for P/M material specifications is to report Charpy impact energy as unnotched impact energy. It is determined in a Charpy (simple-beam) impact test using a single-blown pendulum-type impact machine. The striking direction is 90 degrees to the original compacting direction. (If for other reasons, the Charpy bar is to be tested in a notched condition, then refer to Test Method E 23 for specifications of notch types and testing procedures for notched bars.)

6.8.2 Applicable ASTM Standards:

6.8.2.1 See Test Methods E 23.

6.8.2.2 See the following P/M Material Standards: B 783 and B 823.

FATIGUE TESTING

6.9 Machined Fatigue Test Specimen:

6.9.1 Description and Use—The rotating beam fatigue test specimen may be prepared by machining a sintered Izod blank, to the shape and dimensions shown in Fig. 9. It is very important that the reduced section be free of nicks, scratches, tool marks or any other conditions that can deleteriously affect the properties to be measured. This test specimen is used to determine the fatigue limit (endurance limit) and the fatigue
strength of sintered or heat treated P/M materials on an R. R. Moore type testing machine using rotating bending stresses in accordance with MPIF Standard 56.

6.9.2 Applicable Standards:
6.9.2.1 See MPIF Standard 56.
6.9.2.2 See P/M Material Specification B 783.

THERMAL EXPANSION TESTING
6.10 Machined Thermal Expansion Test Specimen:
6.10.1 Description and Use—This cylindrical test specimen, shown in Fig. 10, may be prepared by machining a sintered Izod test specimen blank, or a sintered Charpy test specimen blank. It is not compacted directly to size because of the extreme length to diameter ratio. This test specimen is used to determine the coefficient of thermal expansion with a push-rod style differential dilatometer using the procedures in Test Method E 228.
6.10.2 Applicable ASTM Standard:
6.10.2.1 See Test Method E 228.

MAGNETIC TESTING
6.11 Magnetic Ring Test Specimen:
6.11.1 Description and Use—This ring shaped test specimen, shown in Fig. 11, has been designed with a diameter and cross-section that allow easy winding and will give reliable and reproducible test data. It is generally compacted directly to size in tooling similar to that shown in Fig. A2.7.
6.11.1.1 Magnetic properties are a function of the state of the material and are adversely affected by machining, tumbling or cold working. P/M magnetic properties are generally measured on as-sintered material, but if the testing is being done to verify the magnetic properties of production parts, the testing shall be done on test specimens in the same state as that of the production parts. If a machined or repressed test specimen is intended to simulate as-sintered material, then the test specimen shall be annealed to eliminate stresses.
6.11.1.2 Permeability, coercivity and other magnetic properties are determined using standard ASTM test methods for magnetic properties. These test methods require a ring test specimen that has a ratio of the mean diameter to the radial width of not less than 10 to 1.
6.11.2 Applicable ASTM Standards:
6.11.2.1 See the following Test Methods: A 34/A 34M, A 341/A 341M, A 596/A 596M, A 773/A 773M, and A 927/A 927M.
6.11.2.2 See the following P/M Material Specifications: A 811, A 839/A 839M, and A 904.

FIG. 9 Machined R. R. Moore (Rotating-Beam) P/M Fatigue Test Specimen

FIG. 10 Machined Coefficient of Thermal Expansion P/M Test Specimen

FIG. 11 Typical P/M Ring Test Specimen for Measuring Magnetic Properties

7. Procedure
7.1 Obtain a test sample from the powder lot that is to be tested following the procedures in Practices B 215.
7.2 Record the following information about the powder lot or mix, as required:
7.2.1 Brand, grade and lot number of base metal powder,
7.2.2 Chemical composition of the alloy if not an elemental powder,
7.2.3 Brand, name, grade and percentage of all additives, and
7.2.4 Type, brand, grade and percentage of admixed lubricant.
7.3 The test specimens or blanks are produced by uniaxially compacting a test portion of the powder using double-action pressing. Information on the required test specimen tooling is presented in the Annexes.
7.3.1 Laboratory Tooling—Insert the lower punch into the die cavity. Position the die and lower punch on the lower press platen so that the die is supported on blocks and the lower punch is at the desired filling height. Follow the sequence in Fig. 12. Pour the powder test portion into the die cavity taking care to ensure that the powder is uniformly and evenly distributed. Insert the upper punch and apply and then release a pre-compacting pressure of approximately 5000 psi (35 MPa).

**NOTE 1**—If the powder mix does not contain an admixed lubricant, the die walls shall be coated with a lubricant prior to each pressing. A suspension of 100 g of zinc stearate in 1 L of methyl alcohol painted on the die walls and allowed to dry has been found to be satisfactory for this purpose. (This suspension is flammable and should be used in a suitable ventilated area.)

7.3.1.1 Remove the spacer blocks that have supported the die. (If the die is supported on springs, then the pre-compacting step is not needed.) Next, apply the final compacting pressure, typically 60 000 to 120 000 psi (415 to 830 MPa) depending upon the compressibility of the powder mix and the required green density of the test specimen. In special cases where the results may be affected by the rate of pressure application, a rate not exceeding 60 000 psi/min. (415 MPa/min.) is recommended.

7.3.1.2 Release the pressure as soon as the maximum pressure is attained, because pressure dwells of as little as 10 s can increase the green density of the test specimen by 0.3 %. Place two spacer blocks between the top of the die and the upper press platen. These ejection blocks should be longer than the combined lengths of the upper punch and the formed test specimen. If possible, remove the upper punch by hand. If not possible, apply pressure so that the ejection blocks push the die down. Then remove the upper punch when it clears the die. Continue to eject the green test specimen until it can be picked off the lower punch. Repeat these steps to obtain the desired number of test specimens.

7.3.2 Production Tooling—When compacting in a tool set adapted to a production P/M press, the die filling, pressing and ejection operations are all controlled by the programmed actions of the press. It usually is necessary for the powder mix to contain an admixed lubricant. A large number of identical test specimens can be rapidly produced when compacting in a production press.

7.4 Carefully deburr each test specimen with fine emery paper and determine the green density following the procedures in Test Method B 331. When producing multiple test specimens care should be taken to ensure that the green densities are held as uniform and consistent as possible. To indicate the density uniformity in a group of test specimens, the arithmetic mean green density (X) and the standard deviation (σ) shall be calculated and noted.

7.5 Record the following information about each green test specimen, as required:
7.5.1 Green dimensions,
7.5.2 Green mass,
7.5.3 Green density,
7.5.4 Type of press and compacting pressure, and
7.5.5 Die wall lubricant (if used).

7.6 If required, sinter the test specimens for the prescribed time at a temperature suitable for the material composition. See Table 1. This shall be done in a protective atmosphere or vacuum laboratory furnace capable of controlling the required sintering cycle or in a production P/M sintering furnace. See Fig. 13. Cool the test specimens to room temperature in the protective atmosphere before removing from the furnace and exposing to air.

7.7 Determine the sintered density of each test specimen following the procedure in Test Method B 328.

7.8 Record the following information about each sintered test specimen, as required:
7.8.1 Sintered dimensions,
7.8.2 Sintered mass,
7.8.3 Sintered density,
7.8.4 Sintering furnace, atmosphere and dew point, and
7.8.5 Heating rate, sintering time and temperature and cooling rate.

7.9 When preparing P/M test specimens by machining sintered blanks, single-point cemented carbide cutting tools with sharp cutting-point-radii are typically used. Machine using high turning speeds, fine feed rates and spray mist lubrication. Grinding may also be used to remove material when preparing a test specimen. Polish the machined test specimens longitudinally with progressively finer emery paper to remove tool marks and finish lap with crocus cloth.

7.10 If required by the testing program, additional operations, for example, heat treatment, steam treatment or oil-impregnation may be performed on the test specimens to duplicate production practice.

---

**TABLE 1 Typical Sintering Temperatures for Powder Metallurgy Materials**

<table>
<thead>
<tr>
<th>P/M Material</th>
<th>°F</th>
<th>°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>1100-1200</td>
<td>600-650</td>
</tr>
<tr>
<td>Brass</td>
<td>1600-1800</td>
<td>870-980</td>
</tr>
<tr>
<td>Bronze</td>
<td>1500-1600</td>
<td>815-870</td>
</tr>
<tr>
<td>Copper</td>
<td>1600-1900</td>
<td>870-1040</td>
</tr>
<tr>
<td>Copper Infiltrated Iron and Steel</td>
<td>2050-2200</td>
<td>1120-1200</td>
</tr>
<tr>
<td>Iron-Bronze</td>
<td>1600-1800</td>
<td>870-980</td>
</tr>
<tr>
<td>Iron and Carbon Steel</td>
<td>2050-2200</td>
<td>1120-1200</td>
</tr>
<tr>
<td>Iron-Copper and Copper Steel</td>
<td>2050-2200</td>
<td>1120-1200</td>
</tr>
<tr>
<td>Iron-Nickel and Nickel Steel</td>
<td>2050-2300</td>
<td>1120-1200</td>
</tr>
<tr>
<td>Low Alloy Steel</td>
<td>2050-2300</td>
<td>1120-1260</td>
</tr>
<tr>
<td>Magnetic Iron</td>
<td>2100-2400</td>
<td>1150-1320</td>
</tr>
<tr>
<td>Nickel Silver</td>
<td>1600-1800</td>
<td>870-980</td>
</tr>
<tr>
<td>Stainless Steel</td>
<td>2100-2400</td>
<td>1150-1320</td>
</tr>
<tr>
<td>Titanium Alloy</td>
<td>2100-2400</td>
<td>1150-1320</td>
</tr>
</tbody>
</table>
7.11 Refer to the ASTM Standard Test Method or Practice for which the test specimens were prepared and follow the procedures and calculations to obtain the property values.

8. Keywords
8.1 compacting tool set; die; metal powder properties; P/M materials; powder metallurgy tooling; powder testing; sintered material properties; test specimens
A1. Test Specimen Tooling—General Information

A1.1 P/M test specimens are produced using the same methods as those used to make P/M parts. This annex describes two types of tooling and presses that are used to compact green P/M test specimens.

A1.2 Laboratory Tooling—If only a few test specimens are needed for the evaluation, they are usually produced using laboratory tooling. This may consist of a simple die supported on blocks and two plain punches or a laboratory tool set made with a spring loaded die and an adjustable lower punch. The compacting force is supplied by an ordinary hydraulic platen press or a compression testing machine. When compacting in laboratory tooling, filling, compacting and ejection are all controlled manually. See Fig. 12 for the sequence of these manual operations.

A1.3 Production Tooling—When larger quantities of identical test specimens are required for a test program, they are then usually compacted in a tool set that has been designed and made to fit a production metal powder compacting press. With this system, the powder filling, compression, and ejection operations are automatically controlled by the programmed actions of the press cycle.
FIG. A1.2 Typical Laboratory Tooling—Transverse Rupture Test Specimen
A2. TEST SPECIMEN TOOLING—MATERIALS AND MANUFACTURE

A2.1 Powder metallurgy tooling shall be made from materials that will resist the abrasive action resulting from compacting metal powder and ejecting green parts, but still have the mechanical strength to withstand the hoop stresses resulting from high compacting pressures. The tooling shall be precisely made and be capable of producing multiple identical test specimens. Before being used, all components of the tooling shall be free of grease or oil and be fully demagnetized. This annex describes the requirements of the components of the tooling used to compact P/M test specimens.

A2.2 Test Specimen Dies—The die cavity for the test specimen is usually wire cut from a nonmachining grade of cemented carbide rated for light-impact applications (U.S. Carbide Industry Grade C-12). The die body length or thickness is dependent on the compression ratio of the powder being tested. Generally, 2 to 3 in. (50 to 75 mm) is adequate for dies that are used to compact P/M test specimens. The walls of the die cavity are finish lapped to a 4 µin. (0.1 µm) or better surface finish preferably parallel to the pressing direction. The perimeter of the die cavity at both the top and bottom of the die body shall have a 0.01 in. (0.25 mm) radius to facilitate punch entrance. The die body insert is securely contained within a steel die case. The series of drawings starting with Fig. A2.1 show the specifications for the dies that are needed to produce the P/M test specimens referenced in ASTM standards.

A2.3 Die Cases—The ring to hold the carbide die insert is usually made of AISI H-11 chromium hot-work tool steel hardened to 40 to 48 HRC. The amount of shrink between the die case and the die insert is typically 0.0015 to 0.0025 in. per in. (mm per mm) of insert diameter. The outside diameter of the die case shall be machined to fit the clamping system in the press table or die set platen in which the tooling is to be used.

A2.4 Punches—The upper and lower punches may be made of AISI A-2 or A-7 air hardening, medium-alloy, cold-work tool steel hardened to 60 to 62 HRC. The punches should fit the die with 0.0003 to 0.0005 in. (0.008 to 0.013 mm) clearance on each side and be lapped to a surface finish of 4 µin. (0.1 µm) or better. The punches shall move smoothly in the die cavity. The corner between the punch face and side is kept sharp to minimize flash on the green compact. The lower punch should be 0.25 in. (6 mm) longer than the thickness of the die to allow complete ejection of the green specimen. The upper punch is usually shorter. The ends of the punches may be machined to fit in the upper and lower punch clamps of the tool set or compacting press.

A2.5 Core Rods—For small diameter and long core rods, AISI M-2, molybdenum high-speed steel, in the form of centerless ground drill rod blanks is routinely used. For irregular core rods, various grades of tool steel hardened to 58 to 60 HRC or cemented carbide are the materials of choice. Core rods are lapped in the longitudinal direction to a 4 µin. (0.1 µm) or better surface finish. They shall move freely and smoothly in both upper and lower punches and are attached to the core rod support by means of a core rod adapter and core rod clamp.
FIG. A2.2 Die—Transverse Rupture Test Specimen

*DEMAN MAGNETIZE EACH PIECE
*ALL WORKING SURFACES TO BE 0–4 MICRO INCH FINISH
*TOLERANCES: 

<table>
<thead>
<tr>
<th>XX</th>
<th>±0.01</th>
</tr>
</thead>
<tbody>
<tr>
<td>XXX</td>
<td>±0.005</td>
</tr>
<tr>
<td>XXXX</td>
<td>±0.002</td>
</tr>
<tr>
<td>ANGLES</td>
<td>±0.50°</td>
</tr>
</tbody>
</table>

STEEL (40–45 HRC)
C12 CARBIDE

.5000 [12.700]
1.2500 [31.750]
4X R.010 [R0.25]
2.25 [57.2]
SHRINK FIT
R.010 [R0.25] BOTH ENDS
SECTION A–A
2.50 [63.5]

FIG. A2.3 Die and Core Rod—Typical Radial Crushing Strength Test Specimen

STEEL (40–45 HRC)
C12 CARBIDE

Ø 1.4000 [Ø 35.560]
R.010 [R0.25] BOTH ENDS
Ø 0.7500 [Ø 19.050]

*DEMAN MAGNETIZE EACH PIECE
*ALL WORKING SURFACES TO BE 0–4 MICRO INCH FINISH
*TOLERANCES: 

<table>
<thead>
<tr>
<th>XX</th>
<th>±0.01</th>
</tr>
</thead>
<tbody>
<tr>
<td>XXX</td>
<td>±0.005</td>
</tr>
<tr>
<td>XXXX</td>
<td>±0.002</td>
</tr>
<tr>
<td>ANGLES</td>
<td>±0.50°</td>
</tr>
</tbody>
</table>
FIG. A2.4 (a) Die—Flat Unmachined Tension Test Specimen

Die Dimensions

<table>
<thead>
<tr>
<th></th>
<th>in.</th>
<th>mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>A—Half length of reduced section</td>
<td>0.625</td>
<td>15.88</td>
</tr>
<tr>
<td>B—Grip length between centers</td>
<td>3.187 ± 0.001</td>
<td>80.95 ± 0.03</td>
</tr>
<tr>
<td>C—Width at grip section</td>
<td>0.342 ± 0.001</td>
<td>8.69 ± 0.03</td>
</tr>
<tr>
<td>D—Width at center</td>
<td>0.225 ± 0.001</td>
<td>5.72 ± 0.03</td>
</tr>
<tr>
<td>E—End radius</td>
<td>C/2</td>
<td>C/2</td>
</tr>
<tr>
<td>F—Half width at grip section</td>
<td>0.171 ± 0.001</td>
<td>4.34 ± 0.03</td>
</tr>
<tr>
<td>L—Overall length</td>
<td>3.529 ± 0.001</td>
<td>89.64 ± 0.03</td>
</tr>
<tr>
<td>R—Fillet radius</td>
<td>1.00</td>
<td>25.4</td>
</tr>
<tr>
<td>W—Width at end of reduced section</td>
<td>0.235 ± 0.001</td>
<td>5.97 ± 0.03</td>
</tr>
</tbody>
</table>

FIG. A2.4 (b) Die Cavity Detail—Flat Unmachined Tension Test Specimen (continued)
FIG. A2.5 Die—Izod Impact Energy Test Specimen

FIG. A2.6 Die—Charpy Impact Energy Test Specimen
FIG. A2.7 Die and Core Rod—Typical Ring Test Specimen for Magnetic Properties
Standard Test Method for
Rating Grain Size and Frequency of Abnormally Large
Grains in Cemented Tungsten Carbides (Hardmetals)¹

This standard is issued under the fixed designation B 930; the number immediately following the designation indicates the year of
original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A
superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes a procedure for measuring
abnormally large grains and the frequency of those grains in
cemented tungsten carbides (hardmetals).

1.2 This standard does not purport to address all of the
safety concerns, if any, associated with its use. It is the
responsibility of the user of this standard to establish appro-
priate safety and health practices and determine the applica-
bility of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 243 Terminology of Powder Metallurgy²
B 406 Test Method for Transverse Rupture Strength of
Cemented Carbides²
B 657 Test Method for Metallographic Determination of
Microstructure in Cemented Tungsten Carbides²
B 665 Guide for Metallographic Sample Preparation of
Cemented Tungsten Carbides²

3. Terminology

3.1 Definitions—Definitions of powder metallurgy terms
can be found in Terminology B 243.

3.2 Definitions of Terms Specific to This Standard:
3.2.1 E-Rating—the number/cm² of grains larger than a
specified size in a fully-etched specimen.
3.2.2 L-Rating—the size, in µm, of the largest grain ob-
served in a fully-etched specimen.

4. Summary of Test Method

4.1 A polished and fully etched specimen/specimens having
a minimum observable area of 0.5 cm² is examined using a
metallograph. Abnormally large grains, compared to the finer-
grained background material, are identified. These grains are
categorized as: (1) The number of grains larger than a specified
size, or (2) The largest grain observed, or both.

5. Significance and Use

5.1 The microstructure and grain growth of cemented tung-
sten carbides affect the material’s mechanical and physical
properties. The grain size and distribution will affect the
material’s wear resistance and fracture toughness. Abnormally
large grains as compared to the background may introduce an
area of weakness in a sintered part.

5.2 This test method may be used in acceptance testing of
cemented tungsten carbide materials or the tungsten carbide
powder used in their manufacture. The specified grain size used
for the E-Rating is to be agreed upon between purchaser and
supplier.

6. Apparatus

6.1 Metallographic Microscope, capable of magnifications
of up to 1500×.
6.2 Ordinary metallurgical laboratory equipment.
6.3 Equipment for specimen preparation as outlined in
Guide B 665.

7. Test Specimens

7.1 Specimen Size—The recommended specimen shall be
the standard transverse rupture specimen as specified in Test
Method B 406; that is, ground to the following dimensions:
5.00 ± 0.25 mm (0.200 ± 0.010 in) thick by 6.25 ± 0.25 mm
(0.250 ± 0.010 in) wide by 19.0 mm (0.750 ± in) long.
Alternatively, the specimen shall be a size to provide a
minimum 0.5 cm² surface area of examination, or it may
consist of several samples that provide this minimum area.

7.2 Specimen Preparation:
7.2.1 Polish the specimen/specimens according to the pro-
cedure described in Guide B 665, or other suitable metallo-
graphic polishing procedure. If using the recommended speci-
men, polish the 6.25 by 19.0 mm side.
7.2.2 Etch the specimen according to the procedure de-
scribed in Test Method B 657, or other suitable etching
procedure that will reveal the tungsten carbide grain structure.
8. Procedure

8.1 Examine a fully-etched specimen at a minimum of 200× magnification.

8.1.1 For E-rating—Count the number of grains larger than the specified size observed in the full cross-section of the polished and etched specimen/specimens. Divide this number by the total area examined (1.2 cm² for the recommended transverse rupture specimen) to obtain a number of grains/cm².

8.1.2 For L-Rating—Measure the largest dimension of the largest grain observed in the polished and etched specimen/specimens.

9. Report

9.1 The report shall include the following:

9.1.1 Reference to this test method,

9.1.2 Complete identification of the test specimen,

9.1.3 Details of any occurrence that may have affected the results,

9.1.4 The total area examined, in cm²,

9.1.5 The E-Rating as grains/cm², rounded to the nearest whole integer, and

9.1.6 The L-Rating in µm, rounded to the nearest whole integer.

10. Precision and Bias

10.1 No statement regarding precision and bias of this test method can be made at this time. Plans for an interlaboratory study are being formulated.

11. Keywords

11.1 abnormally large grains; cemented carbides; cemented tungsten carbide; E-rating; hardmetals; L-rating; microstructure
Standard Test Method for
Metallographically Estimating the Observed Case Depth of
Ferrous Powder Metallurgy (P/M) Parts

This standard is issued under the fixed designation B 931; the number immediately following the designation indicates the year of
original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A
superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 A metallographic method is described for estimating the
observed case depth of ferrous powder metallurgy (P/M) parts.
This method may be used for all types of hardened cases where
there is a discernible difference between the microstructure of
the hardened surface and that of the interior of the part.

1.2 This standard does not purport to address all of the
safety concerns, if any, associated with its use. It is the
responsibility of the user of this standard to establish appro-
priate safety and health practices and determine the applica-
bility of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
B 243 Terminology of Powder Metallurgy
E 407 Practice for Microetching Metals and Alloys

3. Terminology

3.1 Definitions—Definitions of powder metallurgy (P/M)
terms can be found in Terminology B 243. Additional descrip-
tive information is available in the Related Material section of
Vol 02.05 of the Annual Book of ASTM Standards.

3.2 The metallographically estimated observed case depth is
defined as the distance from the surface of the part to the point
where, at a magnification of 100×, there is a discernible
difference in the microstructure of the material.

4. Summary of Test Method

4.1 The powder metallurgy part is sectioned and the surface
prepared for metallographic evaluation. The metallographic
specimen is etched and the distance is measured from the
surface of the part to the point at which a discernible difference
in the microstructure of the material is observed.

5. Significance and Use

5.1 The engineering function of many P/M parts may
require an exterior portion of the part to have a hardened layer.

Where case hardening produces a distinct transition in the
microstructure, metallographic estimation of the observed case
depth may be used to check the depth to which the surface has
been hardened.

6. Apparatus

6.1 Equipment for the metallographic preparation of test
specimens—see Appendix X1.

6.2 Metallographic Microscope, permitting observation and
measurement at a magnification of 100×.

7. Reagents and Materials

7.1 Etchants such as 2 to 5 % nital, nital/picral combina-
tions, or other suitable etchants. For more information on
suitable etchants refer to Practice E 407.

8. Test Specimens

8.1 Cut a test specimen from the P/M part, perpendicular to
the hardened surface at a specified location, being careful to
avoid any cutting or grinding procedure that would affect the
original microstructure.

8.2 Mounting of the test specimen is recommended for
convenience in surface preparation and edge retention. Edge
retention is important for proper measurement of the observed
case depth.

9. Procedure

9.1 Grind and polish the test specimen using methods such
as those summarized in Appendix X1.

9.2 Etch the specimen with etchants such as 2 to 5 % nital
or nital/picral combinations.

9.2.1 Observed Case Depth:

9.2.1.1 Examine the surface region of the part at a magni-
fication of 100×.

9.2.1.2 Measure the distance from the surface of the part to
the point where there is a discernible difference in the micro-
structure of the material.

NOTE 1—The nature and amount of intermediate transformation prod-
ucts will depend on the material being heat treated, its density, and the
type of surface hardening treatment being used. The sharpness of the
change in the microstructure at the point of transition will therefore vary.
The microstructure expected at this transition point should be agreed
between the producer and user of the part. Magnifications higher than
100× may be used to check the microstructure of the part in the region of

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the transition zone. However, the metallographic estimate of the observed case depth shall be made at a magnification of 100×.

10. Report

10.1 Report the following information:
10.1.1 The type of material and case measured,
10.1.2 The type of etchant used,
10.1.3 The location of the measurement, and
10.1.4 The metallographically estimated observed case depth to the nearest 0.1 mm.

APPENDIX

(Nonmandatory Information)

X1. SAMPLE PREPARATION

X1.1 The methods described in this appendix are proven practices for metallographic preparation of porous P/M materials. It is recognized that other procedures or materials used in preparation of a sample may be equally as good and can be used on the basis of availability and preference of individual laboratories.

X1.2 Method 1:

X1.2.1 The porous samples should be free of oil or coolant. Remove any oil using Soxhlet extraction. Mount and vacuum impregnate samples with epoxy resin, to fill porosity and to prevent the pickup of etchants. Use a sample cup or holder to form the mount. Pour epoxy resin over the sample in the cup to a total depth of about 0.75 in (19 mm). Evacuate the cup to minus 26 in. of mercury (88 kPa) and hold at that pressure for 10 min. Then restore ambient air pressure to force the resin into most of the sample. Cure at room temperature or at 122°F (50°C).

X1.2.2 Grind on 240, 400, and 600 grit wet SiC paper, on a rotating wheel, and change the polishing direction 90° after each paper. Etch samples for 1 min in their normal etchant, for example, 2 % nital, to begin to open the porosity. Rough polishing for 8 to 12 min total on 1 µm alumina (Al₂O₃), long napped cloth (for example Struers felt cloth), at 250 rpm, and 300 gf load, using an automated polisher opens smeared pores. This rough polishing opens and exaggerates the pores. To return the pores to their true area fraction, polish for 4 min at 125 rpm on a shorter nap cloth (for example Struers MOL cloth), with 1 µm diamond paste. Final polishing is done for 20 to 30 s using 0.05 µm deagglomerated alumina, and a long napped cloth (for example, Buehler Microcloth), at 125 rpm, and 75 gf load, on an automated polisher. Polishing may also be done by hand for the times indicated. The first two polishings require moderate pressure and the final polish requires light pressure.

X1.2.3 The metallographic structure should be free of smeared porosity. Generally at 800 to 1000×, the edge of a smeared over pore will appear as a thin gray line outlining one side of the pore, and occasionally outlining most of the pore.

X1.3 Method 2:

X1.3.1 The specimen should be carefully selected so that it is representative of the region of interest. After selection, the specimen may require sectioning to provide a workable specimen. Sectioning may be made employing an abrasive or diamond wheel.

X1.3.2 Heat should be avoided to prevent occurrence of possible changes in microstructure. If slow feeds are employed, a coolant may not be necessary to avoid temperature buildups. If abrasive wheels are used, then a coolant is often necessary to avoid overheating of the specimen.

X1.3.3 If a coolant is employed, it may be retained within the pores. The lubricant must be removed prior to the preparation of the specimen for examination. This may be accomplished by using a Soxhlet extractor or an ultrasonic cleaner. The extraction condenser is the most efficient and the least expensive.

X1.3.4 Generally, specimens to be evaluated for case depth are mounted to provide edge retention. There are many kinds of mounting compounds available. Most common materials include epoxies (powder or liquid), diallyl phthalate, or Bakelite. Of these, Bakelite is sometimes preferred because it is harder and therefore provides improved edge retention. Bakelite requires equipment to apply heat and pressure, whereas the epoxies do not.

X1.3.5 After mounting, the specimen is ground to provide a flat, stress-free surface. A belt grinder is generally used first with care to prevent heating of the specimen. Grit size is dependent on the preference of the metallographer, although finer grits are preferred.

X1.3.6 The specimen is then hand ground on four emery papers, generally of 240, 320, 400, and 600 grit.

X1.3.7 Etch samples for 1 min in their normal etchant, for example, 2 % nital, to begin to open the porosity.

X1.3.8 Wet polishing follows hand grinding and etching. Several polishing media are employed including diamond paste, magnesia, alumina, etc. Grit size varies between 1 and 0.3 µm and is applied to nap-free cloths such as nylon. To remove remaining scratches and stress, a soft cloth with finer polishing compound is employed. Generally a short napped
cloth is preferred. A fine 0.5 µm alumina is recommended. For best results, and to ensure complete freedom of pores from worked metal, repeat the polishing and etching procedure. Final polishing generally requires 3 to 5 min.

X1.3.9 Automated polishing equipment is also available. Automated polishing is accomplished by moving the specimen across a polishing cloth in an abrasive solution undergoing vibrating action. Cloths and abrasives available are numerous and are generally selected by experience of the metallographer.

X1.4 Two additional schemes for the preparation of sintered ferrous materials, one manual and the other automated, are discussed. The first method, basic manual preparation, has most likely been used to prepare more samples for metallographic examination than any other single method. The assumption is made that the sample has been mounted and pre-ground to give a planar surface. Vacuum impregnation with an epoxy resin is recommended for samples to be used in case depth measurement.

**X1.4.1 Basic Manual Sample Preparation:**

**X1.4.1.1 Grind samples using progressively finer abrasive papers.**

(a) Routinely, 240, 320, 400, then 600 grit (U.S. Standard designation) SiC abrasive paper strips are used.

(b) Lubricate and cool the sample with a continuous flow of water.

(c) Rotate the sample 90° before proceeding to the next paper.

(d) Clean the surface of the sample with a soft cloth or paper towel before grinding on each paper.

**Note X1.1—Do not progress to the next paper strip until all evidence of the previous step has been removed.**

**X1.4.1.2 Etching prior to polishing. This step is optional.**

(a) Etch with 2 or 5 % nital prior to the first polishing step.

(b) Rinse with running water and dry with filtered, dry, compressed air.

**X1.4.1.3 Coarse polish—single step.**

(a) Use a slurry made of distilled or deionized water with 1 µm Al₂O₃. Polish using a Nylon cloth.

(b) Charge the cloth with the slurry at the start of the cycle and periodically as the cloth becomes dry.

(c) Pressure applied to the sample should be moderate to heavy and movement should be counter to the direction of the polishing wheel.

(d) Wash the sample with soap and water using a soft material such as cotton.

(e) Rinse with running water.

(f) Dry the surface using filtered, dry, compressed air.

(g) Repeat this step until the porosity appears to be open and the appearance of the specimen is uniform from edge to edge.

(h) Periodically clean the cloth. Keep the surface free of built-up slurry and polishing debris.

**X1.4.1.4 Fine polish—single step.**

(a) Use a slurry made of distilled or deionized water and 0.05 µm Al₂O₃. Polish using a soft, napped, fine, polishing cloth.

(b) Charging of the cloth, pressure applied to the sample, direction of sample movement, and cleaning of the sample are similar to the conditions used in coarse polishing.

(c) Use short polishing times to minimize rounding and relief.

(d) Perform the operations described in X1.4.1.3(d), (e), and (f).

X1.4.1.5 Dry the sample in a vacuum chamber in order to remove entrapped moisture.

X1.4.1.6 Remove any stains by washing with soap and water.

(a) Dry with compressed air.

**X1.4.2 Basic Automated Sample Preparation:**

**X1.4.2.1 Clamp or set the samples in the multi-sample holder.**

(a) Try to prepare materials with similar composition and hardness at one time.

**X1.4.2.2 Grind samples using progressively finer abrasive papers.**

(a) Use 240, 320, 400, then 600 grit (U.S. Standard designation) SiC paper disks. The use of interrupted cut composite disks in place of most of the grinding papers is also acceptable. The disk is usually charged with 15 or 30 µm diamond spray.

(b) Cool and lubricate with a continuous flow of prepared fluid.

(c) Use pressure of 40 to 55 kPa and a time no longer than 30 s.

(d) Rinse the platen and sample before progressing to the next paper.

(e) Dry the samples using filtered, dry, compressed air.

**X1.4.2.3 Etching prior to polishing. This step is optional.**

(a) Etch with 2 or 5 % nital prior to the first polishing step.

(b) Rinse with running water and dry with filtered, dry, compressed air.

**X1.4.2.4 Coarse polish using two steps.**

(a) Polish using 6 µm diamond polish on a hard cloth, that is, Nylon or chemotextile.

(b) Polishing time should be approximately 3 min, at a pressure of 40 to 55 kPa.

(c) Charge the cloth at the start of the cycle and at one minute intervals using aerosol propelled diamond spray.

(d) Ultrasonically clean the samples—do not remove from the holder.

(e) Wash polished surfaces using soap and water.

(f) Dry the surface using compressed air.

(g) Polish using 3 µm diamond polish on a second hard cloth, that is, woven or synthetic silk.

(h) Polishing time should be 2 to 3 min at a pressure of 40 to 55 kPa.

(i) Repeat steps X1.4.2.4(c) through (f) as described above.

(j) Take care not to contaminate the cloth used in the second step of coarse polishing with polish and debris from the first step.

**X1.4.2.5 Fine polish.**

(a) Polish using 1 µm diamond polish on a soft napped cloth.

(b) Polishing time should be 1 to 2 min.
(c) Perform steps X1.4.2.4(c) through (f) as described above (use pressure toward the low end of the range).
X1.4.2.6 Dry the sample in a vacuum chamber in order to remove entrapped moisture.

X1.4.2.7 Remove stains by washing with soap and water.
(a) Dry with compressed air.

1. Scope

1.1 This test method covers the determination of the microindentation hardness of powder metallurgy (P/M) materials. The test method differs from the approach used for pore-free materials in terms of the precautions required to deal with the porosity.

1.2 A method for converting the directly measured indentation lengths to other hardness scales, for example, HRC is described in Appendix X1.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   B 243 Terminology of Powder Metallurgy
   E 384 Test Method for Microindentation Hardness of Materials
   E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 Definitions of powder metallurgy (P/M) terms can be found in Terminology B 243. Additional descriptive information is available in the Related Materials section of Vol 02.05 of the Annual Book of ASTM Standards.

4. Summary of Test Method

4.1 Microindentation hardness testing uses a calibrated machine to force a pyramidal-pointed diamond indenter into the surface of the test material under a known test load. The microindentation hardness value is calculated from the indenting force divided by the projected area of the resulting indentation.

NOTE 1—This test method is designed specifically for use on porous P/M materials. It is intended to be a companion to Test Method E 384. There are specific differences that are intentional; otherwise, the details on equipment and procedures in Test Method E 384 shall be adhered to. The specific differences relate to the presence of porosity in the P/M materials. Special precautions are required during sample preparation to reveal pores and heterogeneous microstructural features so that appropriate test locations may be selected.

5. Significance and Use

5.1 Microindentation hardness testing provides a measure of the hardness of the microstructural constituents of a porous material. It indicates the hardness the material would have if there were no pores present and the material was tested using macroindentation hardness methods.

5.2 Microindentation hardness tests allow the evaluation of specific phases, microstructural constituents, and regions or gradients too small for macroindentation hardness testing.

6. Apparatus

6.1 Microindentation Hardness Testing Machine, capable of applying the required load, equipped with a Knoop or Vickers indenter, and provision for measuring the length of the diagonals of the indentation.

6.2 Apparatus requirements are summarized in method Test Method E 384.

7. Reagents and Materials

7.1 Metallographic Etchants, suitable for the material being tested.

8. Test Specimen

8.1 Specimen Mounting:
8.1.1 Sample mounting is recommended for convenience in surface preparation, edge retention, and ease of testing. The sample should be supported adequately in the mounting medium.

8.2 Specimen Preparation:
8.2.1 Guidelines for grinding and polishing specimens are provided in Appendix X2.

8.2.2 Care should be taken to ensure that the true area fraction of porosity is revealed throughout the entire cross section of the specimen. It is essential in surface preparation to remove all smeared metal and to identify pores clearly so that they may be avoided during testing.

8.2.3 The specimen should be lightly etched prior to micro-indentation hardness testing. Careful etching is necessary as heavy etching obscures features and interferes with the measurement of the diagonals of the indentation.

8.2.4 For heat treated steels, swabbing with or immersion in 2% nital for 4 to 7 s gives an appropriate structure.

9. Procedure

9.1 Support the specimen so that its surface is perpendicular to the axis of the indenter.

9.2 Select a suitable location for testing and an appropriate load and magnification for the test. A 100 gf load is recommended for hardened materials. Lower loads may be used for softer materials or when small regions need to be tested. For the best precision, use the highest load compatible with the feature to be tested. Magnification ranges for various indentation lengths are as follows:

<table>
<thead>
<tr>
<th>Indentation Length (µm)</th>
<th>Magnification</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;76</td>
<td>Max 800, Min 200</td>
</tr>
<tr>
<td>76 to 125</td>
<td>600</td>
</tr>
<tr>
<td>&gt;125</td>
<td>400</td>
</tr>
</tbody>
</table>

9.3 Apply the test load.

9.4 Examine the indentation for possible sources of error such as distorted or unusually large indentations. The two sections of each diagonal should agree within 20% of each other. Discard any distorted or unusually large indentations. Unusually large indentations sometimes occur due to the presence of pores directly under the indentation.

9.5 Measure the length of the diagonals of the indentation, taking care to avoid backlash by moving only in one direction. For Knoop microindentation hardness, read the length of the larger diagonal to 0.1 µm. For Vickers microindentation hardness, measure both diagonals to the nearest 0.1 µm and calculate the average.

9.6 Make additional indentations. Space the indentations, so that adjacent tests do not interfere with each other. The minimum spacing between tests is illustrated in Fig. 1.

9.7 Discard any value if by including this value the hardness range of the other points is more than doubled. In all cases of a discarded value, make a replacement.

10. Calculation or Interpretation of Results

10.1 The Knoop or Vickers microindentation hardness numbers may be calculated using the following formulae or by using tables in Test Method E 384.

10.1.1 Knoop — Using the units of force and length commonly employed, that is, for force \( P \) in gf, and a long diagonal \( d \) in micrometres, the Knoop hardness is calculated:

\[
HK = \frac{14229 P}{d^2}
\]

10.1.2 Vickers — Using the units of force and length commonly employed, that is, for force \( P \) in gf, and the mean of the two diagonals \( d \) in micrometres, the Vickers hardness is calculated:

\[
HV = \frac{1854.4 P}{d^2}
\]

10.1.3 For indentation diagonals measured in millimetres, tables of \( HK \) and \( HV \) values are tabulated in Test Method E 384.
11. Report

11.1 Report the following information:

11.1.1 The identification of the sample and the location at which the microindentation hardness was measured,
11.1.2 The type of indenter, Knoop or Vickers,
11.1.3 The magnification used,
11.1.4 The identity, or description of the phase or microstructural constituent measured,
11.1.5 The type of etchant used, the duration, and method of etching, and
11.1.6 The average of at least five acceptable measurements shall be reported as the microindentation hardness of the material, microstructural constituent, or other feature measured.

11.1.7 Knoop (HK) or Vickers (HV) microindentation hardness shall be reported along with the test load used, for example, 400 HK 100 gf or 400 HV 100 gf. This is the preferred method. However, an alternative method expressing the load in kilograms force may be used in accordance with ISO, for example, 400 HK 0.1 or 400 HV 0.1. Report HK and HV values to the nearest whole number.

12. Precision and Bias

12.1 The repeatability r and reproducibility R of measurements were determined in accordance with Practice E 691. Members of the Powder Metallurgy Parts Association of the Metal Powder Industries Federation conducted the interlaboratory test program. The test sample was prepared from heat treated FL-4605. One Knoop and one Vickers microindentation hardness indent was made in the surface of the test sample, and these indentations were measured by 12 participating laboratories.

12.2 The mean Knoop microindentation hardness value was 701 HK 100 gf with a repeatability of 22 and a reproducibility of 76. Duplicate microindentation hardness results from one laboratory should not be considered suspect at the 95 % confidence level unless they differ by more than 22. For the same test specimen, Knoop microindentation hardness results from two different laboratories should not be considered suspect at the 95 % confidence level unless they differ by more than 76.

12.3 The mean Vickers microindentation hardness value was 716 HV 100 gf with a repeatability of 43 and a reproducibility of 178. Duplicate microindentation hardness results from one laboratory should not be considered suspect at the 95 % confidence level unless they differ by more than 43. For the same test specimen, Vickers microindentation hardness results from two different laboratories should not be considered suspect at the 95 % confidence level unless they differ by more than 178.

13. Keywords

13.1 Knoop microindentation hardness; microindentation hardness; P/M; powder metallurgy; Vickers microindentation hardness

APPENDIXES

(Nonmandatory Information)

X1. CONVERSION TO OTHER HARDNESS SCALES

X1.1 It is sometimes desired to express microindentation hardness values in terms of equivalents to other hardness scales, for example, HRC. There is no direct conversion from microindentation hardness to HRC. Approximate values can be obtained through the procedure described in this appendix.

X1.1.1 The following procedure describes a method for conversion to HRC.

X1.1.2 Obtain four or five standard HRC test blocks that span the range from the low 20’s HRC to the 60’s HRC.

X1.1.3 Remove a small portion from each standard test block, being careful to avoid any procedure that might affect the hardness of the test block material, and make a metallographic mount with the standardized face of the test block at the surface of the mount.

X1.1.4 Polish the specimens using standard procedures (see Appendix X2).

X1.1.5 Using either a Knoop or a Vickers indenter and a 100 gf test load (other loads might be used for a conversion to hardness scales such as HRB or HRF), make five indentations at various points in each of the standard specimens.

X1.1.6 Measure the length of the diagonals of the indentations.

X1.1.7 Prepare a graph with the filar units, micrometres, or Knoop/Vickers microindentation hardness number on the y-axis (ordinate) and HRC on the x-axis (abscissa). Plot all measured diagonals and, using regression analysis (regression of y on x), construct a best-fit curve to the data points.

X1.1.8 In future tests, take any diagonal reading and use the graph to convert to HRC.

NOTE X1.1—The graph that is constructed applies to the specific instrument used for the microindentation hardness test, the test load used, and the person performing the test. A separate graph needs to be plotted for each operator, each test instrument, and for each load used for microindentation hardness testing.

X1.1.9 Precision of the Graphical Conversion:

X1.1.9.1 Seven laboratories participated in an interlaboratory study. Each laboratory developed a regression line for their own instrument. The regression line was plotted based on the results (six-reading averages) of measurements on five HRC standard test blocks with hardness ranging from 25.4 HRC to 63.2 HRC. The seven laboratories found the hardness of a circulated unknown sample to average 56.5 HRC.

X1.1.9.2 With this test method, 95 % of any future readings would be expected to repeat in a laboratory within 4.0 HRC
points at this level; for six-reading averages within 1.6 HRC points. For a laboratory to duplicate any of the other laborato-
ries, 95% of the readings should be within 5.3 HRC; for six-reading averages within 2.2 HRC.

X2. SAMPLE PREPARATION

X2.1 The methods described in this appendix are proven practices for metallographic preparation of porous P/M materials for microindentation hardness testing. It is recognized that other procedures or materials used in preparation of a sample may be equally as good and can be used on the basis of availability and preference of individual laboratories.

X2.2 Method 1:

X2.2.1 The porous samples should be free of oil or coolant. Remove any oil using Soxhlet extraction. Mount and vacuum impregnate samples with epoxy resin, to fill porosity and to prevent the pickup of etchants. Use a sample cup or holder to form the mount. Pour epoxy resin over the sample in the cup to a total depth of about 0.75 in. (19 mm). Evacuate the cup to minus 26 in. of mercury (88 kPa) and hold at that pressure for 10 min. Then restore ambient air pressure to force the resin into most of the sample. Cure at room temperature or at 122°F (50°C).

X2.2.2 Grind on 240, 400, and 600 grit wet SiC paper, on a rotating wheel, and change the polishing direction 90° after each paper. Etch samples for 1 min in their normal etchant, for example, 2% nital, to begin to open the porosity. Rough polishing for 8 to 12 min total on 1 µm alumina (Al₂O₃), long napped cloth (for example, Struers felt cloth), at 250 rpm, and 300 gf load, using an automated polisher opens smeared pores. This rough polishing opens and exaggerates the pores. To return the pores to their true area fraction, polish for 4 min at 125 rpm on a shorter nap cloth (for example, Struers MOL cloth), with 1 µm diamond paste. Final polishing is done for 20 to 30 s using 0.05 µm deagglomerated alumina, and a long napped cloth (for example, Buehler Microcloth), at 125 rpm, and 75 gf load, on an automated polisher. Polishing may also be done by hand for the times indicated. The first two polishings require moderate pressure and the final polish requires light pressure.

X2.2.3 The metallographic structure should be free of smeared porosity. Generally at 800 to 1000×, the edge of a smeared over pore will appear as a thin gray line outlining one side of the pore, and occasionally outlining most of the pore.

X2.2.4 The specimen should be etched prior to microindentation hardness testing. Careful etching is necessary because heavy etching obscures features and may interfere with the measurement of the diagonals. For heat-treated steels, swabbing with or immersion in 2% nital for 4 to 7 s gives a suitable structure. Martensite will be very light and the darker etching non-martensitic transformation products such as upper bainite or fine pearlitic will be evident by contrast. Materials with complex, multi-constituent microstructures should be tested in the lightly etched condition.

X2.3 Method 2:

X2.3.1 The specimen should be carefully selected so that it is representative of the region of interest. After selection, the specimen may require sectioning to provide a workable specimen. Sectioning may be made employing a hacksaw, bandsaw, abrasive, or diamond wheel. A hacksaw is sufficient for soft materials. However, if harder materials are of interest, then an abrasive or diamond wheel may be required.

X2.3.2 Heat should be avoided to prevent occurrence of possible changes in microstructure. If slow feeds are employed, a coolant may not be necessary to avoid temperature build-ups. If abrasive wheels are used, then a coolant is often necessary to avoid overheating of the specimen.

X2.3.3 If a coolant is employed, it may be retained within the pores. The lubricant must be removed prior to the preparation of the specimen for examination. This may be accomplished by using a Soxhlet extractor or an ultrasonic cleaner. The extraction condenser is the most efficient and the least expensive.

X2.3.4 Generally, specimens to be evaluated for microindentation hardness are mounted to provide edge retention. There are many kinds of mounting compounds available. Most common materials include epoxies (powder or liquid), diallyl phthalate, or Bakelite. Of these, Bakelite is sometimes preferred because it is harder and therefore provides improved edge retention. Bakelite requires equipment to apply heat and pressure, whereas the epoxies do not.

X2.3.5 After mounting, the specimen is ground to provide a flat, stress-free surface. A belt grinder is generally used first with care to prevent heating of the specimen. Grit size is dependent on the preference of the metallographer, although finer grits are preferred.

X2.3.6 The specimen is then hand ground on four emery papers, generally of 240, 320, 400, and 600 grit.

X2.3.7 Etch samples for 1 min in their normal etchant, for example, 2% nital for P/M steels, to begin to open the porosity.

X2.3.8 Wet polishing follows hand grinding and etching. Several polishing media are employed, including diamond paste, magnesia, alumina, etc. Grit size varies between 1 and 0.3 µm and is applied to nap-free cloths such as nylon. To remove remaining scratches and stress, a soft cloth with finer polishing compound is employed. A short napped cloth is generally preferred. A fine 0.05 µm alumina is recommended. For best results, and to ensure complete freedom of pores from worked metal, repeat the polishing and etching procedure. Final polishing generally requires 3 to 5 min.

X2.3.9 Automated polishing equipment is also available. Automated polishing is accomplished by moving the specimen across a polishing cloth in an abrasive solution undergoing vibrating action. Cloths and abrasives available are numerous and are generally selected by experience of the metallographer.

X2.3.10 The specimen should be etched prior to microindentation hardness testing. Careful etching is necessary because heavy etching obscures features and may interfere with the measurement of the diagonals. For heat-treated steels,
swabbing with or immersion in 2% nital for 4 to 7 s gives an appropriate structure. Martensite will be very light and the darker etching non-martensitic transformation products such as upper bainite or fine pearlite will be evident by contrast. Materials with complex, multi-constituent microstructures should be tested in the lightly etched condition.

X2.4 Two additional schemes for the preparation of sintered ferrous materials, one manual and the other automated, are discussed. The first method, basic manual preparation, probably has been used to prepare more samples for metallographic examination than any other single method. The assumption is made that the sample has been mounted and pre-ground to give a planar surface. Vacuum impregnation with an epoxy resin is recommended for samples to be used in microindentation hardness testing.

X2.4.1 Basic Manual Sample Preparation:

X2.4.1.1 Grind samples using progressively finer abrasive papers.

(a) Routinely, 240, 320, 400, then 600 grit (U.S. Standard designation) SiC abrasive paper strips are used.

(b) Charge the cloth with the slurry at the start of the cycle and periodically as the cloth becomes dry.

(c) Rinse with running water.

(d) Lubricate and cool the sample with a continuous flow of water.

(e) Periodically clean the cloth. Keep the surface free of entrapped moisture.

(f) Pressure applied to the sample should be moderate to heavy and movement should be counter to the direction of the polishing wheel.

(g) Rinse with running water.

(h) Use a slurry made of distilled or deionized water and 1 µm Al₂O₃. Polish using a Nylon cloth.

(i) Periodically clean the cloth. Keep the surface free of built-up slurry and polishing debris.

X2.4.1.2 Etching prior to polishing. This step is optional.

(a) Etch with 2 or 5% nital prior to the first polishing step.

(b) Rinse with running water and dry with filtered, dry, compressed air.

X2.4.1.3 Coarse polish—single step.

(a) Use a slurry made of distilled or deionized water with 1 µm Al₂O₃. Polish using a Nylon cloth.

(b) Charge the cloth with the slurry at the start of the cycle and periodically as the cloth becomes dry.

(c) Pressure applied to the sample should be moderate to heavy and movement should be counter to the direction of the polishing wheel.

(d) Wash the sample with soap and water using a soft material such as cotton.

(e) Rinse with running water.

(f) Dry the surface using filtered, dry, compressed air.

(g) Repeat this step until the porosity appears to be open and the appearance of the specimen is uniform from edge to edge.

(h) Periodically clean the cloth. Keep the surface free of built-up slurry and polishing debris.

X2.4.1.4 Fine polish—single step.

(a) Use a slurry made of distilled or deionized water and 0.05 µm Al₂O₃. Polish using a soft, napped, fine polishing cloth.

(b) Charging of the cloth, pressure applied to the sample, direction of sample movement, and cleaning of the sample are similar to the conditions used in coarse polishing.

(c) Use short polishing times to minimize rounding and relief.

(d) Perform the operations described in X2.4.1.3 (d), (e), and (f).

X2.4.1.5 Dry the sample in a vacuum chamber in order to remove entrapped moisture.

X2.4.1.6 Remove any stains by washing with soap and water.

(a) Dry with compressed air.

X2.4.1.7 The specimen should be etched prior to microindentation hardness testing. Careful etching is necessary because heavy etching obscures features and may interfere with the measurement of the diagonals. For heat-treated steels, swabbing with or immersion in 2% nital for 4 to 7 s gives an appropriate structure. Martensite will be very light and the darker etching non-martensitic transformation products such as upper bainite or fine pearlite will be evident by contrast. Materials with complex, multi-constituent microstructures should be tested in the lightly etched condition.

X2.4.2 Basic Automated Sample Preparation:

X2.4.2.1 Clamp or set the samples in the multi-sample holder.

(a) Try to prepare materials with similar composition and hardness at one time.

X2.4.2.2 Grind samples using progressively finer abrasive papers.

(a) Use 240, 320, 400, then 600 grit (U.S. Standard designation) SiC paper disks. The use of interrupted cut composite disks in place of most of the grinding papers is also acceptable. The disk is usually charged with 15 or 30 µm diamond spray.

(b) Cool and lubricate with a continuous flow of prepared fluid.

(c) Use pressure of 40 to 55 kPa and a time no longer than 30 s.

(d) Rinse the platen and sample before progressing to the next paper.

(e) Dry the samples using filtered, dry compressed air.

X2.4.2.3 Etching prior to polishing. This step is optional.

(a) Etch with 2 or 5% nital prior to the first polishing step.

(b) Rinse with running water and dry with filtered, dry compressed air.

X2.4.2.4 Coarse polish using two steps.

(a) Polish using 6 µm diamond polish on a hard cloth, for example, Nylon or chemotextile.

(b) Polishing time should be approximately 3 min, at a pressure of 40 to 55 kPa.

(c) Charge the cloth at the start of the cycle and at 1 min intervals using aerosol propelled diamond spray.

(d) Ultrasonically clean the samples. Do not remove from the holder.

(e) Wash polished surfaces using soap and water.

(f) Dry the surface using compressed air.

(g) Polish using 3 µm diamond polish on a second hard cloth, that is, woven or synthetic silk.

(h) Polishing time should be 2 to 3 min at a pressure of 40 to 55 kPa.

(i) Repeat steps X2.4.2.4 (c) through (f) as described above.
(j) Take care not to contaminate the cloth used in the second step of coarse polishing with polish and debris from the first step.

X2.4.2.5 Fine polish.
(a) Polish using 1 µm diamond polish on a soft napped cloth.
(b) Polishing time should be 1 to 2 min.
(c) Perform steps X2.4.2.4 (c) through (f) as described above (use pressure toward the low end of the range).

X2.4.2.6 Dry the sample in a vacuum chamber in order to remove entrapped moisture.

X2.4.2.7 Remove stains by washing with soap and water.

(a) Dry with compressed air.

X2.4.2.8 The specimen should be etched prior to microindentation hardness testing. Careful etching is necessary because heavy etching obscures features and may interfere with the measurement of the diagonals. For heat-treated steels, swabbing with or immersion in 2 % nital for 4 to 7 s gives an appropriate structure. Martensite will be very light and the darker etching non-martensitic transformation products such as upper bainite and fine pearlite will be evident by contrast. Materials with complex, multi-constituent microstructures should be tested in the lightly etched condition.
Standard Test Method for Effective Case Depth of Ferrous Powder Metallurgy (P/M) Parts Using Microindentation Hardness Measurements

This standard is issued under the fixed designation B 934; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reaffirmation. A superscript epsilon (ε) indicates an editorial change since the last revision or reaffirmation.

1. Scope

1.1 This test method covers a procedure for determination of the effective case depth of powder metallurgy (P/M) parts.
1.2 A microindentation hardness traverse procedure is described to determine effective case depth. This test method may be used to determine the effective case depth for all types of hardened cases.
1.3 The procedure for determining the microindentation hardness of powder metallurgy materials, as described in Test Method B 933 shall be followed.
1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   B 243 Terminology of Powder Metallurgy
   B 933 Test Method for Microindentation Hardness of Powder Metallurgy (P/M) Materials
   E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials
   E 384 Test Method for Microindentation Hardness of Materials
2.2 Other Standard:
   SAE J 423 Recommended Practice, Methods of Measuring Case Depth

3. Terminology

3.1 Definitions of powder metallurgy (P/M) terms can be found in Terminology B 243. Additional descriptive information is available in the Related Material section of Volume 02.05 of the Annual Book of ASTM Standards.
3.2 Definitions of Terms Specific to This Standard:
   3.2.1 case —that portion of a part, extending inward from the surface that has a microindentation hardness, after hardening, equal to or greater than a specified hardness.
   3.2.2 effective case depth —the perpendicular distance from the surface of the hardened case to the furthest point where a microindentation hardness value equivalent to 50 HRC is maintained, unless otherwise specified.

4. Summary of Test Method

4.1 The powder metallurgy part is sectioned and the surface prepared for examination. Microindentation hardness measurements are taken at various depths below the part surface. The distance where the microindentation hardness falls below the equivalent of 50 HRC is defined as the effective case depth, unless otherwise specified.

5. Significance and Use

5.1 The engineering function of many P/M parts may require an exterior portion of the part to have a specified case depth and microindentation hardness. Measurement of effective case depth is used to determine the depth to which the microindentation hardness of the exterior portion of a part has been increased over that of the interior of the part.

6. Apparatus

6.1 Knoop or Vickers Hardness Indenters, using 100 gf (0.9807 N) loads are recommended following Test Method E 384. The type of hardness indenter and load used shall be agreed upon between customer and producer.
6.2 Calibrated Optical Instrument, Micrometer Stage, or other suitable means to measure the distance from the surface of the part to the center of the impression with a precision of 0.1 mm.
7. Test Specimen

7.1 Cut a test specimen from the P/M part, perpendicular to the hardened surface at a specified location, being careful to avoid any cutting or grinding procedure that would affect the original microindentation hardness.

7.2 Mounting of the test specimen is recommended for convenience in surface preparation, edge retention, and microindentation hardness measurement. Edge retention is important for proper depth measurement of the case.

7.3 Grind and polish the test specimen using methods recommended in Appendix X2 of Test Method B 933. The area to be traversed should be polished so the microindentation hardness impressions are unaffected, that is, the lighter the indenter load, the finer the finish necessary. Care should be taken to ensure that the true area fraction of porosity is revealed throughout the entire cross-section of the specimen. It is essential in surface preparation to remove all smeared metal and to identify pores clearly so that they may be avoided during testing.

7.4 The specimen should be lightly etched prior to microindentation hardness testing. Careful etching is necessary as heavy etching obscures features and interferes with the measurement of the diagonals of the indentation.

7.5 For heat treated steels, swabbing with or immersion in 2% nital for 4 to 7 s gives an appropriate structure.

8. Procedure

8.1 Measure microindentation hardness at a series of known intervals from the surface of the test specimen toward the interior. Take a minimum of three acceptable microindentation hardness measurements at each depth. Space the indentations so that adjacent tests do not interfere with each other. The minimum spacing between tests is illustrated in Fig. 1. Use a calibrated optical instrument, micrometer stage, or other suitable means to measure the distance from the surface of the part to the center of the impression.

8.2 Microindentation impressions should not be placed in soft regions such as copper or the center of nickel-rich regions. Randomly encountered upper bainite or fine pearlite in the martensite should not be excluded as a measurement location.

8.3 Plot microindentation hardness versus distance from the part surface (see Fig. 2). The effective case depth shall be the distance at which the microindentation hardness falls below the equivalent of 50 HRC unless a different value is specified (see Note 1). Plot definition will dictate the required number of readings, particularly in the critical region of effective case depth. The procedure described in Appendix X1 of Test Method B 933 shall be used for conversion to HRC.

NOTE 1—No compositional change occurs in induction hardened materials. The hardness of martensite is affected by the carbon content of the steel. Some lower-carbon steels will not reach the equivalent of 50 HRC when fully hardened. All concerned parties should agree upon the specified effective case depth hardness if other than 50 HRC.

NOTE 2—For routine quality control testing, where the effective case depth is reasonably well known, a somewhat simplified method of estimating effective case depth may be used. This method makes the assumption that the curve that represents microindentation hardness versus depth below the surface of the part may be regarded as a straight line in the region of the effective case hardened depth. Microindentation hardness may be measured at two depths from the surface selected, such that, on the basis of past experience, one will be less than the estimated effective case hardened depth and one will be greater. The two depths selected should lie at about equal distances from the estimated effective case hardened depth. At least five determinations of microindentation hardness shall be carried out at each of the selected depths below the part surface. On a plot of microindentation hardness versus depth from the surface, draw a straight line between the average microindentation hardness value at each of the two depths and read off the distance from the
surface of the part at which the specified microindentation hardness value is reached. This is the effective case depth.

*Note 3—*An alternative method may be used for routine quality control testing. Where a minimum effective case depth is specified, measure the microindentation hardness at a distance from the surface of the part that is equal to or greater than the specified minimum depth. At least five determinations of microindentation hardness shall be carried out at the desired location. If the average microindentation hardness at this depth is equal to or greater than the effective case depth hardness, the part meets the specified requirement.

8.4 Where a maximum effective case depth is specified, measure the microindentation hardness at a distance from the surface of the part that is less than the specified maximum depth in order to confirm that the part has been case hardened. At least five determinations of microindentation hardness shall be carried out at the desired location. This average microindentation hardness should be equal to or greater than that specified for the effective case hardness. If it is, measure the microindentation hardness at a distance from the surface of the part that is equal to the specified maximum depth. At least five determinations of microindentation hardness shall be carried out at the desired location. If the average microindentation hardness is less than the value specified for the effective case hardness, the maximum effective case depth specification is met. If it is greater than or equal to the specified value, the maximum effective case depth is exceeded.

9. **Report**

9.1 The report shall include:

9.1.1 The type of material and case measured (and when possible the type of process used to produce the case),

9.1.2 The location of the measurement,

9.1.3 The type of microindentation hardness indenter and the load used,

9.1.4 The load used in testing shall be expressed as the load in grams, for example, HV100 gf or HK 100 gf (preferred method), or the load in kilograms, for example, HV 0.1 or HK 0.1, and

9.1.5 The effective case depth to the nearest 0.1 mm and the microindentation hardness specified.

10. **Precision and Bias**

10.1 **Precision**—The precision of this test method is under review by Subcommittee B09.05 on Structural Parts.

10.2 **Bias**—No bias can be defined since there is no standard case-hardened material for which the effective case depth may be measured.

11. **Keywords**

11.1 case depth; effective case depth; powder metallurgy; P/M
Standard Test Method for Acid Resistance of Porcelain Enamels
(Citric Acid Spot Test)\(^1\)

This standard is issued under the fixed designation C 282; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (\(\epsilon\)) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers a procedure for evaluating porcelain enamels in their resistance to citric acid exposure at room temperature. No attempt is made to categorize porcelain enamels as to their acid-resistance or non-acid-resistance properties, since the requirements in the several branches of the industry differ.

1.2 The test method is applicable for ware of various shapes providing they contain a substantially flat area approximately 50 mm in diameter.

1.3 The test method is not applicable to finishes on chemical and hospital ware, which may come in contact with strong mineral acids, nor to cooking utensils, which may come in prolonged contact with hot acid solutions.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Summary of Test Method

2.1 The test method consists of a 15 min exposure of the test surface to a small pool of 10 % citric acid, and an evaluation of the effect in terms of the change in appearance and the "relative cleanability" of the surface resulting from the treatment.

3. Significance and Use

3.1 This test method is intended specifically for testing the porcelain enamel finish on stoves, refrigerators, table tops, sinks and other sanitary ware, laundry appliances, architectural units, etc., where the surface may come in contact with food acids at room temperature.

3.2 Citric acid has been chosen as the test medium because it is one of the most common of the food acids and will generally provide a measurable result in its action on porcelain enamel.

4. Apparatus

4.1 Dropper Bottle, or medicine dropper,
4.2 Watch Glass, 25 mm in diameter with fire polished edge,
4.3 Towel, soft cotton, and
4.4 Drafting Pencil, conventional graphite, degree 3B.

5. Reagents and Materials

5.1 Citric Acid Solution—Dissolve 10 g of anhydrous citric acid crystals (H\(_3\)C\(_6\)H\(_5\)O\(_7\)) in 100 mL of water. Solution shall be prepared not more than 48 h prior to use.

5.2 Cleaner Solution—Dissolve 10 g of trisodium phosphate (Na\(_3\)PO\(_4\)) in 1 L of tap water.

6. Sampling

6.1 The test specimens may be articles of commerce, pieces cut from articles of commerce, or laboratory specimens prepared especially for this test.

NOTE 1—Processing variables in the application and drying and firing operations materially affect the degree of attack by the acid on the surface of porcelain enamels. Sample specimens used for classification of acid resistance must be processed under identical conditions to the commercial ware they represent.

7. Procedure

7.1 Thoroughly wash area to be tested using a soft cotton towel moistened with a warm, 1 % solution of trisodium phosphate. Rinse in warm, running tap water, and dry with a soft towel by blotting. Store the specimen at a temperature of 26 ± 1°C (79 ± 2°F) for a time sufficient to bring it within this range prior to and during the test.

NOTE 2—If, when rinsing, the water gathers in drops on the surface, repeat washing treatment until water spreads evenly.

7.2 On articles of commerce, select areas that are horizontal or nearly horizontal in service. Place the specimen in a position such that a flat area at least 38.1 mm in diameter is horizontal. With the specimen and the citric acid solution at 26 ± 1°C (79 ± 2°F), place several drops of the solution on the test area to form a pool, and immediately cover with a clean watch glass in the inverted position. Use a quantity of solution that is just sufficient to fill the inverted watch glass except for a small air bubble (three to six drops are usually required, depending upon the dropper and the curvature of the watch glass). After 15 min
of treatment, remove the watch glass and immediately rinse the spot of solution from the surface. Dry the specimen with a dry, clean, soft cotton towel by blotting (not rubbing).

**Note 3**—The test surface must be thoroughly dry before grading. The presence of a slight film of water on the surface may change the rating of specimens near the borderline between classes.

**8. Grading**

8.1 Grade the test specimen within 2 h after exposure to the test solution using the procedure outlined in the diagram, Fig. 1. Rate each specimen with the appropriate classification grade, AA, A, B, C or D, as explained in Table 1. The criteria referred to in Fig. 1 and Table 1 are described as follows:

**8.1.1 Visual Examination**—View the specimen at various angles with respect to the light source and eye, in order to detect whether the “treated area” differs in any respect from the “protected area.” Use partially diffused daylight, supplemented if necessary by artificial light, the total intensity being approximately that available within a few feet of an outside window, but do not examine in direct sunlight. During observation, hold the specimen no nearer the light source (such as a window) than the minimum diameter of the source.

**Note 4**—The term “treated area” refers to that portion of the specimen which has been subjected to acid solution confined by the watch glass during treatment. The term “protected (untreated) area” refers to the area adjacent to the treated area.

**Note 5**—Some colored porcelain enamels exhibit a change in color as a result of the acid treatment. Since the test is designed primarily to evaluate durability, color changes as revealed by this test should be ignored except where otherwise specified.

8.1.2 Dry-Rubbing Test—Using the flat point of a degree 3B, conventional graphite drafting pencil held in a normal writing position and applied with firm pressure, draw two or more approximately parallel lines extending across the treated area. Starting with gentle pressure and then applying gradually increasing pressure, rub repeatedly across the lines with a dry, clean, soft cotton towel as illustrated in Fig. 2. If marks are completely removed from the treated area, the specimen shall be rated as Class AA. Otherwise, continue as directed.

**Note 6**—The pencil point should be prepared by holding a sharpened pencil in a normal writing position and rubbing on abrasive paper (1/0 Emery polishing or 400-grit silicon carbide or aluminum oxide abrasive paper) until the minimum diameter of the flat, circular cross section is half that of the full graphite core.

8.1.3 Blurring-Highlight Test—In a well-lighted location, hold the specimen so that the image of a small light source, such as a frosted lamp bulb, is observed as a highlight in the protected area, the line of vision being within 45° perpendicular to the surface (a desk lamp with an incandescent bulb is recommended for this purpose). Focus the eyes on the image of the light source, then slowly shift the specimen just sufficiently to bring this image into the treated area observing it as it passes across the boundary line between the two areas. Ignore any

<table>
<thead>
<tr>
<th>Classification</th>
<th>Requirements</th>
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</thead>
<tbody>
<tr>
<td>Class AA</td>
<td>No visible stain, and</td>
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<tr>
<td></td>
<td>Pass dry-rubbing test</td>
</tr>
<tr>
<td>Class A</td>
<td>Pass blurring-highlight test, and</td>
</tr>
<tr>
<td></td>
<td>Pass wet-rubbing test</td>
</tr>
<tr>
<td>Class B</td>
<td>Pass blurring-highlight test, and</td>
</tr>
<tr>
<td></td>
<td>Fail wet-rubbing test</td>
</tr>
<tr>
<td>Class C</td>
<td>Fail blurring-highlight test, and</td>
</tr>
<tr>
<td></td>
<td>Pass disappearing-highlight test</td>
</tr>
<tr>
<td>Class D</td>
<td>Fail disappearing-highlight test</td>
</tr>
</tbody>
</table>

**FIG. 1 Flow Sheet of Test Procedure for Classification of Treated Specimens**

**FIG. 2 Method of Applying and Rubbing Pencil Marks on the Treated Area**
color difference in the enamel due to staining. If a definite blurring of the image is observed as it passes from the protected to the treated area, the specimen shall be rated as Class C or Class D. Proceed to 8.1.5 to determine final classification. If a blurring image is not observed, the specimen shall be rated as Class A or Class B. Proceed to 8.1.4 to determine final classification.

8.1.4 Wet-Rubbing Test—Using the procedure specified in 8.1.2 mark the treated area and rub the marks with a clean, soft cotton towel which has been dipped in water and twisted to remove any excess. For this test, make new marks in a location other than that used for the dry-rubbing test. Do not use soap, abrasive, or similar cleaning material. If the marks are completely removed from treated area, the specimen shall be rated as Class A. Otherwise it shall be rated as Class B.

8.1.5 Disappearing-Highlight Test—This test is similar in all respects to the blurring-highlight test specified in 8.1.3, except that the criterion in this case is the complete disappearance of the highlight in the treated area. If the highlight is visible in the treated area, the specimen shall be rated as Class C. If the highlight disappears in the treated area, the specimen shall be rated as Class D.

9. Report

9.1 Report the assigned grade for each specimen. If the citric acid treatment is performed at temperatures outside the stated tolerances, this variation should be reported.

10. Reproducibility

10.1 Since variations may occur during enameling which affect the acid resistance, several specimens should be treated to determine the class of commercial ware.

10.2 Ratings of individual specimens shall be considered as due to actual differences in the acid resistance of the specimens, not to differences in the test procedure.

11. Precision and Bias

11.1 The precision and bias of this test method is being established.
Standard Test Methods for
Resistance of Porcelain Enameled Utensils to Boiling
Acid1

This standard is issued under the fixed designation C 283; the number immediately following the designation indicates the year of
original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A
superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope
1.1 These test methods cover the determination of the
resistance of porcelain enamel coatings used on utensils to
attack by boiling acid.
1.2 This standard does not purport to address all of the
safety concerns, if any, associated with its use. It is the
responsibility of the user of this standard to establish appro-
priate safety and health practices and determine the applica-
bility of regulatory limitations prior to use.

2. Significance and Use
2.1 Test Method A provides the producers of porcelain
enameled utensils with a quality control method of testing for
resistance to boiling acid for parts randomly selected from the
production line.
2.2 Test Method B provides the supplier of porcelain
enamel raw materials with a laboratory method for testing the
resistance of different coatings (intended for use on utensils) to
boiling acid.

TEST METHOD A

3. Apparatus
3.1 Hot Plate, capable of maintaining over its entire surface
a uniform temperature that will keep the test solution at a
rolling boil (see section 7.5). The heating element should
occupy an area at least 8 in. (203 mm) in diameter.
3.2 Chemical Glassware—Beakers, a desiccator, and a
500-mL graduated cylinder.
3.3 Drier, capable of maintaining a temperature of at least
220°F (104°C).
3.4 Boiling Acid Resistance Apparatus, as shown in Fig. 1.
Three sets will be required.
3.5 Cutting Equipment, suitable for cutting a 3 ¼-in. (82-
mm) diameter plate from the bottom of a porcelain-enameled

1 These test methods are under the jurisdiction of ASTM Committee B08 on
Metallic and Inorganic Coatings and are the direct responsibility of Subcommittee
B08.12 on Materials for Porcelain Enamel and Ceramic-Metal Systems.
Test Method A is based on the boiling acid resistance test developed by the
Enameled Utensil Manufacturers’ Council; see Section 16 of Commercial Standard
CS100-47, Porcelain Enameled Steel Utensils, United States Department of Com-
merce.

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test specimen. This serves as a seal between the test specimen and the heat-resistant glass tube, which shall be placed over it. Place another rubber jar ring on top of the glass tube, set the cover-plate on it, and clamp in place by tightening the wing nuts. Tighten the wing nuts until a seal is obtained between the glass tube and the test specimen. This can be determined by inspecting the seal ring, when the nuts are tight enough, the outside edges of the rubber jar rings will lift from the test specimen. Use the bottom jar rings only twice (once on each side); then discard for new ones.

7.5 Bring approximately 500 mL of the citric acid solution to boiling, pour 150 mL of the solution into each glass tube, and set the glass condensing tubes in position. Promptly center the three test units on the hot plate, as shown in Fig. 3, and leave the test units in place for a period of \(2\frac{1}{2}\) h. After the solutions begin to boil, decrease the wattage input so that the solutions remain at a rolling boil for the remainder of the test period.

7.6 At the end of the test period, remove the units and condenser tubes from the hot plate and pour out the solutions, then rinse the insides of the tubes and the test specimens with distilled water, and dismantle the apparatus. Remove the test specimens, rinse again with distilled water scrub the test specimens gently with a nylon brush (10 to 12 strokes) to remove loose residue in the attack area, and dry in the drier at 220°F (104°C) for 10 min. Place the specimens in a desiccator and cool to room temperature.

7.7 After the test specimens are cool, again weigh them separately on the analytical balance.

8. Calculations and Report

8.1 The difference between the initial and the final weight of each specimen, divided by four, gives the loss in weight per square inch of exposed area (wetted surface) (Note 2). Take the average loss per square inch for three specimens from three identical utensils for the boiling-acid resistance of the enamel.

Note 2—Wetted surface area is that surface which is attacked by the test solution.

8.2 If, however, any one of the three results does not agree within \(\pm 10\%\) of the average, another set of three plates from utensils identical with those of the first set shall be tested. The individual values of loss in weight per square inch for the second set of plates shall be averaged with the individual values of the first set, any individual value not within \(\pm 10\%\) of the average for the six plates shall be discarded, and the average of the remaining values shall be taken as the boiling-acid resistance of the enamel.

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**FIG. 1 Boiling-Acid Resistance Apparatus**

<table>
<thead>
<tr>
<th>in.</th>
<th>mm</th>
<th>in.</th>
<th>mm</th>
<th>in.</th>
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<th>mm</th>
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<td>1/4</td>
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<td>57</td>
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<td>82</td>
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<tr>
<td>1/4</td>
<td>6.4</td>
<td>2/4</td>
<td>57</td>
<td>1/2</td>
<td>3/4</td>
<td>86</td>
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</tr>
<tr>
<td>3/8</td>
<td>9.5</td>
<td>2.31</td>
<td>59</td>
<td>5/8</td>
<td>4</td>
<td>102</td>
<td>5/8</td>
</tr>
</tbody>
</table>

(a) Assembly

(b) Details
TEST METHOD B

9. Apparatus
9.1 Same as Section 3.

10. Reagent
10.1 See 4.1.

11. Test Specimens
11.1 The test specimens shall consist of three plates $\frac{3}{4}$ in. (82 mm) in diameter, cut from a sheet of raw Utensil Steel stock.

12. Preparation of Test Specimens
12.1 After the test specimens have been cut from the steel sheet, the edges are rounded with a file to insure uniform coverage.

12.2 The test specimens are then metal prepared by the test users preferred method (that is, cleaning or nickel flash pickling).

12.3 One hole $\frac{1}{4}$ in. (3.2 mm) in diameter is punched in each test specimen, the center of which is $\frac{1}{4}$ in. (6.4 mm) from the outer edge of the specimen plate. This hole is necessary for hanging the specimen during firing of the coating being tested.

12.4 The enamel coating to be tested may be applied to the three test plates by dipping or spraying at 30 gms per sq. ft. dry (323 gms per sq. meter). Spraying is the preferred method for minimizing edge beading which may occur during dip application methods. Glass beads may chip off during the test leading to greater weight losses for that specimen.

12.5 The three test specimens (after drying if a wet application method is used) are fired at the optimum time and temperature as established in the test users facility.

12.6 Thoroughly wash the test specimens with soap and water to remove any grease picked up in handling. Rinse with distilled water. Place the specimens in a drier at 220°F (104°C). After 10 min remove the test specimens and place them in a desiccator containing calcium chloride.

13. Procedure
13.1 See 7.1-7.7.

14. Calculation and Report
14.1 See 8.1 and 8.2.

15. Precision and Bias
15.1 The precision and bias of these test methods are being established.
FIG. 3 Position of Test Units of Boiling-Acid Resistance Apparatus on Hot Plate

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Standard Test Methods for
Sieve Analysis of Wet-Milled and Dry-Milled Porcelain
Enamel

This standard is issued under the fixed designation C 285; the number immediately following the designation indicates the year of
original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A
superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the determination of the
fineness of frit in wet- or dry-milled porcelain enamels and
other ceramic coatings for metals by means of the No. 200
(75-µm) or No. 325 (45-µm) sieve.

1.2 The two methods appear as follows:

Sections

<table>
<thead>
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</thead>
<tbody>
<tr>
<td>Method B—Routine Method</td>
<td>10 to 14</td>
</tr>
</tbody>
</table>

1.3 Method A is intended for use where a referee method of
higher accuracy is required, while Method B is intended to
meet the needs of normal enamel plant production control
operations where a rapid, simplified method of sieve testing is
required. The accuracy of the simplified method has proved to
be entirely adequate for this use. The simplified test, however,
is not recommended where high accuracy is required.

1.4 This standard does not purport to address all of the
safety concerns, if any, associated with its use. It is the
responsibility of the user of this standard to establish appro-
priate safety and health practices and determine the applica-
bility of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

E 11 Specification for Wire-Cloth Sieves for Testing Pur-
oposes

3. Significance and Use

3.1 The fineness of the frit has a direct bearing on many of
its properties, such as fusibility, tearing, gloss, opacity, suspen-
sion in the slip, and ease of spraying.

METHOD A—REFEREE METHOD

4. Apparatus

4.1 Balance—The balance or scale shall be of at least 500-g
capacity, and accurate to 0.1 g.

4.2 Sieves—The sieves shall conform to Specification E 11. They shall include the No. 40 (425-µm) sieve and also the No.
200 (75-µm) or the No. 325 (45-µm) sieve (Note 1), or both. A
No. 325 sieve shall be used when the fineness is such that, from
a sample containing 100 g of dry solids, less than 2 g is
retained on a No. 200 sieve. An 8-in. (203-mm) full-height
sieve is recommended. This height is preferred because there is
less tendency to flood or splash, and also because it fits
commercial automatic tapping and shaking machines. All
sieves used for testing shall be standardized initially and after
every 50 tests against a reference sieve tested by the National
Bureau of Standards and bearing its precision seal. The
correction for the sieve used in this test shall be determined by
sieving tests made in conformity with the procedure of this test
method. Identical samples shall be sieved through the reference
sieve and the test sieve. Test materials shall be chosen so that
5 to 10 percent of the material will be retained on the reference
sieve. The difference between the percentage residue on the
reference sieve and that on the test sieve is the amount of
correction which shall be algebraically added to, or subtracted
from, the correction for the reference sieve to obtain the final
correction (Note 2). The No. 40 sieve need not be calibrated.

NOTE 1—Tyler Standard Sieves of 35, 200, and 325 mesh correspond,
respectively, to ASTM sieves No. 40, 200, and 325 (U.S. Standard Sieve
Series numbers).

NOTE 2—for example, when comparing the reference sieve with a test
sieve, should 8.5 g be retained on the reference sieve and 7.5 g on the test
sieve, the total correction for the test sieve would then be 8.5 – 7.5,
or + 1.0.

4.3 Dryer—A suitable means for drying the sieves and slip
sample, without exceeding a temperature of 250 °F (122 °C),
shall be provided. No dryer is needed for sieve tests of
dry-milled enamel.

4.4 Mechanically Operated Sieve Shaker—The mechanical
shaking device shall be such as to produce a lateral and vertical
motion of the sieve, accompanied by a jarring action so as to
keep the sample moving continuously over the surface of the
sieve. If a machine shaker is to be used, the thoroughness of
sieving shall be tested by comparing and calibrating with the
hand method of sieving, as described in Section 6.
5. Sample

5.1 Wet-Milled Enamel—Select a sample of slip representative of the material to be tested. Protect samples from evaporation. Determine the water content of the slip by drying a representative 100-g sample of slip to constant weight at a temperature not exceeding 250 °F (122 °C). Pass the sample through a No. 40 sieve before weighing, and discard the material retained on the sieve. Sample the slip after stirring by weighing out to the nearest 0.1 g, a quantity calculated to contain 100 g of dry solids.

5.2 Dry-Milled Enamel—Select a sample representative of the material to be tested and containing 100 g of dry solids, weighed to the nearest 0.1 g.

6. Procedure for Wet-Milled Enamel

6.1 Wet Sieving—Transfer the weighed sample to the No. 200 or No. 325 sieve. Wash the sample through the sieve with a gentle flow of water from a rubber hose until the water passing through the sieve appears clear and free of cloudiness. This usually requires 2 min. Exercise care to prevent any loss of sample because of splashing or overflowing. Dry the sieve with its residue until the residue easily moves about as a dry powder when the sieve is shaken. Complete the sieving and weighing operation in accordance with 6.2 or 6.3. Make tests in duplicate.

6.2 Hand Sieving—Hold the sieve, with pan and cover attached, in one hand at an angle of about 20 deg from the horizontal. Move the sieve up and down in the plane of inclination at a rate of about 150 times per minute, and strike against the palm of the other hand at the top of each stroke. After every 25 strokes, turn the sieve about one sixth of a revolution in the same direction. Continue the operation until not more than 0.05 g passes through the sieve in 1 min of continuous sieving. Weigh the portion of the sample retained on the sieve to the nearest 0.1 g.

6.3 Machine Sieving—If a mechanically operated sieve shaker is used, vary the time during which the sieve (with pan and cover attached) and the sample are shaken, and note the length of time necessary to operate the sieve shaker in order to get the same result as that obtained with hand sieving. Calibrate the machine-sieving operation in terms of hand sieving.

7. Procedure for Dry-Milled Enamel

7.1 Transfer the sample quantitatively to the No. 200 or 325 sieve. Complete the sieving and weighing operation in accordance with 5.2 or 5.3. Make tests in duplicate.

8. Calculation and Report

8.1 Using the average of duplicate runs, report the fineness of the frit in percentage by mass (to the nearest 0.1 %) of the dry solids content of the sample retained on the No. 200 or 325 sieve. The weight in grams is equivalent to weight percentage.

9. Precision and Bias

9.1 Precision—It is generally accepted within the porcelain enamel industry that duplicate tests run by the same operator in the same laboratory should show a precision of ± 0.5 or less. Failure to duplicate determinations within this limit indicates the necessity for repetition of the test. The precision of duplicate tests is negatively influenced by variations in operator technique, sieve binding and wear, and, when it is used, the condition of mechanical sieving equipment. It is also influenced by changing conditions in the test sample which occur over time and which result in particle agglomeration due to chemical reactions.

9.2 Bias—No justifiable statement on bias can be made since the true value of sieve analysis cannot be established by an acceptable standard sample.

METHOD B—ROUTINE METHOD

10. Apparatus

10.1 Balance—The balance or scale shall be at least 200-g capacity, and accurate to 0.1 g.

10.2 Sieves—The sieves shall conform to Specification E 11 and shall include the No. 40 (425-µm) and the No. 200 (75-µm) sieves. An 8-in. (203-mm) full-height sieve is recommended. This height is preferred because there is less tendency to flood or splash, and also because it fits commercial automatic tapping and shaking machines. A sieve properly cared for will have 500 tests. It should, however, be compared from time to time with a master standard or reference sieve. It is recommended that an extra sieve be purchased and preserved as a master standard against which all sieves in use or subsequently purchased can be standardized.

NOTE 3—Tyler Standard Sieves of 35 and 200 mesh correspond, respectively, to ASTM sieves No. 40 and 200 (U.S. Standard Sieve Series numbers).

10.3 Container—A container suitable for weighing a 100-g sample.

10.4 Dryer—See 4.3

10.5 Mechanically Operated Sieve Shaker—See 4.4.

11. Sample

11.1 Select a sample of slip from the mill before unloading and pass it through a No. 40 sieve before weighing. Discard the material retained on the sieve. The sample shall consist of 100 g of slip for wet-milled porcelain enamel or 100 g of ground frit for dry-milled porcelain enamel.

12. Procedure

12.1 Wet-Milled Porcelain Enamel—Transfer the 100-g sample of slip to the No. 200 sieve. Wash the sample through the sieve with a stream of gently running water, care being exercised to prevent splashing or overflowing of the sieve. Continue washing until the water passing through the sieve appears clear and free from cloudiness. This usually requires at least 2 min. Dry the sieve with its residue until the residue easily moves about as a dry powder when the sieve is shaken (Note 4). Shake the sieve in an automatic shaking and tapping machine, or by hand, until no further material passes through the meshes. This usually requires from 5 to 7 min. Transfer the residue remaining on the sieve to the balance and weigh to the nearest 0.1 g.

NOTE 4—It is recommended that, at this point, the residue be transferred to another sieve for shaking. The use of a second sieve minimizes
the error caused by the clogging when both washing and shaking are accomplished with the same sieve.

12.2 Dry-Milled Porcelain Enamel—Place the 100-g sample of frit powder in a No. 200 sieve and shake in an automatic shaking and tapping machine, or by hand, until no further material passes through the meshes (Note 5). This usually requires from 5 to 7 min. Transfer the residue remaining on the sieve to the balance and weigh to the nearest 0.1 g.

Note 5—The end point is usually taken as the time at which not more than 0.1 g of material passes through the sieve with 1 min shaking. A little experience will indicate to the operator when the shaking operation is complete.

13. Calculation and Report

13.1 Report the weight in grams of the residue on the screen as equivalent to weight percentage of the original slip, or frit powder, specimen.

14. Precision and Bias

14.1 Precision—It is generally accepted within the porcelain enamel industry that duplicate tests run by the same operator in the same laboratory should show a precision of ± 0.5 or less. Failure to duplicate determinations within this limit indicates the necessity for repetition of the test. The precision of duplicate tests is negatively influenced by variations in operator technique, sieve binding and wear, and, when it is used, the condition of mechanical sieving equipment. It is also influenced by changing conditions in the test sample which occur over time and which result in particle agglomeration due to chemical reactions.

14.2 Bias—No justifiable statement on bias can be made since the true value of sieve analysis cannot be established by an acceptable standard sample.
Standard Terminology Relating to Porcelain Enamel and Ceramic-Metal Systems

This standard is issued under the fixed designation C 286; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These definitions pertain to the terminology used in the porcelain enamel and ceramic-coated metal industries.

1.2 Words adequately defined in standard dictionaries are not included. Included are words that are peculiar to these industries.

1.3 Hyphenated words, double words, or phrases are listed alphabetically under the first word; additional important words are cross-referenced.

1.4 When a word or phrase, listed as a synonym, is not separately defined, the defined word or phrase is the accepted or preferred form.

2. Referenced Documents

2.1 ASTM Standards:

- A 424 Specification for Steel, Sheet, for Porcelain Enameling
- A 919 Terminology Relating to Heat Treatment of Metals
- C 282 Test Method for Acid Resistance of Porcelain Enamels (Citric Acid Spot Test)
- C 283 Test Method for Resistance of Porcelain Enameled Utensils to Boiling Acid
- C 285 Test Methods for Sieve Analysis of Wet-Milled and Dry-Milled Porcelain Enamel
- C 313 Test Method for Adherence of Porcelain Enamel and Ceramic Coatings to Sheet Metal
- C 314 Test Method for Flatness of Porcelain Enameled Panels
- C 346 Test Method for 45-Deg Specular Gloss of Ceramic Materials
- C 347 Method of Test for Reflectivity and Coefficient of Scatter of White Porcelain Enamels
- C 374 Test Methods for Fusion Flow of Porcelain Enamel Frits (Flow-Button Methods)
- C 448 Test Methods for Abrasion Resistance of Porcelain Enamels
- C 614 Test Method for Alkali Resistance of Porcelain Enamels
- C 633 Test Method for Adhesion or Cohesive Strength of Flame-Sprayed Coatings
- C 743 Test Method for Continuity of Porcelain Enamel Coatings
- C 756 Test Method for Cleanability of Surface Finishes

3. Terminology

abrasion resistance—the degree to which a porcelain enamel will resist attack by abrasive materials.

Note 1—See Test Methods C 448.

acid annealing—an annealing process in which ferrous metal shapes are coated with acid before and in conjunction with the annealing.

cyst acid resistance—the degree to which a porcelain enamel will resist attack by acids.

Note 2—See Test Method C 283 and Test Method C 282.

adherence—(1) the degree of adhesion of a porcelain enamel or other ceramic coating to a metal substrate.

Note 3—See Test Method C 313.

(2) Stress necessary to cause separation of one material from another at their interface.

Note 4—See Test Method C 633.

aging—the storing of porcelain enamel slips or powders before use. The change occurring in slips or powders with the lapse of time.

air atomizing—air used to atomize powder and to control powder/air mix and powder cloud density.

air fluidizer—air used to impart fluid-like properties to powder via a fluid bed.

alkali resistance—for porcelain enamels, the degree to which...
a porcelain enamel will resist attack by aqueous alkaline solutions.

Note 5—See Test Method C 614.

Alligator hide—a defect characterized by an extreme roughness of the porcelain enamel surface: a severe case of orange peel.

Aluminum enamel—a porcelain enamel specifically designed for application to aluminum.

Annealing—see Terminology A 919.

Annealing acid—see acid annealing and annealing.

Anti-scale compound—a preparation that is applied to burning tools to protect them from scaling in service.

Back emission—the electrical breakdown of air due to excessive charge build-up in the porcelain enamel powder film during powder application. This is due to the self-limiting characteristic of electrostatic powders.

Back ionization—see back emission.

Ball mill—in porcelain enamels, a dense, ceramic-lined rotating cylinder in which ceramic materials are wet or dry ground, generally using pebbles or porcelain balls as grinding media.

Base coat—for two coat-one fire application, the thin layer of bonding frit applied first and used to promote adherence after firing to the metal substrate.

Base metal—the metal to which porcelain enamel is applied. Basis metal—see base metal.

Basket, pickle—see pickle basket and pickling.

Batch smelter—any smelter that operates as a periodic unit, being charged, fired, and discharged according to a predetermined cycle.

Beading—(1) the application of porcelain enamel, usually of a contrasting color, to the edge or rim of porcelain enameled articles.

(2) Removal of excess slip from the edge of dipped ware.

(3) In dry processing enameling, a bead of porcelain enamel along the edge of ware.

Beading enamel—any of the special porcelain enamels used for beading.

Bisque—a coating of wet-process porcelain enamel that has been dried, but not fired.

Blackboard enamel—see chalkboard enamel.

Black edging—a black porcelain enamel applied over the ground coat and exposed in specified areas by brushing the cover coat bisque prior to firing (see also edging).

Black speck—a defect that appears in the fired cover coat as a small dark spot.

Blank—the piece cut from metal sheet that is to be used in forming the finished article.

Blemish—in dry process enameling, an insignificant imperfection in the porcelain enamel surface.

Blister—a defect caused by gas evolution consisting of a bubble that forms during fusion and remains when the porcelain enamel solidifies.

Blow-off resistance—the degree to which a deposited layer of powder resists being blown off by a standard jet of air.

Blue enamel—(1) in dry-process porcelain enameling, an area of enamel coating so thin that it appears blue in color.

(2) In wet-process enameling, a cover coat applied too thin to hide the substrate.

Boiling—a defect visible in the fired porcelain enamel caused by gas evolution which results in the formation of blisters, pinholes, black specks, dimples, or spongy surface.

Bolt-hole brush—a special round brush used to remove porcelain enamel bisque from in and around small openings in the ware.

Bond—see adherence.

Box furnace—a furnace in which, periodically, a load of ware is introduced; fired, and removed.

Break out—in dry process enameling, a defect characterized by an area of blisters with well defined boundaries.

Bright annealing—see Terminology A 919.

Brush—to remove bisque in a definite pattern by means of a brush.

Brush, bolt-hole—see bolt-hole brush.

Brushing—see brush.

Bubble structure—size and spatial distribution of voids within the fired porcelain enamel.

Buck—a special support for ware during the firing of porcelain enamel on heavy ware.

Burning—see firing.

Burning bars, points, or tools—equipment used to suspend or support ware during the firing operations.

Burning tool mark—a defect in the porcelain enamel appearing on the surface opposite to the point of contact with the supporting burning tool.

Button test—a test designed to determine relative fusibility of porcelain enamel frit or powder and so called because the completed specimens resemble buttons.

Note: 6—See flow button and Test Methods C 374.

Cast iron enamel—a porcelain enamel specifically designed for application to cast iron.

Ceramic coating—an inorganic, essentially nonmetallic coating, on metal.

Ceramic colorant—see color oxide.

Ceramic ink—an ink containing a ceramic pigment that develops its color on firing. Also known as stamping, screening, or printing ink.

Ceramic-metal coating—a mixture of one or more ceramic materials in combination with a metallic phase applied to a metallic substrate which may or may not require heat treatment prior to service. This term may also be used for coatings applied to nonmetallic substrates, for example, graphite.

Cermet coating—see ceramic-metal coating.

Chalkboard enamel—a special type of mat porcelain enamel used to provide a writing surface for chalk.

Chalky or chalked—the condition of a porcelain enameled surface that has lost its natural gloss and become powdery.

Charge decay—loss of charge on the deposited powder due to electrical leakage.

Charge decay rate—loss of charge per unit of time.

Charge retention—the ability of an electrically charged layer to retain its initial charge.
charge to mass ratio—ratio of the charge on a powder expressed in coulombs to the mass of the powder expressed in kilograms.

chipping—fracturing and breaking away of fragments of a porcelain enameled surface.

cleanability—the relative ease with which soils or stains can be removed from a material.

Note 7—See Test Method C 756.

cleaner—a solution, usually alkaline, used to remove oil, grease, drawing compounds, and loose dirt from metal as a step in preparing the surface for porcelain enameling.

clear frit—a frit that remains essentially transparent or nonopaque when processed into a porcelain enamel.

coating—see ceramic coating and ceramic-metal coating.

coefficient of scatter—the rate of increase of reflectance with thickness at infinitesimal thickness of porcelain enamel over an ideally black backing.

Note 8—See Test Method C 347.

cold-rolled steel—a low-carbon, cold-reduced and annealed sheet steel.

color oxide—a material used to impart color to a porcelain enamel.

colored frit—a frit containing a colorant in order to produce a strong color in the porcelain enamel.

comb-rack—(1) a burning tool shaped like a comb used for supporting ware during firing.

(2) A comb-like tool for supporting ware during the metal pickling operation.

comeback—the time required for a box furnace to return to temperature after the introduction of a load of ware.

cone-screen test—a method for testing fineness of enamel with a cone-shaped sieve. (see also screen test)

consistency—the properties of a slip that control its draining, flowing, and spraying behavior.

continuity of coating—the degree to which a porcelain enamel or ceramic coating is free of defects, such as bare spots, boiling, blisters or copperheads, that could reduce its protective properties.

Note 9—See Test Method C 743.

continuous cleaning (coating)—a term describing a type of porcelain enamel designed to provide the continuous removal, at normal use temperatures, of food soils accumulated on the interior surfaces of ovens.

continuous furnace—a furnace into which ware is fed continuously and through which it progresses during firing.

continuous smelter—a type of smelter into which the raw mix is fed continuously and from which the molten product is discharged continuously.

contrast ratio—the ratio of the reflectance of a coating over black backing to its reflectance over a backing of reflectance of 0.80 (80 percent).

Note 10—See Test Method C 347.

cooling zone—that part of the continuous furnace in which the ware is allowed to cool after firing.

copper enamel—a porcelain enamel specifically designed for application to copper.

copperhead—a defect occurring in sheet metal ground coat that appears as a small freckle or pimple-like spot, reddish brown in color.

cover coat—a porcelain enamel finish applied and fused over a ground coat or direct to the metal substrate.

covering power—the degree to which a porcelain enamel coating obscures the underlying surface.

cracking—a defect in the bisque consisting of fractures or separations.

cracked—a mottled textural effect in a wet process porcelain enamel resembling a wrinkled surface.

crawling—a defect in the porcelain enamel appearing as agglomerates or irregularly shaped islands.

craze, crazing—a defect appearing as one or more fine cracks in the porcelain enamel.

crinkled—a textural effect in a porcelain enamel surface having the appearance of fine wrinkles or ridges.

crossbend test—a test in which fired or bisque porcelain enamel panels are progressively distorted by bending to determine the resistance of the coating to cracking.

cup gun—a spray gun with a fluid container as an integral part.

cupping—the pouring of slip over areas of a part during draining to produce uniform application.

curling—a defect similar to crawling.

curtains—a defect in sheet steel ground coatings characterized by a draped pattern of darkened areas that are sometimes blistered. May also appear in cover coats applied over the ground coat or direct-on.

decarburized enameling steel—a special type of steel sheet of extremely low carbon content, suitable for porcelain enamel cover coat application direct to the metal (Type I of Specification A 424).

decarburized steel—see decarburized enameling steel.

decking—the multiple layer loading of ware for firing.

def-enameling—the removal of porcelain enamel from the base metal.

deflocculating—the thinning of the consistency of a slip by adding a suitable electrolyte.

delayed fishscaling—a fishscaling defect that occurs after the final porcelain enamel processing (see also fishscaling).

devitrification—a surface defect manifested by loss of gloss as a result of crystallization.

dimple—a shallow depression in the porcelain enamel, sometimes a defect.

dipping—the process of coating a metal shape by immersion in slip, removal, and draining. In dry process enameling, the method of coating by immersing the heated metal shape for a short time in powdered frit.

dipping weight—see pick-up.

direct fire—a method of maturing porcelain enamel wherein the products of combustion come in contact with the ware.

direct-on—see cover coat.

double draining—a defect evidenced by flowing of the slip on the ware, which occurs after it appears that draining has been completed.
double-face ware—ware that has a finish coat on both surfaces.

draining—the part of the dipping or flowcoating process in which the excess slip flows from suitably positioned ware.

drain line—a nonuniform thickness of coating appearing as a line or streak in dipped or flow-coated ware.

drain time—time required for porcelain enamel slip applied by dipping, slushing, or flow coating to complete movement across the surfaces of a coated part.

dredge, dredging—in dry process enameling, (1) the application of dry, powdered frit to hot ware by sifting.

(2) The sieve used to apply powdered porcelain enamel frit to the ware.

drying crack—a defect characterized by a fissure in the bisque.

dry milling—the grinding of porcelain enamel materials without a liquid vehicle.

dry process enameling—a porcelain enameling process in which the metal article is heated to a temperature above the maturing temperature of the coating (usually 1600 to 1750°F, (approximately 870 to 955°C)), the coating materials applied to the hot metal as a dry powder, and fired.

dry spray—a defect confined to sprayed ware manifesting itself in the fired porcelain enamel as a rough, sandy texture.

dry weight—the weight per unit area of the bisque.

dust coat—a relatively thin, sprayed coating of slip.

dusting—(1) In dry-process enameling, see dredging.

(2) A spraying defect characterized by a piling up of almost dry slip in confined areas.

(3) The removal of extraneous material from the bisque before firing.

(4) See dry spray.

edging—(1) the process of removing bisque from the edge of a piece of ware to expose the underlying porcelain enamel.

(2) The spraying of special slip onto the edge of the ware. edging brush—a stiff-bristled brush with metal guide, used to remove bisque from edges of ware before the firing operation.

eggshell or eggshelling—the texture of a fired ceramic coating similar in appearance to the surface of an eggshell. In porcelain enamel, usually a defect.

ejector air—air used to convey powder from pump to the part being coated.

electrophoretic deposition—the process of depositing material on a workpiece from a porcelain enamel slip suspension due to the movement of particles under the influence of an impressed direct current voltage.

electrostatic powder porcelain enamel—a mixture comprised of frit and additives ground and/or blended together to form a powder suitable for dry electrostatic application.

electrostatic retention—the tenacity with which a charged, electrostatically deposited powder porcelain coating adheres to the work piece before it is fired.

enamel—see porcelain enamel.

enamel, aluminum—see aluminum enamel.

enamel, beading—see beading enamel.

enamel, blackboard—see chalkboard enamel.

enamel, cast iron—see cast iron enamel.

enamel, chalkboard—see chalkboard enamel.

enamel, copper—see copper enamel.

enamel, jewelers’—see jewelers’ enamel.

enamel, reclaim—see reclaim.

enamel scrapings—see scrapings.

enameling iron—a very low-carbon, low-metalloid, cold-rolled sheet steel, produced specifically for use as a base metal for porcelain enamel.

etched—an altered surface texture resulting from chemical attack.

fall-off—tendency of an electrostatically deposited powder to fall off the work piece during normal processing.

filter—see plugging compound.

film strength—the relative resistance of the bisque to mechanical damage.

fineness of enamel—a measurement of the degree to which a frit has been milled in wet or dry form, usually expressed in grams residue retained on a certain mesh screen from a 50-cm³ or a 100-g sample.

fire marks—a defect characterized by tiny indentations similar in appearance to shallow pinholes.

firing—the controlled heat treatment of ceramic ware in a kiln or furnace to develop the desired final properties.

firing range—the time-temperature interval in which a porcelain enamel or ceramic coating is satisfactorily matured.

firing temperature—the degree of sensible heat attained by the ware during the maturing of the coating.

firing time—the period during which the ware remains in the firing zone of the furnace to mature the coating.

firing zone—that portion of the furnace, usually a continuous furnace, through which the ware passes and that remains at or near the firing temperature of the coating.

first point of no break—the amount (weight-mass) of porcelain enamel slip retained when it stops sliding off an enameled pick-up panel and is observed to drain smoothly from the panel without showing a wavy pattern on the wet surface (known also as “yield point”).

fishscaling—a defect appearing as small half-moon shaped fractures somewhat resembling the scales of a fish.

fishscaling, delayed—see delayed fishscaling.

flaw—in dry process enameling, a defect of the ware that is cause for rejection.

flocculating—the thickening of the consistency of a slip by adding a suitable electrolyte.

flow-button—the pellet of frit used in the Fusion Flow Test.

flow coating—the process of coating a metal shape by causing the slip to flow over its surface and allowing it to drain.

flux—a substance that promotes fusion in a given ceramic mixture.

fork—a piece of metal equipment used during the firing operation for placing ware in, and removing it from a box furnace.
frit, clear—see clear frit.

frit, colored—see colored frit.

frit, porcelain enamel—the small friable particles produced by quenching a molten glassy material (see also clear frit and colored frit).

fritting—the rapid chilling of the molten glassy material to produce frit.

furnace, box—see box furnace.

furnace, continuous—see continuous furnace.

fusion flow—the relative flow of various glasses or frits in the molten state.

NOTE 12—See Test Methods C 374.

fusion test, button—see button test.

fuzzy texture—a defect characterized by a myriad of minute bubbles, broken bubbles, and dimples in the porcelain enamel surface.

gassing—(1) the formation of gas bubbles due to bacterial contamination in the milled porcelain enamel slip.

(2) See boiling.

gassy surface—a defect characterized by poor gloss and fuzzy surface texture.

glass—a term sometimes used for porcelain enamel or frit.

glass-coated steel, glass-lined steel, glassed steel—designations generally applied to a class of porcelain enamels that have high resistance to chemical attack at elevated temperatures and pressures.

glass eye—a defect consisting of a large unbroken blister.

gloss—the shine or luster of a porcelain enamel.

NOTE 13—See Test Method C 346.

graining—a process for producing a decorative finish by transferring a pattern to the porcelain enamel surface by means of rolls.

graining paste—a mixture of color oxides, fluxes, and oils.

graining roll—a specialized type of roll used for transferring the grain pattern to the porcelain enamel.

graniteware—a one-coat porcelain enameled article with a mottled pattern produced by controlled corrosion of the metal base prior to firing.

ground coat—(1) a porcelain enamel applied directly to the base metal to function as an intermediate layer between the metal and the cover coat.

(2) on sheet steel, a porcelain enamel coating containing adherence-promoting agents which may be used either as an intermediate layer between the metal and the cover coat or as a single coat over the base metal.

ground-coat boiling—see boiling.

hairline or hairlining—a defect manifested in finished ware as a line or system of lines in a strain pattern, having the appearance of cracks healed by fusion.

hanging rack—see burning bars, points, or tools.

haloing—the formation of a contrasting discoloration around the edges of the workpiece when compared to interior areas.

hardness—the relative refractoriness of a porcelain enamel or frit.

hollow ware—a class of utensils such as pots, pans, and kettles.

hospital—a special department in the porcelain enamel shop where damage to fired ware may be repaired.

impact test—a test to determine the resistance of a porcelain enamel to fracture caused by a sudden blow.

iron, enameling—see enameling iron.

jar mill—a small ball mill (see also ball mill).

jewelers’ enamel—a special type of porcelain enamel used in the manufacture of jewelry, insignia, and art objects.

jumpro or jumping—see poppers.

lift—a defect characterized by the spontaneous separation of large pieces of porcelain enamel from the base metal.

liver—in dry process enameling, a defect characterized by a wave-like form of abnormally thick porcelain enamel.

lump—in porcelain enamels, a rounded projection in the enamel surface, usually a defect.

luster—an iridescent decorative surface appearance.

marbleized finish—a surface appearance, obtained by coloring and graining, that resembles variegated marble.

maturing temperature—the temperature at which porcelain enamel must be held for a selected time to achieve the desired properties.

metal blister—bloating of the metal sheet.

metal substrate—see base metal.

mill addition—any of the materials added to the ball mill charge of a frit.

neutralizer—a dilute alkaline solution with which sheet metal ware is treated as a part of the pickling process subsequent to the acid treatment. A chemical or mixture of chemicals which, when added to water, produces the dilute alkaline solution.

nickel dipping, nickel flashing, or nickel pickling—a process for depositing metallic nickel on steel by galvanic action, reduction, or both.

nits or nitty enamel—a porcelain enamel blemish in dry process enameling characterized by minute surface pits visible only on close examination.

one-coat ware, one-coat work—(1) articles finished in a single coat of porcelain enamel.

(2) Sometimes a contraction of one-cover-coat ware, in which the finish consists of a single cover coat applied over ground coat.

one-fire finish—a porcelain enamel on the finished product processed in a single firing.

opacifier—a material that imparts or increases the diffuse reflectance of porcelain enamel.

opacity—the property of reflecting light diffusely and nonselectively; properly defined in Test Method C 347 under the term contrast ratio.

orange peel—a surface condition characterized by an irregular waviness of the porcelain enamel resembling an orange skin in texture; sometimes considered a defect.

overspray—the slip from the spray gun not deposited on the ware. Also, spray application of a light coat of slip to an unfired porcelain enamel.

particle size distribution—the percentage by mass or by
number of each fraction into which a powder sample has been classified with respect to sieve number or microns.

pebble mill—see ball mill.

peeling—a defect characterized by the spontaneous detachment of pieces of porcelain enamel from cast iron.

pickle basket—a basket fabricated from corrosion-resistant material to hold ware during pickling.

pickle pills—small gelatin capsules containing chemicals used for testing the strength of pickling solutions.

pickling—the chemical process of preparing the metal surface for porcelain enameling.

pick-up—the amount of slip retained per unit area on dipped ware.

pigskin—a surface defect characterized by a texture similar to that of pigskin.

pinhole, pinholing—a porcelain enamel surface defect caused by gas evolution and characterized by a small hole resembling a pin prick that may extend to the base metal.

pin mark or point mark—a visible imprint on the back of ware left by processing tools; sometimes synonymous with burning tool mark.

pit—a defect similar to a dimple but slightly smaller.

plugging compound—a putty-like mixture of inorganic materials used to fill holes in iron castings to ensure an even surface for porcelain enameling.

point bars—see burning bars, points, or tools.

pop-off—in dry process enameling, a defect appearing as a small conical piece of porcelain enamel, either partially or entirely separated from the ware.

poppers—a defect characterized by randomly occurring, relatively small, circular shaped areas of ground coat appearing in the first cover coat sheet steel porcelain enamel.

porcelain enamel—a substantially vitreous or glassy, inorganic coating bonded to metal by fusion at a temperature above 800°F (425°C approximate).

pot furnace—a furnace used to smelt porcelain enamel raw batch contained in a crucible.

powder adhesion—the ability of an electrostatically charged powder to remain attached by static attraction to a grounded substrate.

powder porcelain enameling—process by which the application of porcelain enamel is achieved by dry electrostatic spraying.

powder porcelain resistivity—the opposition that a porcelain powder offers to the flow of direct current, equal to the voltage drop across the powder, divided by the current through the powder. (Also known as electrical resistance)

powder retention—Same as electrostatic retention.

powder to air ratio—ratio of the mass of powder delivered to the spray gun to the total volume of air used to convey and aspirate it.

preheat zone—that portion of a continuous furnace through which the ware passes before entering the firing zone.

pressure tank—a container from which slip is removed by air pressure.

primary boiling—the evolution of gas during the initial firing of porcelain enamel; sometimes a defect.

process fishscaling—fishscaling that appears during the drying or firing cycle of cover coat application.

pyro—a common expression for the compound tetrasodium pyrophosphate (Na₄P₂O₇), either hydrous or anhydrous.

quenching—see fritting.

reboiling—gas evolution occurring and recurring during repeated firing of the ground coat; sometimes a defect.

recirculating dip tank—a dip tank provided with a means for keeping the slip in constant circulation.

reclaim—overspray that is removed from the spray booth and reconditioned for use.

reflectance—the fraction of incident light that is diffusely reflected, measured relative to magnesium oxide under standard conditions.

Note 14—See Test Method C 347.

reflectivity—the reflectance of a coating so thick that additional thickness does not change the reflectance.

Note 15—See Test Method C 347.

refractory composite coating—a combination of heat-resistant ceramic materials applied to a metallic substrate which may or may not require heat treatment prior to service. This term may also be used for coatings applied to nonmetallic substrates, for example, graphite.

rheology—the science of measuring the flow and deformation properties of matter. For porcelain enamel slips, the most important parameter is their yield point.

ripple—in dry process enameling, a surface defect characterized by pronounced waviness, uniform over a considerable area.

rotary smelter—any of the cylindrical smelters that depend on slow rotation about a horizontal axis for agitation of the molten mass.

rubbing stone—a shaped abrasive used in stoning porcelain enamel.

sagging—(1) a defect characterized by a wavy line or lines appearing on those surfaces of porcelain enamel that have been fired in a vertical position.

(2) A defect characterized by irreversible downward bending in an article insufficiently supported during the firing cycle.


sanitary ware—porcelain enameled ware such as sinks, lavatories, and bathtubs.

scab—in porcelain enameling metal sheets or castings, a defect having the appearance of a loose piece of metal, tongue, or flap on the surface.

scale—the oxide formed on the surface of the metal during heating.

scaling—the process of forming scale with or without acid fumes; sometimes refers to spontaneous detachment of scale.

scrapings—the overspray that has been recovered from a spray booth.

screen test—a standard test for fineness of porcelain enamel slip or powder.
scumming—a defect characterized by areas of poor gloss on the surface of porcelain enamel.

self-limiting powder porcelain—the maximum thickness of electrostatically-charged powder that can be deposited as a surface film.

semi-muffle furnace—a furnace with a partial muffle, in which the products of a combustion come in contact with the ware.

set—a flow property of porcelain enamel slip affecting the rate of draining, residual thickness, and uniformity of coating.

setting-up agent or set-up agent—an electrolyte used to increase the measured pick-up of a slip.

shiner or shinier-scale—a defect characterized by minute fishscaling occurring in overfired ground coat.

shorelines—a defect characterized by a series of rings or lines in the surface of porcelain enamel similar in appearance to the lines on the shore produced by receding water.

sliding—a defect in the draining characteristics of slip wherein patches of the coating slide, producing an uneven coating.

slip, slurry—a suspension of finely divided ceramic material in liquid.

slump test—a test to determine consistency of slip whereby measurement is made of the spreading of a specified volume of slip over a flat plate.

slushing—the manipulation of dipped ware to distribute the slip uniformly and remove excess material.

smelt—a specific batch or lot of frit.

smelter—a furnace in which the raw materials of the frit batch are melted.

smelter drippings—drippings of molten glassy material formed on the crown of the smelter.

softening temperature—the temperature, under specified conditions, at which porcelain enamel or frit begins to flow.

soilability—the relative ease with which extraneous matter attaches to or builds up on the surface of a material.

solubility, excessive—the tendency of a porcelain enamel frit to dissolve, as a function of time and temperature, in the medium in which it is present in amounts sufficient to adversely affect the rheology of the porcelain enamel slip.

spall, spalling, or spontaneous spalling—a defect characterized by chipping that occurs without apparent external causes.

spark test—an electrical test in which a spark is used to detect discontinuity of coating.

specking—the discoloration of an enamel surface due to foreign particles in the fired glass.

speckled ware—a decorative finish with particles of one color appearing in a uniform background of another color or shade.

spider—a defect characterized by a starshaped fracture in the porcelain enamel.

spongy enamel—a defect characterized by masses of bubbles occurring in local areas giving rise to a spongy appearance. spontaneous chipping—see spall, spalling, or spontaneous spalling.

spray sagging—a process defect characterized by a wavy line or lines appearing on vertical surfaces of sprayed ware prior to drying.

squeegee oil—a liquid mixture of organic materials used as the vehicle in squeegee paste.

squeegee paste, screening ink, screening paste—a mixture of squeegee oil and finely divided inorganic materials such as color oxides and fluxes.

stainability—the relative ease with which a material is penetrated and discolored by a foreign material.

star marks—a defect sometimes occurring in sheet steel iron cover coats where the dried ware is set down too hard on the firing fixture points and the enamel coating is fractured.

starring—see back emission and self-limiting.

stars—a defect similar to star marks appearing in the surface as a series of small hairlines radiating from a common center. They are typical of porcelain enamel powder systems.

steel, cold-rolled—see cold-rolled steel.

stippled finish—a pebbly textured porcelain enamel, often multicolored.

stoning—the operation of removing by abrasion the undesirable portions of porcelain enamel.

strainline or strainlining—see hairline or hairlining.

swab test—a low-voltage electrical test used to evaluate continuity of porcelain enamel.

tearing—a defect in the surface of porcelain enamel, characterized by short breaks or cracks which have been healed.

transfer efficiency—the amount (weight/mass) deposited on a specified target divided by the spray gun output (weight/mass) per unit of time.

triangle bars—burning bars of triangular cross section (see also burning bars, points, or tools).

tube furnace—a muffle furnace in which combustion occurs within alloy tubes.

two coat-one fire—the application of two different coats of enamel followed by a single firing step.

U-type furnace—a continuous furnace wherein the ware travels in a U-shaped path.

top-fired muffle furnace—see porcelain enamel.

warp test—see Test Method C 314.

water mark, water spot—an appearance defect characterized by a depressed spot.

water streak—a defect occurring in the bisque characterized by a washed-out pattern in the form of a streak.

wet milling—the grinding of porcelain enamel materials with sufficient liquid to form a slurry.

wet process enameling—a method of porcelain enameling in which slip is applied to a metal article at ambient temperature, dried and fired.

zero carbon steel—see decarburized enameling steel.
Standard Test Method for 45-deg Specular Gloss of Ceramic Materials

This standard is issued under the fixed designation C 346; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1 Scope

1.1 This test method covers the determination of the specular gloss of porcelain enameled specimens, but may be applicable to other specimens having similar reflection characteristics. This test method may be used to compare the gloss of porcelain enameled specimens or to provide an index of acid or abrasion resistance by measurement of gloss loss.

NOTE 1—Specular gloss is one of several related appearance attributes that produce the sensation of glossiness. For this reason, specular gloss measurements may not always correlate well with visual rankings of glossiness.

NOTE 2—Improved correlations with visual judgments can sometimes be achieved by the use of instruments with different geometries than those specified herein. Refer to Test Method D 523 for 20, 60, and 85-deg geometries. Values generally cannot be predicated for one geometry from measurements made with another.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

D 523 Test Method for Specular Gloss

E 1347 Test Method for Color and Color-Difference Measurement by Tristimulus (Filter) Colorimetry

3. Terminology

3.1 Definitions:

3.1.1 specular gloss—the ratio of reflected to incident light, times 1000, for specified apertures of illumination and reception when the axis of reception coincides with the mirror image of the axis of illumination.

NOTE 3—In this operational definition, the quantity defined as specular gloss is composed of surface-reflected and body-reflected components. For some low-gloss measurements, an approximate evaluation of the surface-reflected component of specular gloss may be required (see Section 9).

3.1.2 45-deg specular gloss—the fraction of visible light incident upon the specimens at 45° to the normal that is reflected in the direction of mirror reflection.

NOTE 4—Under ideal conditions, the incident beam should consist of parallel light, and only light reflected in the true direction of mirror reflection should be accepted for measurement.

3.1.3 source aperture—the angular size (solid angle) of the light source (lamp filament, if an incandescent source is used) measured from the center of the incident beam lens.

3.1.4 receptor aperture—the angular size (solid angle) of the receptor window, measured from the center of the receptor lens.

4. Significance and Use

4.1 This test method may be used to compare the gloss of porcelain enamel, ceramic, and other finishes or to provide a comparison of their resistance to attack from acid, alkali, or other environmental factors by measurement of gloss loss.

5. Apparatus

5.1 Instrumental Components—The apparatus shall consist of an incandescent light source and lens furnishing an incident beam of rays of required aperture, means for locating the surface of the specimen, and a receptor located to receive the required pyramid of rays reflected from the specimen. The receptor shall be a photosensitive device having maximum response near the middle of the visible region of the spectrum.

5.2 Geometric Conditions—The axis of the incident beam shall be 45° from the perpendicular to the specimen surface. The axis of the receptor beam shall be coincident with the mirror image of the axis of the incident beam. A flat piece of polished black glass in the specimen position shall form an image of the source in the center of the receptor window. The angular dimensions of the source and receptor shall be as specified below:

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1 This test method is under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.12 on Materials for Porcelain Enamel and Ceramic-Metal Systems, and is based on the “Gloss Test for Porcelain Enamels,” Bulletin T-18, of the Porcelain Enamel Institute, 1111 N. 19th St., Suite 200, Arlington, VA 22209.

2 Annual Book of ASTM Standards, Vol 06.01.

6. Specular Gloss Standards

6.1 Specular Gloss Standards—Primary working standards shall be highly polished, plane, black-glass surfaces, or surfaces of liquids for which the gloss is calculated from the angle of incidence and the refractive index of the material by using Fresnel’s equation. Polished black glass of refractive index 1.540 shall be assigned a 45-deg specular gloss value of 55.9. For the usual variation of refractive index of black glass, a change in index of 0.001 changes the gloss reading by 0.14.

6.2 Secondary Working Standards—Secondary working standards of ceramic tile, glass, porcelain enamel, or other materials having hard and uniform surfaces may be calibrated from the primary standards on a glossmeter determined to be in strict conformance with the requirements prescribed in 5.2.

6.3 For greatest accuracy, always orient the standards to the position in which they were originally calibrated.

6.4 The importance of the cleanliness of glass standards cannot be overemphasized. They must always be handled carefully to avoid abrading the surfaces. One or two deep scratches will not reduce the gloss as much as a large number of almost imperceptible abrasions.

6.4.1 Never use abrasive cleaners or scouring powders on standards.

6.4.2 When standards are washed in soap and water, a thin film of soap or oil may remain on the tile surface, thus resulting in a spurious gloss value. This effect has been found to be most noticeable for metal tiles and for higher angle and low gloss films of soap or oil may remain on the tile surface, thus resulting in a spurious gloss value. This effect has been found to be most noticeable for metal tiles and for higher angle and low gloss.

6.4.3 The use of a mild detergent is recommended. Reproducible cleaning of standards can be accomplished by brushing the manufacturer.

6.5 The importance of the cleanliness of glass standards cannot be overemphasized. They must always be handled carefully to avoid abrading the surfaces. One or two deep scratches will not reduce the gloss as much as a large number of almost imperceptible abrasions.

6.5.1 Never use abrasive cleaners or scouring powders on standards.

6.5.2 When standards are washed in soap and water, a thin film of soap or oil may remain on the tile surface, thus resulting in a spurious gloss value. This effect has been found to be most noticeable for metal tiles and for higher angle and low gloss.

6.5.3 The use of a mild detergent is recommended. Reproducible cleaning of standards can be accomplished by brushing them in a solution of detergent and water, 3 g/L (1 tbsp/gal) with a nylon brush followed by a forced rinse in hot water (temperature near 66°C (150°F)), with the tile immediately blotted dry with a clean paper towel. The tile must not be rubbed with the hand or paper towel, either during washing or drying.

7. Test Specimens

7.1 Only surfaces of good planarity shall be tested, if possible, since surface warpage, waviness, or curvature will affect test results seriously.

8. Procedure

8.1 Operate the glossmeter according to the instructions of the manufacturer.

8.2 Set the instrument to read the assigned gloss value of a highly polished working standard; then read the gloss of lower glass standards having poorer image-forming characteristics. If the instrument readings for the latter standards do not agree with the assigned values to within two gloss units, the instrument optics may require readjustment; preferably, this should be done by the manufacturer.

8.3 Measure the gloss of at least three portions of each specimen surface. This will give an indication of gloss uniformity.

8.4 For evaluation of change of gloss of a single specimen, which may be used as a measurement of surface deterioration of porcelain enameled or ceramic specimens, exercise particular care to see that the glossmeter is in exactly the same position on the specimen for measurements before and after treatment and that the illuminating beam is oriented the same on the specimen. For best results, make several measurements on each specimen in different reproducible positions.

Note 5—For small square or rectangular specimens, such as those frequently used for laboratory tests, a specimen stop attached to the glossmeter head will permit exact duplication of specimen position and orientation.

9. Evaluation of Components of Specular Gloss

9.1 When required (Note 6), an approximate evaluation of the two components of specular gloss may be made as follows:

9.1.1 Body-Reflected Component—Obtain an approximate evaluation of the body-reflected component (formerly called “diffuse correction”) of specular gloss by one of the following two procedures:

9.1.1.1 Adjust a goniophotometer to read 45-deg specular gloss; then illuminate the specimen perpendicularly and view it at 45 deg with the same receptor aperture as specified in 5.2. Read the magnitude of the body-reflected component.

9.1.1.2 Alternatively, measure the 45-deg, 0-deg luminous directional reflectance in accordance with Method E 97; express their reflectance as a decimal fraction and multiply by 5.5 to obtain an approximate value for the body-reflected component of specular gloss.

9.1.2 Surface-Reflected Component—Subtract the body-reflected component from the measured specular gloss to obtain the surface-reflected component.

Note 6—Evaluation of surface-reflected and body-reflected components of specular gloss will ordinarily be required only when comparing low-gloss specimens having quite different diffuse (body) reflectance.

10. Calculation

10.1 In computing the percent of gloss retained or lost, make a separate computation for each location measured and average results after computation.

10.2 Calculate percent change in gloss as follows:

Gloss loss, \( \% = \frac{G_i - G_f}{G_i} \times 100 \)

Gloss retained, \( \% = \frac{G_f}{G_i} \times 100 \)

where:

\( G_i \) = initial gloss (before treatment), and

G_f = final gloss (after treatment)
\[ G_f = \text{final gloss (after treatment)}. \]

11. Report

11.1 Report the average specular gloss reading of each specimen. When required, report the surface-reflected and body-reflected components separately.

11.2 Report the presence of any specimen for which portions of the test surface differ in gloss from the average by more than 10 % of the average.

11.3 Identify the glossmeter by the name of the manufacturer and model designation.

11.4 Identify the standards used, if desired.

12. Precision and Bias

12.1 The precision and bias of this test method is being established.
Standard Test Methods for Fusion Flow of Porcelain Enamel Frits (Flow-Button Methods)

1. Scope
1.1 These test methods cover evaluation of the relative fusion flow characteristics of samples of a given porcelain enamel frit by comparison with an established standard for that frit.

1.2 Two test methods are included, differing only in certain details of the samples and in the apparatus and procedure for preparation of test specimens. Both test methods give equally reproducible results and provide a satisfactory basis for comparison of fusion flow of the sample with that of the established standard.

1.2.1 Test Method A employs granular particles of frit to which a bonding agent has been added. Button specimens are formed under high pressure in a hydraulic press.

1.2.2 Test Method B employs crushed, sized particles of frit to which a bonding agent has been added. Button specimens are formed in a steel mold by hand.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Document
2.1 ASTM Standards:
E 11 Specification for Wire-Cloth Sieves for Testing Purposes

TEST METHOD A

3. Apparatus
3.1 Mortar, of hard steel, resistant to abrasion by the porcelain enamel frit, and conforming to the dimensions shown in Fig. 1.

NOTE 1—Suitable mortars are available commercially under the designation “tool steel crushing mortar.”

3.2 Sieves—No. 12 (1.70-mm) and No. 200 (75-µm) sieves conforming to Specification E 11.

4. Sample
4.1 A representative sample of the frit to be tested shall be obtained, mixed thoroughly, and reduced by quartering to about 25 g. This sample shall be crushed in a hard steel mortar to pass a No. 12 (1.70-mm) sieve and be retained on a No. 200 (75-µm) sieve.

5. Test Specimen
5.1 Weigh 3.5 ± 0.05-g samples of the standard frit and of the material to be tested and thoroughly mix each of these samples with 4 or 5 drops of a 1 % gum arabic solution. Mold fusion button test specimens of the standard and of the sample as directed in 5.2.

5.2 Place the sample in the steel mold. Place the mold assembly containing the sample in a hydraulic press and bring...
the press up to 3000 to 3500 lbf (13.3 to 15.6 kN) total load, and immediately release (Fig. 2). Force the formed button out of the mold with the plunger, taking care not to damage the button in any way that might change the dimensions.

5.3 Dry the test specimens in a suitable oven at 100°C for 1 h.

NOTE 3—Special care must be exercised in drying buttons formed by this method, before firing, in order to prevent bloating and possible eruption of the button when first placed in the furnace.

6. Procedure

6.1 Mounting—Place the dried fusion buttons on a fired groundcoat plate. The location of the test specimens with respect to the standard may be modified as desired. Although the gage of metal used for the ground-coated plate is not of critical importance, most satisfactory results will be obtained when the metal is not heavier than 18 gage nor lighter than 22 gage (1.02 to 0.044 mm). Use the same gage of metal consistently from test to test in order to reduce the number of variables to a minimum. The groundcoat enamel used to coat the groundcoat plate shall have a median firing temperature no more than 50°F (28°C) below the median firing temperature for the most refractory frit being tested. Place the groundcoat plate with fusion buttons in place in a horizontal position on the fusion flow rack, which shall previously have been heated to furnace temperature, and place the entire test assembly in the furnace immediately.

6.2 Firing—Fire in a furnace in which the atmosphere is oxidizing and entirely free of any products of combustion. Heat distribution within the furnace shall be such that no portion of the fusion buttons will vary more than ±5°F (±3°C) from any other button or portion thereof. The temperature shall approximate the median firing temperature for the standard that is used as a basis for comparison. Allow the buttons to remain in the horizontal position (Fig. 5(a)) until such time as fusion of the buttons is evident from the rounded appearance of the tops of the buttons. Then release the groundcoat plate to the vertical position (Fig. 5(b)) and allow the fusion buttons to flow a minimum of 50 mm, following which remove the test assembly from the furnace and allow the buttons to cool. When the flow is completed, the sides of the fusion buttons should be approximately parallel; that is, there should be no excessive spreading of the fusion buttons in the horizontal position, indicating that the buttons were allowed to remain in the horizontal position for too long a time.

6.3 Measuring Flow—Measure the greatest length of the fusion flow to the nearest 1 mm for each fusion button, including the standard.

7. Report

7.1 Report the length of the fusion flow for each test specimen in comparison with the standard run at the same time. For example, if a given specimen flows 50 mm against a flow for the standard of 55 mm, the results shall be reported as “50/55 mm.” If the width of the test specimen is observed to vary more than 10% from the width of the standard, length-times-width values shall be calculated for the comparison.

TEST METHOD B

8. Apparatus

8.1 Mortar—See 3.1.

8.2 Sieves—No. 60 (250-µm) and No. 200 (75-µm) sieves conforming to Specification E 11.

NOTE 4—Tyler Standard Series sieves No. 60 (0.0097-in. openings) and No. 200 (0.0029-in. openings) correspond to ASTM sieves Nos. 60 and 200 (U.S. Standard Sieve series numbers).

8.3 Steel Mold Assembly, consisting of a die, plunger, and backup disk as shown in Fig. 6. The inside diameter of the mold shall be 1/2 in. (12.7 mm) over its length of 11/4 in. (31.8 mm).

8.4 Fusion Flow Rack—See 3.5.

9. Sample

9.1 A representative sample of the frit to be tested shall be obtained, mixed thoroughly, and reduced by quartering to about 25 g. This sample shall be crushed by impact in a hard steel mortar to pass a No. 60 (250-µm) sieve and be retained on a No. 200 (75-µm) sieve. In order to avoid the excessive reduction of fines, they shall be removed frequently during the process of reduction by throwing the sample on the sieve and continuing the crushing of the coarser particles until all the sample passes through the sieve.

10. Preparation of Test Specimens

10.1 Weigh 3.5 ± 0.05-g samples of the standard and of the material to be tested and thoroughly mix each of these samples with 4 or 5 drops of a 1% gum arabic solution. Mold fusion button test specimens of the standard and of the sample as directed in 10.2.

10.2 Place the mixture in the steel mold with the plunger in place and tamp the mixture to form a solid button (Note 5). Force the formed button out of the mold, taking care not to damage the button in any way that might change the dimensions.

NOTE 5—Since the height of the button will generally be controlled by the density of the material, for materials of approximately the same
density such as leadless frits or lead-bearing frits, it will be found convenient to score the side of the plunger at a point representing the average height of the button for materials of the same density and tamp the powder to this mark each time a button is formed.

10.3 Dry the test specimens (Note 3).

11. Procedure

11.1 Mount and fire the fusion buttons and measure the flow as directed in Section 6.

12. Report

12.1 Report the results of the test as directed in Section 7.

13. Precision and Accuracy

13.1 No statement is made for precision or accuracy of this method because the results are used to compare the relative fusion flow characteristics of a given porcelain enamel frit, with those of an established standard to determine anticipated performance.
FIG. 4 Fusion Flow Rack

Note—(a) Fusion Buttons in Horizontal Position

(b) Fusion Buttons in Vertical Position

FIG. 5 Fusion Test Assembly

Note—in. = 25.4 mm.
FIG. 6 Details of Mold for Test Specimens—Test Method B

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Designation: C 375 – 58 (Reapproved 1999)

Standard Classification of Water Used in Milling of Porcelain Enamel

This standard is issued under the fixed designation C 375; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope
1.1 This classification covers water used in the milling of porcelain enamel frit.

2. Referenced Documents
2.1 ASTM Standards:
D 511 Test Methods for Calcium and Magnesium in Water
D 512 Test Methods for Chloride Ion in Water
D 513 Test Methods for Total and Dissolved Carbon Dioxide in Water
D 516 Test Method for Sulfate Ion in Water
D 858 Test Methods for Manganese in Water
D 1068 Test Methods for Iron in Water
D 1126 Test Method for Hardness in Water
D 1293 Test Methods for pH of Water
D 1888 Test Methods for Particulate and Dissolved Matter, Solids, or Residue in Water
D 3370 Practices for Sampling Water

3. Classification
3.1 Three classes of water are covered, based on the maximum impurity analyses as defined in Table 1. For porcelain enamel frits, Class A water should cause no difficulties in the production of a high quality finish. Class B water may be used by slight compensations in processing. Mill addition water falling into Class C should be treated before use in order to preclude faulty enamel production.

4. Methods of Analysis
4.1 Determine the elements and properties listed in Table 1 in accordance with the following ASTM methods:
4.1.1 Sampling—Practices D 3370.
4.1.2 Bicarbonate—Test Methods D 513.
4.1.3 Calcium and Magnesium—Test Methods D 511.
4.1.4 Chloride—Test Methods D 512.
4.1.5 Hardness—Test Method D 1126.
4.1.6 Iron—Test Methods D 1068.
4.1.7 Manganese—Test Methods D 858.
4.1.8 pH—Test Methods D 1293.
4.1.9 Sulfate—Test Method D 516.
4.1.10 Total Solids—Test Methods D 1888.
### TABLE 1 Classification of Water for Use in Milling Porcelain Enamel

<table>
<thead>
<tr>
<th>Class</th>
<th>Calcium</th>
<th>Magnesium</th>
<th>Iron</th>
<th>Manganese</th>
<th>Sulfate</th>
<th>Chloride</th>
<th>Hardness</th>
<th>Total Solids</th>
<th>Bicarbonate</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>31</td>
<td>7.5</td>
<td>0.30</td>
<td>0.005</td>
<td>25</td>
<td>13</td>
<td>38.5</td>
<td>147</td>
<td>117</td>
<td>7.6</td>
</tr>
<tr>
<td>B</td>
<td>43</td>
<td>10.0</td>
<td>0.62</td>
<td>1.05</td>
<td>102</td>
<td>18</td>
<td>53.0</td>
<td>198</td>
<td>86</td>
<td>7.5</td>
</tr>
<tr>
<td>C</td>
<td>53</td>
<td>18.0</td>
<td>0.43</td>
<td>...</td>
<td>105</td>
<td>18</td>
<td>71.0</td>
<td>284</td>
<td>78</td>
<td>7.3</td>
</tr>
</tbody>
</table>

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1. Scope

1.1 This test method covers the determination of the resistance of porcelain-enamed utensils to thermal shock. This test method is adaptable to any porcelain-enamel utensil that can be filled with water to a depth of 1 in. (25 mm).

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Apparatus

2.1 Hot Plate, capable of maintaining over its entire surface a uniform temperature, that is, less than 20°F (−6.7°C) variation over the entire surface. Surface temperature of the hot plate shall be capable of being adjusted from 475 to 825 ± 10°F (245 to 440 ± 5.5°C). The surface temperature of the hot plate, determined by any suitable surface temperature measuring device, may be varied by changing the wattage input to the hot plate. If the hot plate surface temperature be calibrated to wattage input, the desired temperature may then be set by adjusting wattage input. The wattage input or similar calibration shall be made for each surface temperature (see 6.2 and 6.3).

2.2 Timing Device, a clock or similar instrument having a sweep second hand and an integrating minute hand.

2.3 Container, suitable for holding approximately 4 gal of water.

2.4 Container, for pouring water into utensil.

2.5 Sponge, for wiping utensil dry.

3. Quenching Water

3.1 Either tap or distilled water shall be used.

NOTE 1—In areas where tap water is extremely hard, distilled water is recommended.

4. Test Specimens

4.1 The test specimens shall consist of five identical utensils.

NOTE 2—“Identical utensils” signifies utensils of the same size, shape, and finish.

5. Preparations for Test

5.1 Level the hot plate. Switch on the hot plate and adjust the surface temperature to 475 ± 10°F (245 ± 5.5°C). Allow the hot plate to preheat at this temperature for at least 1 h to obtain a uniform temperature over the entire heating surface.

5.2 Place the quenching water in a large container. Adjust the temperature of the water to 70 ± 2°F (21 ± 1°C). Measure out the amount of water required to fill a test utensil to a depth of 1 in. (25 mm); this amount of water shall be used for quenching.

NOTE 3—The test utensils must be at room temperature at the start of the test.

6. Procedure

6.1 Center a dry utensil directly on the preheated hot plate. After 3 min remove the utensil from the hot plate and immediately fill the utensil to a depth of 1 in. (25 mm) with quenching water. After 10 s pour out the quenching water and wipe the inside of the utensil with a wrung-out damp sponge. After a total nonheating period of 20 s replace the utensil on the hot plate.

6.2 Immediately after centering the utensil on the hot plate, adjust the input to the hot plate so that the hot plate surface temperature will reach 500 ± 10°F (260 ± 5.5°C). Allow the utensil to heat dry for 8 min with the hot plate adjusted so that this new surface temperature will be reached.

NOTE 4—Do not remove the utensil for surface temperature measurements during the test. The surface temperature of the hot plate should be calibrated against wattage input before the test is started. The surface temperature of the hot plate is the maximum temperature to be reached during the 8-min heating period (see 2.1).

6.3 After the 8-min heating period, remove the utensil and quench as before. Repeat this procedure according to the schedule given in Table 1. Continue the test until the utensil fails or has withstood quenching after the 825°F (440°C) heating period.

NOTE 5—A failure is the removal of the enamel from the utensil, generally accompanied by a cracking noise, during the heating period. Fish-scaling and crazing are not considered thermal-shock failures.
TABLE 1 Heating Schedule

<table>
<thead>
<tr>
<th>Time of Quench, min:s</th>
<th>Cycle</th>
<th>Hot Plate Surface Temperature,°F (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>3:00</td>
<td>1</td>
<td>475 ± 10 (245 ± 5.5)</td>
</tr>
<tr>
<td>11:20</td>
<td>2</td>
<td>500 ± 10 (260 ± 5.5)</td>
</tr>
<tr>
<td>19:40</td>
<td>3</td>
<td>525 ± 10 (275 ± 5.5)</td>
</tr>
<tr>
<td>28:00</td>
<td>4</td>
<td>550 ± 10 (287 ± 5.5)</td>
</tr>
<tr>
<td>36:20</td>
<td>5</td>
<td>575 ± 10 (301 ± 5.5)</td>
</tr>
<tr>
<td>44:40</td>
<td>6</td>
<td>600 ± 10 (315 ± 5.5)</td>
</tr>
<tr>
<td>53:00</td>
<td>7</td>
<td>625 ± 10 (329 ± 5.5)</td>
</tr>
<tr>
<td>61:20</td>
<td>9</td>
<td>675 ± 10 (357 ± 5.5)</td>
</tr>
<tr>
<td>69:40</td>
<td>11</td>
<td>725 ± 10 (385 ± 5.5)</td>
</tr>
<tr>
<td>78:00</td>
<td>13</td>
<td>775 ± 10 (413 ± 5.5)</td>
</tr>
<tr>
<td>86:20</td>
<td>15</td>
<td>825 ± 10 (440 ± 5.5)</td>
</tr>
</tbody>
</table>

8. Report

8.1 Report the average of the ratings for five identical utensils as the thermal shock resistance value.

9. Precision and Bias

9.1 No justifiable statements can be made regarding the precision and bias of this test method due to the fact that it is applicable to a wide variety of utensils of different design rather than a single, specific size and design of utensil. Since design, base metal composition, fabrication, and processing, as well as porcelain enameling, will give rise to variables in the thermal shock resistance of porcelain-enamed utensils, each design of utensil should be considered separately.
Standard Test Methods for Abrasion Resistance of Porcelain Enamels

This standard is issued under the fixed designation C 448; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope
1.1 These test methods cover determination of the resistance of porcelain enamels to surface abrasion and subsurface abrasion.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents
2.1 ASTM Standards:
C 346 Test Method for 45-deg Specular Gloss of Ceramic Materials

3. Summary of Test Methods
3.1 The first of the tests described herein is intended for the determination of the resistance to surface abrasion of porcelain enamels for which the unabraded 45° specular gloss is more than 30 gloss units. It consists essentially of measuring the specular gloss of the specimens before and after a specified abrasive treatment of the surface, and taking the percentage of the original specular gloss that is retained after treatment as the surface abrasion index.

3.2 The second test is intended for the determination of the resistance to surface abrasion of porcelain enamels for which the unabraded 45° specular gloss is 30 gloss units or less. It consists of determining the weight loss by a specified abrasive treatment and multiplying this weight loss by an adjustment factor associated with each abrasive tester, lot of abrasive, and lot of calibrated plate glass standards used. The adjusted weight loss is taken as an index of resistance to surface abrasion.

3.3 The third test is intended for the determination of the resistance of porcelain enamels to subsurface abrasion. It consists of determining the slope of the linear portion of the abrasion time-weight loss curve and multiplying by an adjustment factor associated with each abrasion tester, lot of abrasive, and lot of calibrated plate glass standards used. The adjusted slope is taken as an index of resistance to subsurface abrasion.

4. Significance and Use
4.1 When a porcelain enamel is first subjected to abrasion of the type involved in these tests, the rate of wear or attrition is relatively low. As the enamel is subjected to continued abrasion, the rate of wear increases until it reaches a steady value. Thereafter, the rate of wear remains almost constant until the enamel is penetrated and the underlying ground coat or metal exposed. The abrasion that occurs during the period of increasing rate-of-weight loss is defined as surface abrasion and results in reduced gloss and cleanability with high-gloss enamels and a modification of color, appearance, or surface texture, or combination thereof with low-gloss enamels. The abrasion that occurs during the period of steady rate-of-weight loss is defined as subsurface abrasion and results in the destruction of the continuity of the coating. These two types of abrasion are not necessarily proportional, and since it is desirable to be able to determine the resistance of porcelain enamel to both types of abrasion, it is necessary to deal with each one separately.

5. Apparatus
5.1 Balance, having a capacity of approximately 200 g and accurate to 0.0001 g.

5.2 Alloy Balls—Rust-resisting alloy balls of the type used for bearings, 5/32 in. (4 mm) in diameter. About 4 lb (1.8 kg) are required. Second-grade balls are satisfactory.

5.3 Apparatus for Measuring Specular Gloss, conforming to the requirements of Test Method C 346 and having a repeatable precision of not less than ± 0.5 gloss unit. Means shall be provided for positioning the specimen, with respect to the gloss head, so that, for a given orientation, the position of the specimen during the final gloss reading may be controlled to within 1/16 in. (1.6 mm) of that during the original gloss reading.

NOTE 1—Care should be taken to keep the interior of the glossmeter free of dust. If a glossmeter is used with the opening at the top, a piece of phenolic resin or similar material should be placed over the opening when

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1 These test methods are under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and are the direct responsibility of Subcommittee B08.12 on Materials for Porcelain Enamel and Ceramic-Metal Systems.


2 Annual Book of ASTM Standards, Vol 02.05.

3 Balls meeting the requirements of this paragraph are available from the Mobay Corporation, 5601 Eastern Avenue, Baltimore, MD 21224.

4 Suitable instruments are available from: Pacific Scientific Company, Gardner/Neotec Instrument Division, 2431 Linden Lane, Silver Spring, MD 20910; and Hunter Lab, 11495 Sunset Hills Road, Reston, VA 22090.
the instrument is not in use. The lenses should be cleaned at regular intervals with a soft camel’s-hair brush.

The black gloss standard should be kept free of scratches, and should be protected by wrapping with a soft cloth when not in use.

5.4 Apparatus for Oscillating Specimens — The apparatus used for oscillating the specimens during test shall impart to nine specimens simultaneously a horizontal circular motion such that every point on each specimen describes a circle \( \frac{7}{8} \) in. (22.2 mm) in diameter. The apparatus shall be provided with an automatic timing device capable of being preset to within 1 s of the desired time and shall operate at a frequency of 300 ± 3 cpm (for machine manufactured prior to July 1981) or 345 ± 3 cpm (for machines manufactured after July 1981). The apparatus shall be operated on a firm and level surface.

5.5 Retaining Rings — The retaining rings shall be constructed of metal lined with rubber. The inside height and diameter of the rubber-lined ring shall be 13\( \frac{1}{16} \) in. (30.2 ± 1.6 mm) and 3\( \frac{7}{16} \) in. (87.3 ± 1.6 mm), respectively. Provision shall be made for clamping the retaining ring to an enameled metal specimen to produce a watertight seal. Means shall be provided for introducing an abrasive charge after the specimen is secured. Nine retaining rings are required.

5.6 Buret, of suitable capacity to deliver 20 mL of water at 20°C (68°F).

6. Abrasives

6.1 For the surface abrasion tests the −70 +100-mesh fraction of Pennsylvania-type glass sand, preferably as ground from quartzite (quartz rock), shall be used. For the subsurface abrasion test No. 80 grit aluminum oxide abrasive medium shall be used.

7. Reference Standards

7.1 Specimens of standard calibrated polished plate glass which shows a coefficient of variation no greater than 1.5 % when tested for surface abrasion in accordance with the procedure as specified in Section 10, and which meets requirements of Table A1.4 shall be used as reference standards.

8. Test Specimens

8.1 Test specimens shall be 4\( \frac{1}{8} \) ± \( \frac{1}{4} \) in. (111.1 ± 6.4 mm) square, and may be prepared by enameling metal blanks of that size or by cutting a larger piece. Specimens for the weight loss tests should not exceed the capacity of the analytical balance selected for obtaining the weights of the specimens. When gloss measurements are to be made, the specimens tested should be as flat and free of orange peel or wavy surface as possible. Variations from flatness decrease the accuracy of gloss measurements.

8.2 Six specimens shall be tested for each determination of resistance to surface abrasion or resistance to subsurface abrasion.

9. Specimen Preparation

9.1 Before making any measurements, wash each specimen with a soft sponge moistened with a warm 1 % solution of trisodium phosphate (distilled water not essential) and rinse in warm, running tap water. If, when rinsing, the water gathers in drops on the surface, repeat the washing treatment until the rinse water spreads evenly. While the specimen is still wet, rinse it with ethyl alcohol. A small stream of alcohol from an ordinary chemical wash bottle will suffice for rinsing. Pure ethyl alcohol is preferable, but if it is not available, ethyl alcohol that has been denatured with up to 5 % of a noncorrosive, highly volatile organic compound such as methyl alcohol may be used. Allow the specimens to air dry in a vertical position and then place in a desiccator. This will prevent damage and moisture absorption of the specimens which can adversely affect the weight values. Gloss and weight determinations should be obtained within a 15-min period after the specimens have been cleaned and placed in the desiccator.

NOTE 2 — Other denaturants, approved by the U. S. Bureau of Internal Revenue, that are not objectionable for this use are ethyl ether and benzene, either alone or in combination with methyl alcohol. If ethyl alcohol is not available, isopropyl alcohol or acetone may be used, but pure ethyl alcohol is recommended if available. Avoid alcohol denatured with an ingredient of low volatility, which will remain as a surface film on the specimen when used to rinse it before or after treatment. Surface films may significantly affect gloss readings.

10. Resistance to Surface Abrasion of Porcelain Enamels

10.1 Marking of Specimens and Determining Initial 45° Specular Gloss — Mark each specimen so that its orientation may be controlled. A mark on the back at one edge will suffice. Place this edge against the specimen guide on the gloss head for the first reading. Then make three other readings, turning the specimen clockwise through 90° between readings.

NOTE 3 — The measured gloss of light-transmitting specimens of such materials as transparent or translucent glass or plastic may be affected by light reflected from the back side of the specimen or transmitted through the specimen from the room. To minimize errors from this source, the following precautions should be taken:

1. Roughen the back of transparent specimens to eliminate specular reflection from this surface.

2. Cover back and edges of light-transmitting specimens with an opaque black cloth when measuring gloss to prevent normal room illumination from being transmitted through the specimen, or light from the instrument from being reflected back through the specimen from a light surface in contact with the back of the specimen. This is particularly important when using a glossmeter with the specimen holder on top.

3. Put no labels near the center of light-transmitting specimens.

10.2 Determining Correct Abrasion Time — The correct abrasion time is the time required to reduce the 45° specular gloss of a standard plate glass specimen to 53 ± 1 %.\(^7\) Determine this time by abrading six standard plate glass specimens and calculating the average percentage 45° specular gloss retained. A good trial time is 6.117 min (184 counts) on machines manufactured prior to July 1981, or 4.367 min (150 counts) on machines manufactured after July 1981.

\(^7\) This value may change slightly from time to time when it is necessary to replenish the supply of standards. In any case, the corrected value will be furnished with each lot of standards (see Annex A4).
10.3 Securing Specimens to Table of Abrasion Tester and Introducing Abrasive Mixture—Center each specimen in one of the nine available positions and secure by means of the retaining ring. Tighten the two wing nuts simultaneously and uniformly. The amount of tightening shall be just sufficient to provide a watertight seal between the retaining ring and the specimen. Introduce an abrasive charge of 175 ± 0.15 g of 5/32-in. (4-mm) rust-resisting alloy balls, 3 ± 0.01 g of -70 +100 mesh Pennsylvania-type glass sand, and 20 ± 0.2 mL of water, in that order, through the hole in the top of each retaining ring. After the abrasive charge is introduced, seal the hole in the top of the retaining ring with a cork or rubber stopper.

10.4 Treatment of Specimens—Set the automatic timing device for the previously determined time required to reduce the 45° specular gloss of a standard plate glass specimen to 53%. Set the selector switch to the “automatic” position and close the toggle switch, starting the oscillator. The abrasion tester will then stop after the prescribed abrasion time.

10.5 Cleaning Specimens after Treatment:

10.5.1 Clean the sample specimens and equipment as follows:

10.5.1.1 Immediately after treatment, loosen the wing nuts that secure the specimens to the abrasion tester. Carefully remove the sample panel, retaining ring apparatus and abrasive medium as a sealed unit. (This can be accomplished by compressing the panel to the retaining ring apparatus with the hands and rotating the assembly from the confines of the securing posts of the abrasion tester. The ball bearings, the abrasive grit, and water can be discharged through a sieve that will allow the sand and water to pass through, but will retain the ball bearings. This method facilitates the cleaning and drying of the ball bearings so that they can be used in subsequent testing cycles.) If any of the ball bearings are dropped and cannot be accounted for during the cleaning of the individual retaining ring assemblies, another 175 ± 0.15 g of ball bearings shall be used in subsequent testing cycles.

10.5.1.2 Scrub the abraded portion very lightly with a clean sponge that has been saturated with warm water, rinse with warm, running water, and, while still wet, rinse with alcohol and place in a vertical position to dry.

10.6 Determining Final 45° Specular Gloss—Make the final gloss readings within 15 min after the specimens have been given the final rinsing, following the procedure outlined in 10.1.

10.7 Computation of Surface Abrasion Index—For each of the four orientations of the specimens, divide the final specular gloss reading by the initial reading and multiply by 100. The average percentage residual specular gloss for the four positions shall be taken as the surface abrasion index of the specimen. The average index of six specimens after treatment shall be taken as the abrasion index of a given index of enameled metal.

Note 4—If it is desirable, nine samples may be treated simultaneously, this procedure being repeated until six specimens of each sample have been tested. If less than nine samples are to be tested, any arrangement desired may be used and the arrangement may be such that the number of operations required to test six specimens of each sample is a minimum.

10.8 Use of Standard Plate Glass Specimens—As a check on the performance of the apparatus, test standard, calibrated, plate glass specimens at regular intervals. If the computed surface abrasion index obtained in a check test of six standard glass plates falls outside the limits 52.0 to 54.0 but within the limits 51.5 to 54.5, adjust the abrasion time in accordance with 10.2. If a value below 51.5 or above 54.5 is obtained, defective technique or equipment is indicated, and the source of difficulty should be found and remedied.

Note 5—This check test need not be made each time enameled metal specimens are tested. The time between check tests will be determined by the number of tests made by the laboratory.

11. Resistance to Surface Abrasion of Porcelain Enamels Having 45° Specular Gloss of 30 Gloss Units or Less

11.1 Determining Initial Weight of Specimens—Weigh each specimen to the nearest 0.1 mg within 15 min after it has been rinsed with alcohol.

Note 6—When weight determinations are to be made, the specimens should be handled with care to prevent chipping, which may introduce significant errors. This precaution is particularly important for glass plates, and in determining surface abrasion by weight loss.

11.2 Securing Specimens to Table of Abrasion Tester and Introducing Abrasive Mixture—Secure the test specimens to the table of the abrasion tester and introduce the abrasive mixture in accordance with 10.3.

11.3 Treatment of Specimens—Set the selector switch to the “automatic” position, set the automatic timing device for 10 min (300 counts) on machines manufactured prior to July 1981, or 10 min (342 counts) on machines manufactured after July 1981.

11.4 Cleaning Specimens After Treatment—After treatment, clean the specimens in accordance with 10.5. If the retaining ring has left a black mark on the specimen, this may be removed by scrubbing lightly with a soft sponge. Do not scrub the abraded area.

11.5 Determining Final Weight of Specimens—Determine the final weight of each specimen in accordance with 11.1.

11.6 Computation of Surface Abrasion Index—For each specimen, multiply the weight loss by an adjustment factor determined as specified in 12.7, but using Pennsylvania-type glass sand. The average adjusted weight loss is taken as the surface abrasion index of the enamel (Note 4).

11.7 Use of Standard Plate Glass Specimens—As a check on the performance of the apparatus, test standard, calibrated plate glass specimens at regular intervals in accordance with 10.8 (Note 5).

12. Resistance to Subsurface Abrasion

12.1 Determining Initial Weight of Specimens—Determine the initial weight of each specimen in accordance with 11.1.

12.2 Securing Specimens to Table of Abrasion Tester and Introducing Abrasive Mixture—Secure the test specimens to the table of the abrasion tester and introduce the abrasive mixture in accordance with 10.3, except use 3 ± 0.01 g of No. 80 grit aluminum oxide abrasive medium in place of the glass sand.

12.3 Treatment of Specimens—Set the selector switch to the “automatic” position, set the automatic timing device for 15 min (450 counts) on machines manufactured prior to July
1981, or 15 min (513 counts) on machines manufactured after July 1981.

12.4 Cleaning Specimens After Treatment—After treatment, clean the specimens in accordance with 10.5. If the retaining ring has left a black mark on the specimen, this may be removed by scrubbing lightly with a soft sponge. However, do not harshly rescrub the abraded area.

12.5 Determining Final Weight of Specimens—Determine the final weight of each specimen in accordance with 11.1.

12.6 Repeat the steps listed in 12.2-12.5 two times (a total of three 15-min abrasion periods). Designate the specimen weights after 15, 30, and 45 min of abrasion as \( W_{15} \), \( W_{30} \), and \( W_{45} \) respectively.

12.7 Determination of Adjustment Factor—Determine the adjustment factor for the abrasion tester as follows:

12.7.1 Test 24 standard plate glass specimens for eight consecutive 15-min periods as specified in 12.1-12.5, except that it is not necessary to determine the specimen weights after the first three 15-min periods, as these weights are not needed for the subsequent computations. Prior to each weighing, clean the standard plate glass specimens by thoroughly rinsing all surfaces with warm, running tap water; washing all surfaces with a soft sponge moistened with a warm, 1 % solution of trisodium phosphate (distilled water not essential); thoroughly rinsing all surfaces with warm, running tap water; while the specimen is still wet, rinsing front and back with ethyl alcohol. Allow the specimens to air dry in a vertical position and then place them in a desiccator.

12.7.2 For each specimen, subtract the weight at the end of eight 15-min periods, \( W_{120} \), from the weight at the end of four 15-min periods, \( W_{60} \), and divide by 60 to obtain the rate-of-weight loss.

12.7.3 Calculate the average rate-of-weight loss for the 24 specimens.

12.7.4 Divide this average rate-of-weight loss for the last four 15-min periods into 4.5671 (see Annex A4). An example is given in Table 1.  

12.8 Calculation of Subsurface Abrasion Index—For each specimen, subtract the weight after 45 min of abrasion, \( W_{45} \), from the weight after 15 min of abrasion, \( W_{15} \), and divide the difference by 30. The quotient, \( X_a \), is the slope of the linear portion of the abrasion time - weight loss curve and shall be taken as the true rate-of-weight loss of the specimen as determined by the laboratory with its particular abrasion tester. The true rate-of-weight loss for each specimen shall then be multiplied by the adjustment factor determined in accordance with 12.7, giving an adjusted rate-of-weight loss, \( X_{a\text{adj}} \). The average adjusted rate-of-weight loss shall be taken as the subsurface abrasion index of the enamel. An example is given in Table 2 (Note 4).

12.9 As a check to verify that the correct procedure has been followed, plot the average weight loss of the enamel as a function of abrasion time for 15, 30, and 45 min of abrasion. These three points should fall approximately on a straight line.

12.10 Use of Standard Plate Glass Specimens—As a check on the performance of the abrasion tester, test standard, calibrated, plate glass specimens at regular intervals (Note 5). Use six specimens for the check test. These specimens may be taken from the group of 24 specimens previously used for determining the adjustment factor as prescribed in 12.7. The same specimens may be used repeatedly for these check tests. Test the six glass plates for four consecutive 15-min periods as specified in 12.1-12.5. Compute the average rate-of-weight loss for this hour of abrasion and multiply by the adjustment factor for the abrasion tester. The adjusted rate-of-weight loss thus obtained should fall within ± 0.15 mg/min of the appropriate abrasion index value supplied with the standard glass specimens. If an adjusted rate-of-weight loss outside the specified ± 0.15 mg/min tolerances is obtained, this indicates that a significant change has occurred in the apparatus itself or in the test procedure. If the value obtained in a recheck of six different glass plates, in which all variables are closely controlled, confirms the value obtained in the check test, determine a new adjustment factor by ablading the remaining 12 plate glass specimens for four 15-min periods and dividing an average rate-of-weight loss for all 24 specimens into the 4.5671 (See Annex A4). Use this adjustment factor in subsequent computations. If new plate glass specimens are to be

### Table 1 Calculation of Adjustment Factor for Abrasion Tester from Weight-Loss Values for 24 Standard Plate Glass Specimens

<table>
<thead>
<tr>
<th>No.</th>
<th>( W_{60} ), g</th>
<th>( W_{120} ), g</th>
<th>( \frac{(W_{60} - W_{120})}{60} ), mg/min</th>
<th>( \frac{(W_{15} - W_{120})}{30} ), mg/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>181.981</td>
<td>181.692</td>
<td>289</td>
<td>4.817</td>
</tr>
<tr>
<td>2</td>
<td>179.995</td>
<td>179.701</td>
<td>294</td>
<td>4.900</td>
</tr>
<tr>
<td>3</td>
<td>183.633</td>
<td>183.346</td>
<td>297</td>
<td>4.783</td>
</tr>
<tr>
<td>4</td>
<td>182.734</td>
<td>182.451</td>
<td>283</td>
<td>4.717</td>
</tr>
<tr>
<td>5</td>
<td>176.193</td>
<td>175.911</td>
<td>282</td>
<td>4.700</td>
</tr>
<tr>
<td>6</td>
<td>181.109</td>
<td>180.817</td>
<td>292</td>
<td>4.867</td>
</tr>
<tr>
<td>7</td>
<td>182.212</td>
<td>181.927</td>
<td>285</td>
<td>4.750</td>
</tr>
<tr>
<td>8</td>
<td>176.128</td>
<td>175.837</td>
<td>291</td>
<td>4.850</td>
</tr>
<tr>
<td>9</td>
<td>178.492</td>
<td>178.204</td>
<td>287</td>
<td>4.783</td>
</tr>
<tr>
<td>10</td>
<td>184.363</td>
<td>184.079</td>
<td>284</td>
<td>4.733</td>
</tr>
<tr>
<td>11</td>
<td>173.998</td>
<td>173.712</td>
<td>286</td>
<td>4.767</td>
</tr>
<tr>
<td>12</td>
<td>180.299</td>
<td>180.006</td>
<td>293</td>
<td>4.883</td>
</tr>
<tr>
<td>13</td>
<td>182.742</td>
<td>182.451</td>
<td>291</td>
<td>4.850</td>
</tr>
<tr>
<td>14</td>
<td>180.259</td>
<td>179.977</td>
<td>282</td>
<td>4.700</td>
</tr>
<tr>
<td>15</td>
<td>177.668</td>
<td>177.381</td>
<td>287</td>
<td>4.783</td>
</tr>
<tr>
<td>16</td>
<td>176.877</td>
<td>176.582</td>
<td>295</td>
<td>4.917</td>
</tr>
<tr>
<td>17</td>
<td>181.681</td>
<td>181.397</td>
<td>284</td>
<td>4.733</td>
</tr>
<tr>
<td>18</td>
<td>173.362</td>
<td>173.069</td>
<td>293</td>
<td>4.883</td>
</tr>
<tr>
<td>19</td>
<td>184.004</td>
<td>183.714</td>
<td>290</td>
<td>4.833</td>
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<tr>
<td>20</td>
<td>181.214</td>
<td>180.919</td>
<td>295</td>
<td>4.917</td>
</tr>
<tr>
<td>21</td>
<td>179.292</td>
<td>179.011</td>
<td>281</td>
<td>4.683</td>
</tr>
<tr>
<td>22</td>
<td>174.673</td>
<td>174.384</td>
<td>289</td>
<td>4.817</td>
</tr>
<tr>
<td>23</td>
<td>176.476</td>
<td>176.191</td>
<td>285</td>
<td>4.750</td>
</tr>
<tr>
<td>24</td>
<td>178.610</td>
<td>178.328</td>
<td>282</td>
<td>4.700</td>
</tr>
</tbody>
</table>

**Avg:** 4.797

**Adjustment factor = 5.000/4.797 = 1.042.**

### Table 2 Calculation of Subsurface Abrasion Index of a Porcelain Enamel from Weight-Loss Values for Six Specimens

<table>
<thead>
<tr>
<th>No.</th>
<th>( W_{15} ), g</th>
<th>( W_{45} ), g</th>
<th>( \frac{(W_{15} - W_{45})}{30} ), mg/min</th>
<th>( X_a ), mg/min</th>
<th>( X_{a\text{adj}} ), mg/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>123.043</td>
<td>122.899</td>
<td>4.80</td>
<td>5.002</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>123.675</td>
<td>123.538</td>
<td>4.57</td>
<td>4.762</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>123.113</td>
<td>122.977</td>
<td>4.53</td>
<td>4.720</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>127.498</td>
<td>127.363</td>
<td>4.50</td>
<td>4.689</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>122.662</td>
<td>122.519</td>
<td>4.77</td>
<td>4.970</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>122.907</td>
<td>122.764</td>
<td>4.77</td>
<td>4.970</td>
<td></td>
</tr>
</tbody>
</table>

**Avg:** 4.8522

Subsurface abrasion index = 4.85.
used, test them first for four 15-min periods in accordance with 12.2-12.4. It is not necessary to make weight-loss determinations for these first four abrasion periods. After this initial hour of abrasion to remove the surface, the specimens may be used as described above.

13. Calculations for Single Determination

13.1 Six specimens comprise a sample. For surface abrasion of porcelain enamels having 45° specular gloss of more than 30 gloss units, calculate the grand average of the six average abrasion indices, \( X_a \), each obtained by averaging the percentage residual gloss for the four positions of one specimen, thus obtaining the mean abrasion index for the sample, \( \bar{X}_a \). For subsurface abrasion, average the six individual adjusted rates of weight loss, \( X_g \), to obtain the mean abrasion index for the sample, \( \bar{X}_g \).

13.2 Calculate the statistical error of the determination as follows (Note 7):

\[
e = 1.05 \, s
\]  

where:

\( e \) = statistical error of the mean value for the sample (95% confidence), and
\( s \) = standard deviation of the six average abrasion indices for individual specimens.

Note 7—The factor 1.05 applied only when the number of specimens is 6 and the percentage of confidence is 95. See STP 15D, Part 2, Table 2.

13.3 The standard deviation may be calculated as follows:

\[
s = \sqrt{\frac{n(\Sigma X^2) - (\Sigma X)^2}{n(n - 1)}}
\]  

where:

\( s \) = standard deviation,
\( X \) = value of a single observation,
\( \Sigma X \) = sum of the set of observations, and
\( n \) = number of observations.

13.4 Sample calculations are given below, the recommended number of decimal places being given in each case (Note 8). In the example of surface abrasion, 60 is subtracted from each value, \( X_a \), giving much smaller values, \( X_a' \), which are correspondingly easier to square. Then, to compensate, 60 is added to the average \( X_g' \) to obtain the average \( \bar{X}_g \) (Note 9).

For Surface Abrasion

\[
\begin{array}{ccc}
\text{Specimen No.} & X_a & X_a' - 60 & (X_a')^2 \\
1 & 61.5 & 1.5 & 2.25 \\
2 & 61.3 & 1.3 & 1.69 \\
3 & 62.6 & 2.6 & 6.76 \\
4 & 61.3 & 1.3 & 1.69 \\
5 & 61.6 & 1.6 & 2.56 \\
6 & 62.8 & 2.8 & 7.84 \\
\hline
\text{Sum} & 11.1 & 22.79 \\
\text{Mean} & 1.850 & \\
\end{array}
\]

Applying Eq 2,

\[
s = \sqrt{\frac{6(22.79) - (11.1)^2}{6 \times 5}}
\]

\[= \frac{\sqrt{136.74 - 123.21}}{30} = \frac{13.53}{30} = \sqrt{0.451}
\]

\[s = 0.6716, \quad e = 1.05 \times 0.6716 = 0.705 \text{ (Eq 1)} \]

\[\bar{X}_a = 1.85 + 60 = 61.85 \]

The surface abrasion index is reported as 61.9 ± 0.7.

For Subsurface Abrasion

\[
\begin{array}{ccc}
\text{Specimen No.} & X_g & (X_g')^2 \\
1 & 4.970 & 24.7009 \\
2 & 4.970 & 24.7009 \\
3 & 5.002 & 25.0200 \\
4 & 4.689 & 21.9866 \\
5 & 4.720 & 22.2784 \\
6 & 4.762 & 22.6766 \\
\hline
\text{Sum} & 29.113 & 141.3635 \\
\text{Mean} & 4.8522 & \\
\end{array}
\]

Applying Eq 2,

\[
s = \sqrt{\frac{6(141.3635) - (29.113)^2}{6(6 - 1)}} = \sqrt{\frac{848.181 - 847.5668}{30}} = \sqrt{0.020473} = 0.1431 = 1.05 \times 0.1431 = 0.1503 \text{ (Eq 1)} \]

\[\bar{X}_g = 4.85 \]

The subsurface abrasion index of the group of specimens is reported as 4.85 ± 0.15.

Note 8—Take care to carry the calculations to two or three places beyond the decimal when so indicated in the sample calculations. Otherwise, significant errors are frequently introduced in computing standard deviations.

Note 9—The number to be subtracted should be an integer, just lower than the lowest value in the set.

14. Difference Between Two Determinations

14.1 The significance (or lack of significance) of a difference between two mean values shall be determined from the ratio \( d/e' \) where \( d \) represents the difference in means and \( e' \) the statistical error in the determination of \( d \). From the graph in Fig. 1, the value of the ratio \( d/e' \) may be translated into terms of the percentage confidence that the difference in mean values indicates a systematic difference in the types of specimens being tested rather than mere chance fluctuations in sampling and testing, such as might occur even though both sets of specimens were taken from groups in which the grand averages were equal.

14.2 The statistical error, \( e' \), of the difference, \( d \), between two means shall be determined from the following equation:

\[
e' = \sqrt{e_1^2 + e_2^2}
\]

where:

\( e' \) = error of the difference in means,
\( e_1 \) = error of one mean value, and
\( e_2 \) = error of the other mean value.
14.3 Sample calculations are given below:

For Surface Abrasion:

\[ \bar{X}_1 = 53.30 \pm 0.92 \]
\[ \bar{X}_2 = 55.62 \pm 1.13 \]
\[ d = \bar{X}_2 - \bar{X}_1 = 55.62 - 53.30 = 2.32 \]

Applying Eq 3,
\[ e' = \sqrt{e_1^2 + e_2^2} = \sqrt{(0.92)^2 + (1.13)^2} \]
\[ = \sqrt{2.723} = 1.46 \]
\[ d/e' = 2.32/1.46 = 1.59 \]

For Subsurface Abrasion:

\[ \bar{X}_{a1} = 4.593 \pm 0.143 \]
\[ \bar{X}_{a2} = 4.309 \pm 0.122 \]
\[ d = \bar{X}_{a2} - \bar{X}_{a1} = 4.593 - 4.309 = 0.284 \]

Applying Eq 3,
\[ e' = \sqrt{(0.143)^2 + (0.122)^2} = \sqrt{0.0353} = 0.188 \]
\[ d/e' = 0.284/0.188 = 1.51 \]

14.4 From Fig. 1, it is apparent that a ratio of 1.59 indicates slightly more than 99% confidence and that a ratio of 1.51 indicates slightly less than 99% confidence. A ratio indicating a percentage confidence equal to or exceeding 99% is considered highly significant, 95% or more but less than 99% is considered significant, and 90% or more but less than 95% is considered indicative. The basing of conclusions on differences in which less than 90% confidence can be placed is not recommended. If \( d/e' \) equals or exceeds 1, a confidence of 95% or more is indicated, and the difference is considered significant.

15. Precision and Bias

15.1 The precision and bias of this test method will depend upon the uniformity of the samples being tested and the skill and ability of the operator in following the procedures outlined.

15.1.1 The precision of the surface abrasion test as indicated by the maximum allowable variation in determining the surface abrasion index of standard plate glass specimens having an average surface abrasion index of 53% with a coefficient of variation of 1.5% or less is ± 1% (range 52.0 to 54.0).

15.1.2 The precision of the subsurface abrasion test as indicated by the maximum allowable variation in measuring the subsurface abrasion index of standard plate glass specimens having an adjusted subsurface index of 4.5671 (see Annex A4) with a coefficient of variation of 1% or less is ± 0.15.

15.2 Results of the test may be affected by the frequency of oscillation in cpm delivered by the particular machine used.
A1. CHARACTERISTICS OF ABRASION RESISTANCE TEST MATERIALS

A1.1 This annex specifies the characteristics of abrasion-resistance test materials (alloy balls, Pennsylvania-type glass sand, No. 80 grit aluminum oxide abrasive medium, and standard soda-lime plate glass specimens) sufficiently to permit the purchase of these materials directly from the appropriate manufacturers if the indicated central source of these materials (see Note 3 and Note 6) is no longer the supply source. The characteristics of the abrasion resistance test materials are listed in Tables A1.1-A1.4.

### TABLE A1.1 Alloy Balls

<table>
<thead>
<tr>
<th>Material description</th>
<th>Alloy balls (see 5.2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Procurement source</td>
<td>Bearings, Inc., 2818 Loch Raven Road, Baltimore, Md. 21218</td>
</tr>
<tr>
<td>Characteristics</td>
<td>5/32 in. (4 mm) diameter, rust-resisting, Type 440, 200 grade, 200 balls per pound (0.5 kg)</td>
</tr>
<tr>
<td>Amount required for one specimen</td>
<td>175 ± 0.15 g (see 10.3)</td>
</tr>
</tbody>
</table>

### TABLE A1.2 Pennsylvania-Type Glass Sand

<table>
<thead>
<tr>
<th>Material description</th>
<th>−70 + 100-mesh fraction of Pennsylvania-type glass sand (see 6.1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Characteristics</td>
<td>The abrasive characteristic of this glass sand depends upon the number of cutting surfaces available per sand particle. To obtain the greatest number of cutting surfaces possible it is necessary to specify this silica sand as coming from ground quartzite (quartz rock).</td>
</tr>
<tr>
<td>Amount required for one specimen</td>
<td>3 ± 0.01 g (see 10.3)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Typical screen analysis</th>
<th>Mesh</th>
<th>% Cumulative Retained</th>
</tr>
</thead>
<tbody>
<tr>
<td>Size</td>
<td></td>
<td></td>
</tr>
<tr>
<td>40</td>
<td></td>
<td>...</td>
</tr>
<tr>
<td>50</td>
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<td>trace</td>
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<tr>
<td>70</td>
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<tr>
<td>Pan</td>
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<td>100.–</td>
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</table>
### TABLE A1.3 Aluminum Oxide Abrasive Medium

<table>
<thead>
<tr>
<th>Material description</th>
<th>No. 80 grit aluminum oxide abrasive medium (see 6.1)</th>
</tr>
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<tbody>
<tr>
<td>Trade name</td>
<td>Dynablast</td>
</tr>
<tr>
<td>Characteristics</td>
<td>See composition</td>
</tr>
<tr>
<td>Amount required for one specimen</td>
<td>3 ± 0.01 g (see 12.2)</td>
</tr>
</tbody>
</table>

#### Typical composition and screen analysis

<table>
<thead>
<tr>
<th>Component</th>
<th>Amount, (%)</th>
<th>Mesh Size</th>
<th>% Cumulative Retained</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al₂O₃</td>
<td>95.8</td>
<td>60</td>
<td>0.08</td>
</tr>
<tr>
<td>TiO₂</td>
<td>2.6</td>
<td>70</td>
<td>3.79</td>
</tr>
<tr>
<td>SiO₂</td>
<td>1.0</td>
<td>80</td>
<td>26.91</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>0.2</td>
<td>100</td>
<td>67.74</td>
</tr>
<tr>
<td>MgO</td>
<td>0.2</td>
<td>150</td>
<td>93.38</td>
</tr>
<tr>
<td>ZrO₂</td>
<td>0.1</td>
<td>Pan</td>
<td>100.00</td>
</tr>
</tbody>
</table>

*The silica (SiO₂) value shown is present as combined silica not as free silica. Composition varies slightly with change in grit size.*

### TABLE A1.4 Standard Plate Glass Specimens

<table>
<thead>
<tr>
<th>Material description</th>
<th>Calibrated plate glass standards (see 10.8 and 12.10)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Procurement source</td>
<td>Libbey-Owens-Ford, Company Technical Center, 1701 East Broadway, Toledo, OH 43605</td>
</tr>
<tr>
<td>Characteristics</td>
<td>Size - 4-3/8 by 4-3/8 by 1/4 in. (111 by 111 by 6.4 mm)</td>
</tr>
<tr>
<td></td>
<td>Float glass, center tension between 313 and 362 psi (2157 and 2494 kPa)</td>
</tr>
<tr>
<td></td>
<td>When plate glass is produced by the float process the side of the sheet in contact with the molten tin will show a somewhat higher stress (higher abrasion resistance) than the surface which has not been in contact with the tin. The higher stressed surface should not be used in the calibration and standardization tests (10.2, 10.8, and 11.7) since it is not the calibrated side of the glass. The side of the glass which has been in contact with the molten tin can be readily identified by the fact that it will fluoresce under ultraviolet light. See Typical Composition.</td>
</tr>
<tr>
<td>Amount required for one test</td>
<td>6 Calibrated glass plates (see 10.8 and 12.10)</td>
</tr>
</tbody>
</table>

#### Typical composition, % normal soda-lime plate glass

<table>
<thead>
<tr>
<th>Component</th>
<th>SiO₂</th>
<th>Na₂O</th>
<th>CaO</th>
<th>MgO</th>
<th>R₂O₃(^a)</th>
<th>B₂O₃</th>
<th>K₂O</th>
<th>Li₂O</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>73</td>
<td>15.2</td>
<td>6.7</td>
<td>4.6</td>
<td>1.56</td>
<td>0.03</td>
<td>0.05</td>
<td>&lt;0.01</td>
</tr>
</tbody>
</table>

\(^a\)R₂O₃ includes Fe₂O₃, TiO₂, and ZrO₂.
A2. ABRASION TESTER

A2.1 An abrasion tester that meets the requirements of 5.4 and 5.5 of this test method is shown in Fig. A2.1. The figure shows the abrasion tester with test specimens and retaining rings clamped in place. The pertinent parts of the instrument are numbered and described as follows:

A2.1.1 Rubber-coated retaining rings (specified in 5.5), showing opening in the top for introducing the abrasive charge. Also shown is the method used to clamp the ring and the specimen to the top plate of the abrasion tester, forming a waterproof seal between ring and sample plate.

A2.1.2 Test specimen.

A2.1.3 Top plate of the tester. The plate is supported by a vertical shaft which transmits to it the horizontal circular motion required to cause every point on each specimen to describe a circle 0.875 in. (22.2 mm) in diameter.

A2.1.4 Aluminum housing containing the synchronous drive motor, sealed gear train, and cams required to rotate the top plate.

A2.1.5 ON-OFF toggle switch.

A2.1.6 MANUAL-AUTOMATIC selector switch.

A2.1.7 Circuit breaker button.

A2.1.8 Automatic timer.

A2.1.9 Procurement Source—Keystone Electric Co., 2807 Annapolis Road, Baltimore, MD 21230.

A3. GLOSSMETER CHARACTERISTICS AS APPLIED TO THIS TEST METHOD

A3.1 The instruments that claimed precision of ± 0.5 gloss units can be improved in the upper ranges by frequent checking against the black (high-gloss) standard. Checks after every four readings (with correction if needed) increased the precision of the instrument to ± 0.1 unit.

A3.2 In a similar manner, read an intermediate (25-unit) standard after every four readings of the abraded plates, followed by the high gloss check. High gloss checks alone do not improve low gloss precision.

NOTE A3.1—Intermediate standards are subject to change in gloss value if they are mishandled, scratched, or even cleaned in a careless manner. Glossmeters should therefore always be standardized by means of the black gloss standard. The intermediate (or series of intermediate) standard(s) should be used infrequently to check the long-term stability of the glossmeter. They should be handled as little as possible.

A3.3 Due to minor variations in internal geometry, the precision between instruments and between laboratories ranges from ± 1 to ± 2 gloss units. This can give rise to an 8% variation between laboratories.

A3.4 In view of the above, differences between laboratories should be arbitrated by using the same glossmeter and standards in both laboratories, or by an independent laboratory.
A3.5 Surface abrasion will readily differentiate between different material types, but is not precise enough for control and most mill formula variations.

A4. ABRASION INDICES FOR THE CURRENT BATCH OF STANDARD PLATE GLASS SPECIMENS.

A4.1 Subsurface abrasion index as determined on the machine used as a standard, manufactured prior to July 1981: 4.5671.

A4.2 Subsurface abrasion index as determined on the machine used as a standard, manufactured after July 1981: 5.9000.

A4.3 Surface abrasion index (weight loss method) as determined on the machine used as the standard, manufactured prior to July 1981: 3.8948.

A4.4 Surface abrasion index (weight loss method) as determined on the machine used as the standard, manufactured after July 1981: 3.3666.

A4.5 Subsurface abrasion index (gloss loss method): 53 %.
Standard Test Method for Continuity of Coatings in Glassed Steel Equipment by Electrical Testing

This standard is issued under the fixed designation C 536; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the detection of discontinuities in the glass coating of glassed steel equipment where such discontinuities would result in early failure due to the attack of the chemical contents of the vessel on the metal substrate. It is applicable to (1) provide a manufacturing and inspection test of glassed-steel equipment designed for relatively mild, low-temperature corrosive service, and (2) the field testing of similar equipment used in more severely corrosive environment.

NOTE 1—A manufacturing test method for the latter type of equipment is described in Method C 537.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Document

2.1 ASTM Standards:

C 537 Test Method for Reliability of Glass Coatings on Glassed Steel Reaction Equipment by High Voltage

3. Summary of Test Method

3.1 The test method consists essentially of grounding the metal structure of the equipment being tested to the ground side of a voltage generator and sweeping the surface of the glass with a suitable probe electrically connected to the other side of the generator. Wherever a discontinuity exists, a discharge will give a positive indication that such a discontinuity exists. The voltage is set at 5000 V in order to show existing discontinuities without breaking through the existing glass coating. A built-in current limiting device ensures electrical safety for the operator.

4. Significance and Use

4.1 This test method is designed to detect existing discontinuities in the glass coating of glassed steel equipment while maintaining a voltage low enough as to be unlikely to cause breakdown of the coating. The test is adaptable for manufacturing inspection in the processing of equipment and for field use to find existing defects which can be “plugged” or repaired before serious damage is done to the equipment. As a means of positive detection, it is applicable to process studies, quality control, or specification.

5. Definition

5.1 glassed steel (glass-lined steel or glass-coated steel)—designations generally applied to a class of porcelain enameled that have high resistance to chemical attack at elevated temperatures and pressures.

6. Interferences

6.1 Since the test method is electrical, it is necessary to have a good ground connection between the instrument and the metal substrate of the equipment being tested. It is also necessary that the surfaces of the glass be reasonably clean and dry. A wet surface will conduct enough voltage to any exposed metal to give an indication of a “contact” over a large area instead of at a specific area of discontinuity. Such a capacitance discharge can be distinguished from a true failure by extending over a large area instead of at local spots that could be identified and marked for repair.

7. Apparatus

7.1 The test apparatus comprises a voltage stabilizing transformer, a step-up transformer, a current-limiting resistance, a voltmeter and pilot light, along with a suitably insulated cable and probe handle equipped with interchangeable wire brush-type and point electrodes (see Fig. 1 for a schematic wiring diagram). The current output is limited to 2.5 mA. The wire brush-type electrode is designed for sweeping larger areas of glass coating while the point electrode is better adapted to corners and the more restricted areas.

7.2 A portable 5000-volt d-c tester may be used in this test method.

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1 This test method is under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.12 on Materials for Porcelain Enamel and Ceramic-Metal Systems.


2 Annual Book of ASTM Standards, Vol 02.05.
8. Safety Precautions

8.1 The equipment being tested should be well grounded as well as electrically connected to the ground side of the testing apparatus.

8.2 The probe should be handled only by the insulating plastic handle.

8.3 Keep the probe electrode away from personnel and from conducting surfaces that might lead to personnel.

8.4 Turn the switch off and ground the electrode before changing probe heads. Although the current is low enough to be electrically safe, the involuntary reaction to a surprise discharge can cause injury.

9. Procedure

9.1 Using a 14-gage (1.63-mm) (or heavier) wire, connect the ground connection of the tester to the cleaned metal substrate of the equipment to be tested. A similar connection should be made to a suitable external ground.

9.2 Attach the probe cable to the tester and the desired probe electrode to the end of the probe handle.

9.3 Connect the input to a 115 to 120-V, 60-Hz source, or use a portable 5000-volt d-c unit.4

9.4 While holding the probe by the plastic handle move the switch to the on position. A red pilot light indicates that the tester is in operation.

9.5 Apply the test probe lightly to the surface of the glass coating in a sweeping motion to cover the whole area to be tested. If the interferences are not too great, a spark should also be seen and heard as it arcs between the electrode and the metal substrate.

9.6 Mark each area where a contact is found using suitably colored chalk or crayon.

9.7 Turn the switch off and ground the electrode to the metal ground before laying the probe down or changing the electrodes on the probe.

10. Report

10.1 The report shall include the following:

10.1.1 Date of test,

10.1.2 Description or identification of equipment being tested, and

10.1.3 Number and location of failures.

11. Precision and Bias

11.1 No justifiable statements can be made regarding the precision and bias of this test method because it is designed for application to full-size production vessels and reactors with the result that variables due to design, metal composition, fabrication, and metal processing, as well as porcelain enameling, are introduced into the results.

---

4 Suitable test equipment is available from: The Pfaudler Co., 1000 West Avenue, Rochester, NY 14603; Edlon Plastics, 6 Pheasant Run, Newton, Pa 18940; Buckley’s (Usral) Ltd., Beta Works, Range Road, Hysthe, Kent CT21 6HG, England, United Kingdom.
Standard Test Method for
Reliability of Glass Coatings on Glassed Steel Reaction
Equipment by High Voltage

This standard is issued under the fixed designation C 537; the number immediately following the designation indicates the year of
original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A
superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the reli-
ability of glass coating on metal and is intended for use by
manufacturers of equipment that is designed to withstand
highly corrosive conditions where a failure of the coating in
service would cause extreme damage to the exposed metal. Its
use outside the manufacturer’s plant is discouraged because
improper or indiscriminate testing can cause punctures that are
difficult to repair without returning the equipment to the
manufacturer’s plant. This test method detects not only exist-
ing discontinuities in the glass coating, but also areas where
the glass may be thin enough to be likely to result in premature
failure in service.

1.2 This standard does not purport to address all of the
safety concerns, if any, associated with its use. It is the
responsibility of the user of this standard to establish appro-
priate safety and health practices and determine the applica-

ability of regulatory limitations prior to use. For specific
precautionary statements, see Section 7.

2. Terminology

2.1 Definition:

2.1.1 glassed steel, glass-lined steel, or glass-coated steel—
designations generally applied to a class of porcelain enamels
that have high resistance to chemical attack at elevated
temperatures and pressures.

3. Summary of Test Method

3.1 This test method consists essentially of grounding the
metal structure of the equipment to the ground side of a
direct-current high-voltage generator and sweeping the glass
surface with a high-potential probe on the end of an insulated

handle and cable. Wherever a discontinuity exists or the
coating is thin enough (by reason of a concealed bubble or
conducting inclusion, etc.) so that the dielectric strength of the
remaining glass is less than some preset desired amount, the
dielectric strength of air-plus-remaining-glass breaks down and
a discharge occurs. Built-in current-limiting devices ensure
electrical safety to the operator. A variable voltage control
allows selection of a voltage which assures a predetermined
minimum thickness of glass.

4. Significance and Use

4.1 The widespread use of glassed-steel equipment in highly
corrosive chemical processes has made it necessary to detect
weak spots in the coating and repair them before catastrophic
failure occurs in service. This test is intended to detect
 discontinuities and thin areas in a glass coating on metal to
ensure that the coating is defect free and has sufficient
thickness to withstand the prescribed service conditions. A test
voltage may be selected at any desired value up to 20 000 V,
thus making the test applicable to a wide range of thickness
requirements. When, because of bubbles or defects, the thick-
ness of glass at any spot is less than enough to withstand the
applied voltage, a puncture results with an accompanying
indication of a defect. Remedial action is then required to
repair the defect before the equipment can be used for
corrosive service. (When such defects are found before the
equipment leaves the manufacturer’s plant another application
of glass can usually be applied and fired to become an integral
part of the coating.)

5. Interferences

5.1 Since the test method is electrical, it is necessary to have
a good ground connection between the instrument and the
metal substrate of the equipment being tested. It is also
necessary that the surface of the glass be reasonably clean and
dry. A wet surface will conduct enough of the high voltage to
any exposed metal, including the edges of the glass coating, so
that an indication of a “contact” may be obtained over a large area instead of at localized spots that can be marked and identified for repair.

6. Apparatus

6.1 The tester is composed of a source of high-voltage direct current with a suitable device to limit the current. A constant-voltage transformer is used to supply a more uniform voltage source than the usual 115-V, 60-Hz line to which it is connected. The power supply unit consists of a suitable step-up transformer along with a voltage-control device, a current-limiting rheostat, a rectifier, and appropriate safety and remote control relays, meters, as well as an insulated cable and probe (see Fig. 1 for schematic wiring diagram). The current output is limited to 2.5 mA. The output voltage is variable up to 20 000 V, and the level is indicated by a voltmeter. The handle is insulated and grounded and is designed to use either a wire brush-type or a point probe. The brush probe is used for sweeping larger surfaces of glass coating while the point probe is better adapted to interior corners and the more restricted areas.

7. Safety Precautions

7.1 The instrument and equipment being tested should be well grounded both to a good ground and to each other. All grounding contacts should be clean bare metal and not rusted or painted metal.

7.2 Handle the insulated probe handle so that the hand contacts the ground ring to prevent build-up of a static charge which causes an unpleasant (although not dangerous) sensation on discharge.

7.3 Keep the probe electrode at least 305 mm (12 in.) away from conducting surfaces or personnel. Remember that conducting surfaces may lead to personnel at some distance from the probe. Discharge the probe tip by grounding it after turning off the instrument and before changing probe tips. Although the current is low enough to be electrically safe, the involuntary reaction from a surprise discharge might cause injury.

7.4 Unless the surface to be tested is clean and dry, there may be sufficient conduction along the surface to cause a capacitance discharge even if there is no direct path to ground. Such a capacitance discharge is recognizable from a true failure because the discharge spark is not confined to certain spots but is a general discharge to a large area of the moist glass surface. Continuous application of the probe to such areas serves only to build up a capacitance charge on the surface of the vessel eventually resulting in a discharge through the operator to his discomfort.

7.5 A d-c tester should never be used in a chemical plant for discontinuity testing because of the possibility that a capacitive charge will be developed in the dielectric coating, resulting in an explosion hazard.

8. Procedure

8.1 The following procedure is applicable to commercially available test equipment:

8.1.1 Install ground connections to the instrument and to the equipment to be tested. Provide a separate ground connection between the instrument and the equipment.

8.1.2 Set the voltage-control knob near the minimum setting, connect the instrument to the voltage stabilizer, and connect the latter to the appropriate power line. Turn the power on and allow the test equipment to warm up for 2 to 3 min to ensure stabilization of the voltage.

8.1.3 Engage the overload relay and then the “DC On” switch to apply voltage to the probe.

8.1.4 Hold the probe by the handle with a firm grip on the ground ring and with the electrode well away from all objects and personnel, and slowly raise the voltage until the voltmeter indicates the desired value. Maintain hand contact with the ground ring of the handle all through the test to avoid a buildup
of static electricity in the operator that might result in an uncomfortable and annoying (although not dangerous) spark discharge. Pass the test probe across the coating surface at a speed not to exceed 40 cm/s.

Note 1—The instrument is set to discharge across an internal gap at slightly over 20 000 V. If the voltage is set high enough to cause such a discharge, ionization of the air usually causes the arc to persist until the voltage is reduced to approximately 5000 V. Shortly after the arc has extinguished, the voltage can be reset to the desired value.

8.1.5 When a point of failure in the coating is approached, a spark will jump to the point of failure with a visible and audible discharge or arc. As the path of the arc is shortened, by bringing the electrode close to the point of failure, the current in the arc builds up. At a 2.5-mA current in the arc, the overload relay will trip and the d-c current will cut off. When this happens it is necessary to engage first the overload relay and then the “DC On” switch before the tester is again operative.

8.1.6 Mark the point of failure with suitable chalk (or crayon) for subsequent repair.

9. Report
9.1 The report shall include the following:
9.1.1 Title of test, ASTM designation and issue,
9.1.2 Date of test,
9.1.3 Description or identification of equipment being tested,
9.1.4 Voltage used in the test, and
9.1.5 Number and location of failures.

10. Precision and Bias
10.1 No justifiable statements can be made regarding the precision and bias of this test method due to the fact that this method is designed for application to full-sized production vessels and reactors with the result that variables due to design, metal composition, fabrication, and metal processing, as well as porcelain enameling, are introduced into the results.
Standard Test Method for
Color Retention of Red, Orange, and Yellow Porcelain Enamels

This standard is issued under the fixed designation C 538; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method\(^2\) covers only porcelain enamels containing the cadmium-sulfide or cadmium sulfo-selenide types of pigments. The purpose of the test is to detect those red, yellow, orange, and some green porcelain enamels that are likely to experience appreciable color changes upon exposure to atmospheric corrosion.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Summary of Test Method

2.1 Cupric sulfate solution is placed on the surface of appropriate samples and allowed to remain for 20 h under controlled illumination. The samples are then washed, dried, and examined visually for a darkening of the color in the tested area.

3. Significance and Use

3.1 Correlation between results of this test and outdoor exposure tests of porcelain enamels colored with cadmium sulfo-selenide pigment shows that the cupric sulfate test is an effective indicator of the tendency for such coatings to experience appreciable color changes during weathering. The cupric sulfate test is considered useful for specification acceptance, quality control, and research and development purposes.

4. Interferences

4.1 In some cases, the treatment may change the gloss of the tested area. Any such gloss change shall be disregarded.

5. Apparatus for Control of Illumination

5.1 The light source shall consist of a 110-V, 15-W cool white fluorescent lamp (designation F 15 T8CW), 25.4 mm (1 in.) in diameter by 457.2 mm (18 in.) long. Center the lamp near the top of a box, made from plywood or other suitable material, in such a position that the distance between the specimen surface and the centerline of the lamp is 228.6 mm (9 in.). The box, with inside dimensions of 508 by 152 by 305 mm (20 by 6 by 12 in.) (length, width, height), shall be painted with a matte (flat), neutral white paint on the inside, and shall be vented for air circulation. Venting is accomplished by 11 holes, 12.7 mm (1/2 in.) in diameter, drilled 25.4 mm (1 in.) apart on a centerline 25.4 mm (1 in.) from the inside top, and 11 similar holes drilled on a centerline 25.4 mm (1 in.) from the bottom on each of the two long sides.

6. Reagents

6.1 The cupric sulfate solution is prepared by placing 50 g of cupric sulfate \((\text{CuSO}_4 \cdot 5\text{H}_2\text{O})\), reagent grade, in 100 mL of hot distilled water in a glass container, and permitting the mixture to age at room temperature \((27 ± 5^\circ\text{C} (80 ± 10^\circ\text{F}))\) for 16 h or longer with occasional stirring. This treatment produces a solution with some undissolved material remaining at the bottom of the container.

7. Specimens

7.1 Any size specimen larger than 50.8 by 50.8 mm (2 by 2 in.) may be used. Specimens should be reasonably flat.

8. Procedure

8.1 Clean the specimen by washing with soap and water, followed by copious rinsing with clean, but not necessarily distilled, water. Dry the surface with a clean dry towel by blotting or gentle rubbing.

8.2 Place several drops of the solution on the horizontal test area to form a pool, and immediately cover with a clean 25-mm (1-in.) diameter watch glass (with fire-polished edges) in the inverted position. The quantity of solution used shall be sufficient to fill the inverted watch glass except for a small bubble of entrapped air. Remove any excess solution from the test area not under the watch glass. In some cases, it may be necessary to seal the area of contact between the watch glass and the specimen surface (with some material such as modeling clay or rubber cement) to prevent escape of the solution.

8.3 Place the specimen with watch glass in position under the light-chamber. It should remain in this position for 20 h in a room at \(27 ± 5^\circ\text{C} (80 ± 10^\circ\text{F})\). If the solution has escaped or dried out at the end of the test period, the test is invalid and

\(^{1}\) This test method is under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.12 on Materials for Porcelain Enamel and Ceramic-Metal Systems.


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shall be repeated on a different location on the specimen surface.

8.4 After exposure, remove the watch glass, rinse the treated area with water to remove all of the test solution, and dry with a clean cloth.

8.5 Visually examine the specimen immediately to determine whether there has been any darkening of the treated area. Use partially diffused daylight supplemented, if necessary, by artificial light, the total intensity being approximately that available within a few feet of an outside window, but do not examine in direct sunlight. During observation, hold the specimen no nearer the light source (such as a window) than the minimum width of the source.

8.6 If any darkening can be detected visually, the specimen fails the test. Changes in gloss caused by the treatment shall not be considered in the grading.

9. Report

9.1 Report the type, color, and number of specimens tested, and whether each passed or failed the test.

10. Precision and Bias

10.1 A precision and bias statement is not applicable to this test method. Since it is a pass/fail test.
Standard Test Method for
Linear Thermal Expansion of Porcelain Enamel and Glaze
Frits and Ceramic Whiteware Materials by Interferometric
Method

This standard is issued under the fixed designation C 539; the number immediately following the designation indicates the year
of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A
superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the interferometric determination of linear thermal expansion of premelted frits (porcelain
enamel and glaze) and fired ceramic whiteware materials at temperatures lower than 1000°C (1830°F).

1.2 This standard does not purport to address all of the
safety concerns, if any, associated with its use. It is the
responsibility of the user of this standard to establish appro-
priate safety and health practices and determine the applica-
tility of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
E 289 Test Method for Linear Thermal Expansion of Rigid
Solids with Interferometry

3. Significance and Use

3.1 This test method defines the thermal expansion of
porcelain enamel and glaze frits by the interferometric method.
This determination is critical in avoiding crazing (cracking) of
these glass coatings due to mismatching of the thermal
expansion between the coating and substrate materials.

4. Apparatus

4.1 Sample Preparation Equipment:
4.1.1 Glazed Porcelain Crucible, No. 0.
4.1.2 Fireclay Crucible, 102 mm (4 in.) in diameter.
4.1.3 Rotating Abrasive Grinding Wheel (a silicon carbide
type is satisfactory).
4.2 Micrometer Calipers, having a sensitivity such that the
index can be read to 0.002 mm (0.0001 in.).

4.3 Measuring Apparatus, consisting of fused silica inter-
ferometer plates, viewing apparatus, an electric furnace and
control, potentiometer, pyrometer, and a suitable monochro-
matic light source of known wavelength.

4.3.1 Furnace—The furnace shall be a vertical electric tube
furnace controlled by rheostat or other means so the heating
rate of the furnace can be readily duplicated from room
temperature to 1000°C (1830°F). The heating rate shall not
exceed 3°C (5.5°F)/min.

4.3.2 Temperature Measuring Instrument—A calibrated
platinum versus platinum-rhodium thermocouple (or a
Chromel versus Alumel thermocouple if it is frequently cali-
brated) in conjunction with a potentiometer shall be used. The
potentiometer shall be capable of being read to 2°C (4°F) and
shall have automatic compensation for the temperature of the
reference junction, or the reference junction shall be held at
0°C (32°F) by means of an ice bath.

5. Test Specimens

5.1 For frit samples, three test specimens shall be prepared
as follows:

5.1.1 Fill a No. 0 glazed porcelain crucible with frit, place
the filled crucible inside a 102-mm (4-in.) diameter fireclay
crucible partly filled with silica, and work the small crucible
down into the silica until approximately 75% of the small
crucible is below the level of the silica.

5.1.2 Place the crucible assembly into a furnace at a
temperature high enough to just melt the mass. Hold for 15 min
after the frit has reached the furnace temperature.

5.1.3 Remove the crucible, rapidly transfer it to another
furnace that is at the frit firing temperature, and cool in the
furnace at a rate not to exceed 60°C (110°F)/h.

5.1.4 Break the small crucible open and break up the
vitreous mass. Select six fragments from the interior of the
mass (to avoid side portions diluted by the ceramic crucible)
having minimum conical dimensions of 3 mm (⅛ in.) at the
base and 6 mm (¼ in.) high.

5.2 For fired samples, break and select six samples having
minimum conical dimensions of 3 mm (⅛ in.) at the base and
6 mm (¼ in.) in height. For all samples, grind the base of the
flat cones and cement the flat cone base to the flat end of a glass
rod with heated sealing wax. Grind the piece to a finished cone by rotating the rod while the piece is held against a rotating abrasive wheel (a silicon carbide type is satisfactory).

5.2.1 When a reasonably symmetrical cone with a rounded tip is obtained, remove it from the rod by heating the wax or by pressure with the fingertips. Remove all sealing wax with a knife blade or abrasive paper.

5.2.2 The test cone height may be of the order of 4.8 mm (3/16 in.). These bases must be smooth and flat. Use No. 0 metallurgical paper to approach the desired figure and then use successively finer papers until the final reduction is made with a No. 3/0 paper.

6. Calibration of Furnace\textsuperscript{4}

6.1 Using the following procedure, calibrate the furnace controls to obtain a heating rate of 3°C (5.5°F)/min:

6.1.1 Prepare three conical spacers closely approximating the dimensions of the final test pieces described in Section 5. These spacers shall be ground from fragments of refractory ceramic known to have a softening temperature in excess of 1000°C (1830°F).

6.1.2 Assemble the upper and lower interferometer plates with three refractory spacers as described in Section 7, except fringe development is not necessary. Place this assembly in the furnace test location. Center the hot junction of an 18 or 20-gage thermocouple within the triangle formed by the spacers. It will usually be necessary to extend the thermocouple out through the top of the furnace tube. This thermocouple temperature measurement equipment shall meet the requirements in 4.3.2.

6.1.3 The output of this thermocouple shall be used to establish corrections required in calibrating the furnace temperature measuring system. Both temperature values and heating rates shall be so corrected if differences exist.

7. Procedure

7.1 Assemble (outside the furnace) the three test pieces prepared as described in Section 5 between the two interferometer plates as follows:

7.1.1 Place the plate with the one frosted side down within the refractory specimen crucible.

7.1.2 Place the three test pieces on this plate in an equilateral triangle.

7.1.3 Lower the clear plate onto the test pieces keeping the mark or notch identifying the wedge side in the up position.

7.1.4 Set this assembly at a height comparable to that used inside the furnace.

7.2 Rotate the telescope and center it over the test specimen assembly. Direct the monochromatic light source down the tube. If four to eight fringes are present, the setup is correct. If fewer or more fringes are present, adjust the cone heights. In some cases, mere tapping of the specimen assembly will produce the correct number of fringes. Carefully measure and record the height of each cone. Upon achieving the proper number of fringes, place the refractory ring cover on the crucible and recheck for fringes.

7.3 Without rotating the crucible, gently lower it into the furnace and onto the bottom support so the thermocouple rests at the bottom of the crucible. Cover the top of the furnace with a quartz plate.

7.4 Rotate the telescope and check the fringe pattern. If excessive glare or poor contrast are present, adjust by moving the quartz cover, moving the light source, or releveling the telescope.

NOTE 1—Removal of the telescope eyepiece should reveal a bright dot, which is the true image. This must be in the field or no fringes will be seen. If this bright dot of the true image is not seen when the eyepiece is removed, a great deal of trial and error adjustment of the telescope tripod must be made. A number of false images may also be present. These must be sorted out by inserting the eyepiece and checking to see if fringes are present. If no fringes are seen, the bright dot is a false image.

7.5 Standardize the potentiometer if necessary and set the potentiometer or other temperature measuring instrument to 38°C (100°F).

7.6 Slowly heat the furnace to 38°C (100°F). Center the cross hair of the telescope upon any convenient fringe and record the temperature corresponding to each fifth fringe.

7.7 Continue heating the furnace to maintain a 3°C (5.5°F)/min temperature rise or less. Below 100°C a heating rate not exceeding 1.5°C/min is preferred. For frit samples, when the softening temperature has been reached, as shown by the fringes retreating for at least one fringe, immediately turn off the furnace to avoid reaction with the quartz plates.

8. Calculations

8.1 Calculate the percentage of linear thermal expansion for each reading as follows:

\[
L = (n\lambda/200h) + A_c
\]  

where:

\[L\] = linear thermal expansion, % from starting temperature, \(ti\)°C, to temperature of observation, \(t\)°C,

\[n\] = number of fringes passing the reference point during the change from temperature \(ti\) to temperature \(t\),

\[\lambda\] = wavelength of the light source, \(\mu\)m,

\[h\] = height of the specimen at temperature \(ti\), cm, and

\[A_c\] = air correction from temperature \(ti\) to temperature \(t\), %

(see Table 1).

8.2 Prepare a curve by plotting each temperature reading, \(t\), on the horizontal axis against the corresponding percentage expansion along the vertical axis.

8.3 Calculate the mean coefficient of thermal expansion, \(E\), for any temperature range, \(t_2\) to \(t_3\)°C, within the limits of the test, as follows:

\[
E = L'/(100(t_3 - t_2)]
\]  

where:

\[L'\] = linear thermal expansion, from temperature \(t_2\)°C to temperature \(t_3\)°C as determined from the curve prepared in accordance with 7.2, %,

\[t_2\] = lower temperature in range \(t_2\) to \(t_3\), and

\[t_3\] = higher temperature in range \(t_2\) to \(t_3\).

9. Report

9.1 Report the following:
9.1.1 Designation of material tested,
9.1.2 Method of preparation of test specimen, cooling rate, etc.,
9.1.3 Identification of type of apparatus used,
9.1.4 Data sheet showing:
9.1.4.1 Form and height of test specimens,
9.1.4.2 Wavelength of light source,
9.1.4.3 Starting temperature,
9.1.4.4 Corrected temperature at each reading,
9.1.4.5 Number of fringes, \( n \), at each reading,
9.1.4.6 Calculation, \( n\lambda/200h \), for each reading,
9.1.4.7 Air correction, \( A_c \), for each reading,
9.1.4.8 Percentage of expansion, \( L \), computed for each reading,
9.1.5 The curve (see 8.2) showing temperature plotted against percentage of expansion, and
9.1.6 Mean coefficient of linear thermal expansion per degree Celsius over the desired temperature ranges.

10. Precision and Bias

10.1 The precision and bias of this test method of measuring the linear thermal expansion of porcelain enamel and glaze frits are as specified in Test Method E 289.

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*A If the starting temperature, \( t_0 \), is above 20°C, the entry in the table opposite that of the starting temperature should be subtracted from each of the succeeding corrections.

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Standard Test Method for Alkali Resistance of Porcelain Enamels

This standard is issued under the fixed designation C 614; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of the resistance of a porcelain enamel to a hot solution of tetrasodium pyrophosphate. Although the specific alkali mentioned herein is tetrasodium pyrophosphate the equipment and techniques are equally applicable to other alkali solutions.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Significance and Use

2.1 This test method is intended specifically for testing the resistance to alkaline attack of porcelain enamels to be used as a final finish on washing machines, dishwashers, combination washer-dryers, and similar appliances where the surface is normally exposed to an alkaline environment at elevated temperatures.

3. Apparatus

3.1 Test Chamber, of a type illustrated in Fig. 1, Fig. 3, and Fig. 6. This part of the equipment consists of a stainless steel beaker fitted with an O-ring sealed cover which supports an immersion heater, a stirring device, thermometer and thermistor wells; three specimen holders; a reflux condenser; a flow channeling shield to slide over and be supported by the immersion heater; and a stirring motor.

3.2 Temperature Controller, thermistor-actuated, capable of maintaining the temperature of the alkali solution in the beaker at 96.00 ± 0.20°C.

3.3 Thermometer, calibrated, approximately 15 in. (380 mm) long and ½ in. (7.1 mm) in diameter, with 0.1° divisions.

3.4 Drying Oven, electrically heated.

3.5 Balance, having a sensitivity of 0.1 mg and a capacity of not less than 200 g.

3.6 Desiccator.

3.7 Hot Plate, or burner suitable for heating the test solution.

3.8 Sponge, soft, cellulose.

3.9 Aluminum Alloy Sheets, approximately 3½ by 3½ by ⅛ in. (89 by 89 by 3.1 mm).

4. Reagents and Materials

4.1 Tetrasodium Pyrophosphate, hydrated (Na₄P₂O₇·10H₂O), recrystallized, reagent grade.

4.2 Trisodium Phosphate (Na₃PO₄·12H₂O).

5. Test Specimens

5.1 Preparation of Specimens—The test specimens shall be 3½ in. (89 mm) square and may be prepared by enameling metal blanks of that size or by cutting from a larger piece. Hanging holes, if necessary for firing or weighing or both, may be placed at the corners. The specimens shall be sufficiently flat to permit sealing in the holders. The edges of cut specimens shall be stoned or filed to remove any sharp or loosely adhering fragments of metal or porcelain enamel. It is recommended that the specimens be permanently marked on the back side for identification.

5.2 Number of Specimens—Six specimens shall be tested simultaneously.

5.3 Cleaning of Specimens Before Alkali Exposure—The specimens shall be rinsed with running tap water and washed with a soft cellulose sponge dampened with a 1 % solution of trisodium phosphate. A light pressure and a back-and-forth motion in two directions, 90° apart, shall be used in the washing. After washing, the specimens shall be rinsed first with running tap water, next with distilled water and then allowed to drain in a near vertical position prior to oven-drying at 110°C for 15 min. The specimens shall be cooled in a desiccator for 30 min prior to weighing.

5.4 Weighing of Test Specimens—The specimens shall be weighed immediately upon removal from the desiccator. Weights shall be recorded to 0.0001 g. The weighed specimens shall be stored in a desiccator until tested.
6. Procedure

6.1 Preparation of Test Solution—Prepare the test solution by adding 260 g of tetrasodium pyrophosphate to 4.940 litres of distilled water. Pour the entire amount of prepared solution into the beaker and cover the rectangular slots with lids. Use a freshly prepared solution for each set of six specimens.

6.2 Preheating Test Solution—Place the temperature controller and stirrer in operation and preheat the solution to 96°C. Heating may be expedited by placing the beaker on a hot plate or over a gas burner. When a temperature near 96°C has been reached, remove the beaker from the hot plate and place on an insulated surface. Allow an equilibration period of 15 min for all parts of the equipment to achieve a stable temperature, and adjustment of the control point to yield the desired temperature.

6.3 Preparation for Alkali Exposure—Place two clean, weighed specimens in each specimen-holder box. Shims may be useful in the bottom of the specimen boxes to center the specimens vertically about the exposure openings. Back-up each specimen with a stress distributing aluminum alloy backing sheet and insert the clamping device between the two sheets. Tighten the clamping device only enough to give a leakproof seal. Test the seal by filling the specimen box with water to confirm the absence of leaks. Next, remove the water and preheat the specimen holder assembly in an oven at 110°C for about 15 min.

6.4 Exposure of Specimens—When the test solution has reached 96°C and is under control, remove the lids and insert preheated specimen holder assemblies in the proper openings.

Expose six specimens for 6 h at 96 ± 0.20°C. The exposure time shall start upon insertion of the specimen holders and shall end with their withdrawal. After removal from the holders clean, dry and weigh the specimens as specified in 5.3 and 5.4.

6.5 Measurement of Exposed Area—Calculate the area of the etched portion of the specimen using the average of two diameters, approximately 90° apart, which have been measured to the nearest 0.01 in. (0.25 mm).

7. Calculation

7.1 Calculate the weight loss for each specimen as follows:

\[ L, \text{mg/in.}^2 = \frac{[(B - C) \times 10^{-3}]}{D} \]

or

\[ L, \text{mg/cm}^2 = \frac{[(B - C) \times 10^{-1}]}{E} \]

where:

- \( L \) = weight loss,
- \( B \) = original weight of specimen, g,
- \( C \) = final weight of specimen, g,
- \( D \) = area of etched attack, in.\(^2\)(0.155 \times cm\(^2\)),
- \( E \) = area of etched attack, cm\(^2\) (6.45 \times \text{in.}^2).

8. Treatment of Data

8.1 Single Determination—Six specimens shall constitute a sample. The average of six values of weight loss (\( L \)), shall be termed the mean weight loss, \( L \), for the sample. The statistical error of the determination shall be computed by the use of the equation:

\[ e = 1.15 \sigma \]
where:
\( e \) = the statistical error of the mean value for the sample, at the 95 % confidence level, and
\( \sigma \) = the standard deviation of the six individual weight losses from the average of all six weights.

**Note 1**—The factor 1.15 is applied only when the number of specimens is six, and the confidence level is 95 %.

8.1.1 This standard deviation shall be computed from the generalized equation:

\[
\sigma = \sqrt{\frac{\Sigma X^2}{n}} - \bar{X}^2 \tag{4}
\]

or if the number of specimens, \( n \), is six, from:

\[
\sigma = \sqrt{\frac{\Sigma X^2}{6}} - \bar{X}^2 \tag{5}
\]

where:
\( \Sigma X^2 \) = sum of the squares of the weight loss values of the six individual specimens, and
\( \bar{X}^2 \) = the square of the mean weight loss of the six specimens.

8.1.2 Illustrative calculations are given below. The recommended number of decimal places is given in each case:

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>( L ), mg/in.²</th>
<th>( L^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>7.7</td>
<td>59.29</td>
</tr>
<tr>
<td>2</td>
<td>7.5</td>
<td>56.25</td>
</tr>
<tr>
<td>3</td>
<td>6.9</td>
<td>47.81</td>
</tr>
<tr>
<td>4</td>
<td>8.0</td>
<td>64.00</td>
</tr>
<tr>
<td>5</td>
<td>7.7</td>
<td>59.29</td>
</tr>
<tr>
<td>6</td>
<td>7.9</td>
<td>62.41</td>
</tr>
<tr>
<td>Sum</td>
<td>45.7</td>
<td>348.85</td>
</tr>
<tr>
<td>Mean</td>
<td>7.6167</td>
<td>58.1417</td>
</tr>
</tbody>
</table>
from Eq 3:
\[ \sigma = \sqrt{58.1417 - (7.6167)^2} = \sqrt{0.1276} = 0.357 \]  
(6)

from Eq 1:
\[ e = 1.15 \times 0.357 = 0.411 \]  
(7)

The weight loss \( L \) is reported as \( 7.6 \pm 0.4 \) mg/in\(^2\).

8.2 Difference Between Two Determinations—The significance of a difference between two mean values shall be tested through the use of the ratio \( d:e' \)

where:
\( d \) = the difference in means, and
\( e' \) = the statistical error in the determination of \( d \).

8.2.1 The statistical error, \( e' \), of the difference, \( d \), between two means shall be determined from the following equation:
\[ e' = \sqrt{(e_1)^2 + (e_2)^2} \]  
(8)

where:
\( e' \) = the error of the difference in means,
\( e_1 \) = the error of one mean value, and
\( e_2 \) = the error of the other mean value.

8.2.2 An example to illustrate the use of Eq 4 is given below:
Assume:
\( L_1 = 7.6 \pm 0.5 \),
\( L_2 = 8.2 \pm 0.3 \), and
\( d = 0.6 \).

Then:
\[ e' = \sqrt{(0.5)^2 + (0.3)^2} = \sqrt{0.34} = 0.58 \]  
\( d:e' = 0.6/0.58 = 1.03 \)  
(9)

When \( d:e' \) is equal to or greater than 1.00, the difference is significant at the 95 % confidence level.

8.2.3 The following table shows the relation between values of \( d:e' \) and the confidence level, for samples of six specimens.

<table>
<thead>
<tr>
<th>( d:e' )</th>
<th>Confidence</th>
<th>Interpretation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Over 1.50</td>
<td>99 or higher</td>
<td>highly significant</td>
</tr>
<tr>
<td>Over 1.00 but less than 1.50</td>
<td>95 to 99</td>
<td>significant</td>
</tr>
<tr>
<td>Over 0.80 but less than 1.00</td>
<td>90 to 95</td>
<td>indicative</td>
</tr>
</tbody>
</table>

The basing of conclusions on differences in which less than 90 % confidence can be placed is not recommended.

9. Precision and Bias

9.1 The precision and bias of this method are being developed.
FIG. 4 Alkali Test Equipment
### Dimension Table

<table>
<thead>
<tr>
<th>Dimension</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>in.</td>
<td>4½</td>
</tr>
<tr>
<td>B</td>
<td>in.</td>
<td>3%</td>
</tr>
<tr>
<td>C</td>
<td>in.</td>
<td>1/?4</td>
</tr>
<tr>
<td>D</td>
<td>in.</td>
<td>1½</td>
</tr>
<tr>
<td>E</td>
<td>in.</td>
<td>1%</td>
</tr>
<tr>
<td>F</td>
<td>in.</td>
<td>0.079</td>
</tr>
<tr>
<td>G</td>
<td>in.</td>
<td>¼</td>
</tr>
<tr>
<td>H</td>
<td>in.</td>
<td>6</td>
</tr>
<tr>
<td>I</td>
<td>in.</td>
<td>8</td>
</tr>
<tr>
<td>J</td>
<td>in.</td>
<td>¾</td>
</tr>
<tr>
<td>K</td>
<td>in.</td>
<td>½</td>
</tr>
<tr>
<td>L</td>
<td>in.</td>
<td>2½</td>
</tr>
<tr>
<td>M</td>
<td>in.</td>
<td>2½</td>
</tr>
<tr>
<td>N</td>
<td>in.</td>
<td>3</td>
</tr>
<tr>
<td>P</td>
<td>in.</td>
<td>0.060</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Dimension</th>
<th>Unit</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>mm</td>
<td>108</td>
</tr>
<tr>
<td>B</td>
<td>mm</td>
<td>95</td>
</tr>
<tr>
<td>C</td>
<td>mm</td>
<td>6.7</td>
</tr>
<tr>
<td>D</td>
<td>mm</td>
<td>34.0</td>
</tr>
<tr>
<td>E</td>
<td>mm</td>
<td>47.5</td>
</tr>
<tr>
<td>F</td>
<td>mm</td>
<td>2.01</td>
</tr>
<tr>
<td>G</td>
<td>mm</td>
<td>6.4</td>
</tr>
<tr>
<td>H</td>
<td>mm</td>
<td>152</td>
</tr>
<tr>
<td>I</td>
<td>mm</td>
<td>203</td>
</tr>
<tr>
<td>J</td>
<td>mm</td>
<td>2.40</td>
</tr>
<tr>
<td>K</td>
<td>mm</td>
<td>3.18</td>
</tr>
<tr>
<td>L</td>
<td>mm</td>
<td>64</td>
</tr>
<tr>
<td>M</td>
<td>mm</td>
<td>70</td>
</tr>
<tr>
<td>N</td>
<td>mm</td>
<td>76</td>
</tr>
<tr>
<td>P</td>
<td>mm</td>
<td>1.52</td>
</tr>
</tbody>
</table>

**Note 1**—Box welded from 14-gage (0.079 in.) stainless steel.

**Fig. 5 Detail Specimen Holder**
A—Showing a specimen.
B—The back-up plate.
C—O-ring interior.
D—Clamping plate (partly withdrawn).

**FIG. 6 Specimen Holder Assembly**

<table>
<thead>
<tr>
<th>Dimension</th>
<th>in.</th>
<th>mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>3¼</td>
<td>83</td>
</tr>
<tr>
<td>B</td>
<td>1⅛</td>
<td>28.5</td>
</tr>
<tr>
<td>C</td>
<td>⅛</td>
<td>3.18</td>
</tr>
<tr>
<td>D</td>
<td>⅛</td>
<td>6.4</td>
</tr>
<tr>
<td>E</td>
<td>11/32</td>
<td>17.5</td>
</tr>
<tr>
<td>F</td>
<td>⅛</td>
<td>6.4</td>
</tr>
<tr>
<td>G</td>
<td>⅛</td>
<td>9.5</td>
</tr>
<tr>
<td>H</td>
<td>⅛</td>
<td>19.0</td>
</tr>
<tr>
<td>I</td>
<td>⅛</td>
<td>3.18</td>
</tr>
<tr>
<td>J</td>
<td>3⅜</td>
<td>87</td>
</tr>
<tr>
<td>K</td>
<td>¾</td>
<td>19.0</td>
</tr>
<tr>
<td>L</td>
<td>⅛</td>
<td>7.9</td>
</tr>
<tr>
<td>M</td>
<td>1⅝</td>
<td>41.5</td>
</tr>
<tr>
<td>N</td>
<td>⅛</td>
<td>12.7</td>
</tr>
<tr>
<td>O</td>
<td>⅛</td>
<td>67</td>
</tr>
<tr>
<td>P</td>
<td>⅛</td>
<td>6.4</td>
</tr>
<tr>
<td>Q</td>
<td>7⅛</td>
<td>181</td>
</tr>
<tr>
<td>R</td>
<td>0.060</td>
<td>1.52</td>
</tr>
<tr>
<td>S</td>
<td>0.003</td>
<td>0.076</td>
</tr>
<tr>
<td>T</td>
<td>6⅛</td>
<td>159</td>
</tr>
</tbody>
</table>

**FIG. 7 Stainless Steel Specimen Backup Plate**
Standard Test Method for Reboiling Tendency of Sheet Steel for Porcelain Enameling

This standard is issued under the fixed designation C 632; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of whether a sheet steel for porcelain enameling will reboil under a given set of worse case conditions.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   C 286 Terminology Relating to Porcelain Enamel and Ceramic-Metal Systems

3. Terminology

3.1 Definitions:
   3.1.1 ground coat—a porcelain enamel applied directly to the base metal to function as an intermediate layer between the metal and the cover coat (see Terminology C 286).
   3.1.2 reboiling—gas evolution occurring and recurring during repeated firing of the ground coat, sometimes a defect (see Terminology C 286).
   3.1.3 slip (slurry)—a suspension of finely divided material in liquid (see Terminology C 286).

3.2 Description of a Term Specific to This Standard:
   3.2.1 quarter lines—imaginary lines parallel to the direction of rolling positioned at a distance from the sheet mill edge, equal to one quarter of the sheet width.

4. Summary of Test Method

4.1 Both surfaces of sheet steel sample plates are subjected to a cleaning treatment and are then porcelain enameled with a sheet-steel, ground-coat type finish. Subsequent reheating at various times is performed to reveal whether or not the steel will cause reboiling. The presence or absence of reboiling is determined by visual inspection.

5. Significance and Use

5.1 The results of this test indicate whether or not a sheet steel will cause reboiling under one given set of enameling conditions and enable the enamel processor to establish a possible source of imperfections in the finished coating.

5.2 This test method is normally used to test samples from lots, coils, or lifts of sheet steel which for some reason, production problems, defects evident on enameled parts, or production experience are suspected of causing defects in enameled ware which have the appearance of reboiling. It is often used to establish whether a problem exists with the bare steel or other factors such as the porcelain enamel, processing contamination, and so forth.

6. Apparatus

6.1 This test can be conducted in any laboratory equipped for general porcelain enameling. It does not require facilities for metal surface preparation.

7. Reagents and Materials

7.1 Steel Sheet, sufficient in size to provide samples as described in 8.1 and 8.1.1.

7.2 Porcelain-Enamel, Ground-Coat Slip, obtained from production milling or prepared in laboratory facilities.

7.3 Acetone or Isopropyl Alcohol, reagent grade.

8. Sampling

8.1 Shear sufficient material from within the quarter lines of a selected steel sheet sample from a sheet, coil, or lift to be tested to provide a minimum of five panels, each at least 2 in. (50 mm) square but not greater than 4 by 6 in. (100 by 150 mm).

8.1.1 Choose panels from rust-free areas that do not contain the mill identification stamp.

8.1.2 Identify each panel by a method that enables identity to be monitored after repeated enamel firings. Steel die stamping is the most commonly used technique.

8.1.3 Material sheared from the sheet or coil may be within the range of sizes given in 8.1 or may be sheared into specimens of these sizes after application and firing of the ground-coat enamel.
9. Procedure

9.1 Swab the cut-to-size steel panels with cheesecloth soaked with a solvent such as acetone or isopropyl alcohol.

9.1.1 Scrub the panels by hand with a portion of the ground-coat enamel. Remove the excess enamel from the panels by flushing with water if the panels are to be coated by spraying. Dry the panels thoroughly.

9.1.2 Avoid contact with soils of any nature and store the panels in a desiccator until they are ready for enameling.

9.2 Apply 24 g/ft² (2.6 g/dm²) of enamel slip to each surface (Note 1). Either dipping or spraying may be used, depending upon the method for which the slip is designed. Dry the surfaces adequately. Fire them at a temperature and for sufficient time to fuse the ground-coat enamel to the base metal. This time and temperature should be that recommended by the porcelain enamel manufacturers for optimum adherence (maturing temperature).

Note 1—Thin fired coatings of 2 1/2 to 3 mils (0.06 to 0.08 mm) are desirable so that reboil bubbles can be readily observed.

9.3 Store the fired panels at room temperature for at least 16 h, but not more than 48 h, prior to conducting the reboiling test.

9.4 For determining reboiling, reheat the first panel for a time which is the median firing time expected in part production or that recommended by the frit supplier for optimum enamel properties. If no reboiling occurs, increase firing time for subsequent panels 5 s longer than the previous panel. Firing time should be increased to the point that reboiling occurs or the maximum time expected under worse case production conditions is met. Of course, one may determine maximum time for reboil by decreasing firing time sequentially. A minimum of five panels or a time spread of 25 s is to be used.

9.4.1 The reboiling furnace should be operated at about 50 ± 10°F (27 ± 5°C) below the maturing temperature of the ground-coat enamel.

9.4.2 At the end of the reheat time allotted for each panel, remove the panel from the furnace and air cool it to room temperature.

10. Inspection

10.1 Visually examine the series of panels for evidence of reboiling. The presence of reboiling on any of the steel panels is considered a failure.

10.2 Reboiling is characterized by the emergence of bubbles on the enamel surface. These bubbles rise and subside rapidly and may persist as areas of blistering if arrested in a state of development by removal of the sample from the furnace and cooling to room temperature. The typical appearance of reboiling is shown in Fig. 1.

11. Precision and Bias

11.1 No statement is made about either the precision or bias of this test method for measuring reboiling of sheet steel for porcelain enameling since the result merely states whether there is conformance to the criteria for success specified in the procedure.

FIG. 1 Reboiling of Porcelain Enamel on Steel
Standard Test Method for Adhesion or Cohesion Strength of Thermal Spray Coatings

This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.12 on Materials for Porcelain Enamel and Ceramic-Metal Systems.


1. Scope

1.1 This test method covers the determination of the degree of adhesion (bonding strength) of a coating to a substrate or the cohesion strength of the coating in a tension normal to the surface. The test consists of coating one face of a substrate fixture, bonding this coating to the face of a loading fixture, and subjecting this assembly of coating and fixtures to a tensile load normal to the plane of the coating. It is adapted particularly for testing coatings applied by thermal spray, which is defined to include the combustion flame, plasma arc, two-wire arc, high-velocity oxygen fuel, and detonation processes for spraying feedstock, which may be in the form of, wire, rod, or powder.

Note 1—Thermal spray coating materials include ceramics, such as metal oxides or carbides, and metals. In some cases, a coating is formed of different spray materials, such as an oxide layer sprayed onto a sprayed metal-bonding layer. The substrate generally is a metal, but may be a ceramic, such as an oxide or graphite.

1.2 Usually this test method is performed at ambient temperature. Higher temperature testing is restricted by the need for a suitable adhesive bonding agent. For certain fundamental investigations, it is suggested that very low (cryogenic) temperature be used.

1.3 This test method is limited to testing thermal spray coatings that can be applied in thickness greater than 0.015 in. (0.38 mm). The limitation is imposed because an adhesive bonding agent is used in the test. Those bonding agents established so far for this method tend to penetrate thermal spray coatings and may invalidate results unless the coatings are thick enough to prevent penetration through the coating. Further development may establish that thin layers of certain types of especially dense coatings may be tested satisfactorily. Alternatively, new adhesive bonding agents that would allow reduction of the minimum thickness limitation may become available.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

E 4 Practices for Force Verification of Testing Machines

3. Significance and Use

3.1 This test method is recommended for quality control, acceptance testing; or it may help to develop or qualify a thermal spray operator's equipment and procedure or to aid in developing thermal spray coatings with improved adhesion and integrity.

3.2 This test method is useful for comparing adhesion or cohesion strengths of coatings of similar types of thermal spray materials. The test should not be considered to provide an intrinsic value for direct use in making calculations, such as to determine if a coating will withstand specific environmental stresses. Because of residual stresses in thermal spray coatings, actual strength depends upon the shape of the particular coated part. Also, in use, a coating may be stressed in a more complex manner than is practical for a standard test.

4. Apparatus

4.1 A tension testing machine shall conform to the requirements of Practices E 4. The loads used in determining the adhesion or tensile strength shall be within the loading range of the testing machine, as defined in Practices E 4. Permissible variation shall be less than 1.0 %. It shall be possible to apply increasing tensile load at a constant rate of cross-head travel between 0.030 in./min (0.013 mm/s) and 0.050 in./min (0.021 mm/s). The machine shall include a load-indicating device that registers the maximum load applied before rupture occurs.

4.2 Self-aligning devices, for applying the tensile load to the assembly of the coating and fixtures, shall not permit eccentric load or bending moment to the specimen. Self-alignment is often provided by the manufacturer as an integral part of the testing machine. An alternative, satisfactory apparatus is shown in Fig. 1, which also shows methods of connecting the self-aligning apparatus to an assembled test specimen.

5. Material

5.1 Adhesive Bonding Agent—A suitable adhesive bonding
agent shall be agreed between the purchaser and manufacturer of the coating and shall meet the following requirements.\textsuperscript{3}

5.1.1 The bonding agent shall be capable of bonding the coating to the loading fixture with a tensile strength that is at least as great as the minimum required adhesion and cohesion strength of the coating.

5.1.2 The bonding agent shall be sufficiently viscous not to penetrate through a 0.015-in. (0.38-mm) thickness of the coating. Certain commercial resins that cure or harden at room temperature by means of a curing agent have been proven satisfactory. If any other bonding agent is to be used, it shall first be compared with a proven bonding agent using this test method with the desired thermal spray coating.

\textbf{NOTE 2—}Thermal spray coatings may have an inherent porosity. Excessive penetration of the adhesive bonding agent into this porosity may affect the results determined by this test method. Unless proved satisfactory by comparison testing, any agent requiring elevated temperature for curing should be avoided because viscosity may decrease at high temperature, allowing penetration.

\textbf{NOTE 3—}When liquid epoxy bonding agents are used, there should be a procedure in place to ensure relatively consistent thickness on every sample.

5.1.3 The adhesion strength of the bonding agent shall be determined each time this test method is performed. This shall be done by using the bonding agent to attach a loading fixture to a second loading fixture, in accordance with 6.5, except that the coated substrate fixture of 6.5 is replaced with the second loading fixture.

\textbf{NOTE 4—}One reason for testing the bonding agent each time is to detect improper preparation of the agent if it is a two-part mix. Another reason is that adhesion strength generally decreases with age of the unused agent. If strength is lower than required, more adhesive bonding agent shall be prepared and tested, or the agent shall be discarded and replaced.

6. Test Specimens

6.1 Substrate and Loading Fixtures—Each test specimen is an assembly comprising a substrate fixture, to which the

\textsuperscript{3} A list of satisfactory bonding agents is provided in the annex which follows this standard.
coating is applied, and a loading fixture. The substrate and loading fixtures shall each be circular, solid cylinders of no less than 1.5 in. in length, or as agreed upon by the manufacturer and customer. A suggested detail for either fixture is shown in Fig. 2. One end of each fixture shall be adapted for attachment to the self-aligning loading devices of the tension testing machine. Both ends of each fixture shall have faces parallel to each other and normal to the loading axis. The facing diameters shall be not less than 0.9 in. (23 mm), nor more than 1.0 in. (25 mm). The diameters of the two fixtures shall be the same and shall be measured so that the error is no greater than 0.5%.

NOTE 5—In Appendix X1, an alternative substrate and fixture arrangement is provided that has proved cost effective and simple.

6.1.1 Material for Substrate Fixture—The substrate fixture shall be constructed of metal, preferably metal intended for use as the substrate for the coating. If no such substrate material is specified, the substrate fixture shall be SAE 1018 or 1020 steel.

NOTE 6—If desired because of cost or ease of fabrication, it may be suitable to attach or bond a layer of the specified substrate material to a fixture formed of any convenient metal. Such a layer of substrate material need not be metal. The layer must be substantially thicker than the possible depth of effects on the substrate, such as recrystallization or diffusion zones, that may result from applying the coating. A layer greater than 0.1 in. (2.5 mm) thick should be sufficient.

6.1.2 Material for Loading Fixture—The loading fixture shall be constructed of metal, but material is otherwise optional. It is usually convenient to make the loading fixture of the same material as the substrate fixture; thus, the fixtures may be interchangeable until a coating is applied to one.

6.2 Coating Application—The front facing of the substrate fixture shall be prepared in the manner required by the specification for the coating. (Roughening by grit blasting or surface grinding may be typical preparations.) The coating shall be thermal sprayed onto this prepared surface.

6.3 Coating Thickness—The coating thickness shall be measured with a micrometer by measuring the total length of the coating fixture before and after the coating is applied. (Care must be taken to avoid contaminating the prepared surface before coating.) The final coating thickness shall be more than 0.015 in. (0.38 mm). If the coating is to be ground or machined, the as-sprayed coating shall be approximately 0.005 in. (0.13 mm) thicker to allow for removal of material. The coating thickness shall not vary across the surface by more than 0.001 in. (0.025 mm). (This thickness variation, as measured from the
rear face, does not refer to the ordinary surface texture or roughness typical of thermal spray coatings.) If, upon completion of the thermal spraying, the coating thickness varies in excess of this limit, this shall be corrected by removing the coating and respraying or by grinding or machining the coating surface.

6.4 Grinding or Machining the Coating Surface—The surface of the coating may be finished by grinding or machining when the thickness variation is excessive. If the thickness variation is not excessive, it shall be optional to finish the surface of the coating as a useful and convenient aid in holding the fixtures together parallel and aligned as required for the next step. No specific grinding or machining procedure can be recommended, as this depends on the type of coating material. Usually manufacturers of the coatings have recommendations published or available. Only a rough grinding or machining step is needed, to provide a final coating thickness that does not vary by more than 0.001 in. (0.025 mm). Removal rate shall be insufficient to damage the coating or bond. A recommended method is to use a surface grinder with a magnetic chuck, positioning the rear face of the coated fixture on this magnetic chuck. No other treatment, such as grit blasting, shall be done to the surface of the coating.

6.5 Attachment of Fixtures—The facing of the loading fixture shall be free of oil, grease, or grinding or cutting fluids. The facing shall be mechanically cleaned by such means as machining, grinding, light grit blasting, or rubbing with emory cloth. This facing shall be attached to the surface of the coating, using the adhesive bonding agent according to its manufacturer’s instructions. Excessive adhesive shall be wiped from the assembly with soft paper or cloth. The two fixtures shall be held together parallel and aligned until the bonding agent is cured or hardened. A suitable holding device such as a “V-block” shall be used for the purpose, except such a device is not necessary if the surface of the coating has been ground or machined smooth.

6.6 Number of Test Specimens—The number of test specimens chosen depends upon the purpose of the particular tests under consideration. However, if specimens are to be used for acceptance tests, not less than five specimens of a type shall be tested.

7. Procedure

7.1 Prepare the chosen number of substrate fixtures, and apply a thermal spray coating to each. Finish the coating surface if required.

7.2 Prepare the adhesive bonding agent. Attach cleaned loading fixtures to all the coated substrate fixtures at essentially the same time. In addition, prepare one set of uncoated fixtures for measurement of the adhesion strength of the bonding agent.

7.3 Apply a tensile load to each test specimen at a constant rate of cross-head travel between 0.030 in./min (0.013 mm/s) and 0.050 in./min (0.021 mm/s) until rupture occurs. Record the maximum load applied.

Note 7—Loading fixtures may be gravity or pressure devices. The design of the loading fixtures should enable the correct alignment of the specimen.

8. Calculation

8.1 Calculate the degree of adhesion or cohesion strength as follows:

\[
\text{Adhesion or cohesion strength} = \frac{\text{maximum load}}{\text{cross-sectional area}}
\]

9. Interpretation of Results

9.1 Any interpretation of results depends on the purpose of using this test method and on the description of failure. The adhesion or cohesion strength value measured represents the weakest part of the system, whether in the coating or at an interface. A low-power microscope with a magnification range up to 100× is suggested for determining location of failure (also termed as the “locus” of failure).

9.2 The adhesion strength of the coating is given if failure is entirely at the coating-substrate interface.

9.3 The cohesion strength of the coating is given if rupture is only within the coating. Failure in the bonding agent may be a satisfactory result for a quality control assurance test or for a qualification test, if the strength of the bonding agent is greater than the minimum required adhesion or cohesion strength of the coating.

9.4 If failure occurs in a combination of these locations in one specimen, generally no interpretation of the initial cause can be provided. Fig. 3 diagrams the possible modes of failure.

9.5 For a multicomponent system; for example, a bond coat with a ceramic overlay, then failure at the interface between two coatings is described as “internal adhesive.”

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**FIG. 3** Nomenclature of Specimen Components and Classification of Failure Locii
10. Report

10.1 The report shall include the following:

10.1.1 Coating material or manufacturer’s designation technique used to apply the coating, including type of thermal spray equipment, and spray parameters.

10.1.2 Final coating thickness and statement of whether surface is finished or as-sprayed.

10.1.3 Substrate material.

10.1.4 Description of surface preparation of substrate.

10.1.5 Name or description of bonding agent and details of bonding procedure if different from manufacturer’s instructions.

10.1.6 Number of thermal spray specimens and number of specimens tested.

10.1.7 The adhesion or cohesion strength of each specimen tested.

10.1.8 Average adhesion and cohesion strength, and the maximum and minimum values, in pounds per square inch (or pascals).

10.1.9 Description of failure, including statement of whether failure occurred at the coating-substrate interface, in the coating, in the bonding agent, or a combination of these.

For multilayered coatings, an internal adhesion failure also must be indicated if it is present. Fig. 3 diagrams the possible modes of failure.

10.1.10 Adhesion strength of the bonding agent in the test specimen without a thermal spray coating.

11. Precision and Bias

11.1 No justifiable statements can be made regarding the precision and bias of this test method because it evaluates coatings that exhibit brittle fracture, an unpredictable characteristic.

11.2 This test method is applicable to a wide variety of materials with different characteristics.

11.3 Since design, base metal composition, fabrication, and processing, as well as thermal spraying the coating, will give rise to variables in adherence, each application of this test method should have tolerances and interpretation of adherence set and agreed upon between the purchaser and the manufacturer.

12. Keywords

12.1 adhesion strength; cohesion strength; fracture locus; thermal spray coatings

ANNEX

(Mandatory Information)

A1. ADHESIVE BONDING AGENTS FOR ATTACHMENT OF LOADING FIXTURE TO THERMAL SPRAY COATINGS

A1.1 The following adhesive is recommended for attaching the loading fixture to thermal spray coatings that are primarily metallic or that have a metal matrix. It is not recommended for thermal spray oxide or other porous ceramic coatings because of the possibility of excessive penetration into the coating. This is a two-part mix that is cured at 300°F (149°C) for 1 h. When the adhesive is new, typical adherence strength to thermal spray coatings may range up to approximately 8000 psi (55 MPa) depending on the coating material.

A1.1.1 CONAP 1222, manufactured by CONAP Inc., 184 E. Union St., Allegheny, NY 14706.

A1.2 The following adhesives are recommended for attaching the loading fixture to thermal spray coatings of any type, ceramic or metallic. These are two-part mixes that should be cured at room temperature when used for this test method. When the adhesive is new, typical adherence strength to thermal spray coatings may range up to approximately 4000 psi (28 MPa) depending on the coating material.

A1.2.1 Bondmaster M666 or M777, manufactured by Pittsburgh Plate Glass Co., Adhesive Products Div., 225 Belleville Ave., Bloomfield, NJ 07003 (M777 may be easier to use as it may be more viscous than M666).

A1.2.2 Epon 911F, manufactured by Shell Chemical Co., Adhesives Dept., P.O. Box 831, Pittsburgh, CA 94565.

A1.2.3 Armstrong A-12, manufactured by Armstrong Products Co., Argonne Rd., Warsaw, IN 46580.

A1.2.4 Hysol XA7-H368 Grey, manufactured by Hysol Inc., Olean, NY 14760.
XI. ALTERNATIVE SUBSTRATE AND FIXTURE ARRANGEMENT

X1. Alternative Substrate and Fixture Arrangement that has proved cost effective and simple.

BIBLIOGRAPHY


INTRODUCTION

Porcelain-enamed gray iron is a composite of a vitreous or glassy inorganic coating, bonded to a casting by fusion at temperatures above 800°F (425°C). Porcelain enamels are a family of coatings available in a wide variety of compositions and properties, but all are characterized by their glass-like nature. Selection of an appropriate porcelain enamel must be made on the basis of the end-use requirements. Certain casting design features and processing considerations can facilitate the application and efficient use of the selected enamel.

Two general types of enamels are available for use on cast iron. These are commonly referred to as wet-process and dry-process enamels (see Definitions C 286). In wet-process enameling, a slurry of wet-ground materials is dipped or sprayed on the casting, the water removed by drying, and the coating matured by heating in a furnace for sufficient time to bring about fusion of the glassy particles. In dry-process enameling, dry-powdered glassy material is applied by dusting onto a redhot casting that has been ground-coated by the wet process prior to firing. The partially matured dusted coating is returned to the furnace to complete the fusion process. In general, wet-process enamels are thinner over-all than dry-process enamels.

1. Scope

1.1 These practices are intended to indicate certain casting characteristics and pre-enameling practices which will facilitate finishing by the wet- or dry-process methods of porcelain enameling. All of the listed recommendations are based on experiences with gray iron casting and enameling.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
A 48 Specification for Gray Iron Castings
A 74 Specification for Cast Iron Soil Pipe and Fittings
A 126 Specification for Gray Iron Castings for Valves, Flanges, and Pipe Fittings
A 278 Specification for Gray Iron Castings for Pressure-Containing Parts for Temperature up to 650°F
C 286 Terminology Relating to Porcelain Enamel and Ceramic-Metal Systems

3. Recommended Casting Characteristics

3.1 Design of the casting should be such as to minimize variations in temperature during firing and cooling. Section thickness should be uniform to eliminate possible warping and fire cracking of castings; to facilitate an even rate of heating and cooling and to prevent possible spalling, hairlining, and blistering of the porcelain enamel.

3.2 When a variation in section thickness is unavoidable, the transition of the two sections should be gradual and smooth. Abrupt changes in sections give rise to significant differences in heating and cooling rates, resulting in nonuniform coating conditions.

3.3 Special styling techniques should be used for designing appendages, internal passages, and lug-fastening faces so as not to emplace a mass of metal near an otherwise uniform enameling surface. These design considerations should include a thorough review of the available mold-making techniques in conjunction with the pattern designer.

3.4 Where functional or mating surfaces of an enameled casting are a design consideration, allowances must be included for the thickness of the coating and the method of application. The optimum thickness of wet-process enamels is...
about 10 mils (0.25 mm) in dry process enamels it is about 40 mils (1.0 mm).

3.5 Sharp edges on castings should be avoided, because neither the wetnor dry-process coatings will adequately cover sharp edges. Inside and outside corners should be rounded to uniform thickness and generous radii provided for fillets and outside corners.

3.6 Material identifications for the castings should be selected from appropriate ASTM specifications which are found under the various headings for gray iron.2

3.6.1 An example of the more desirable types of iron for enameling purposes are the normally ferritic Class 20 irons (see Specification A 48 for Gray Iron Castings). They cast more readily into complex shapes, and are better suited to the coating process.

3.6.2 Some applications, such as valve bodies, may require other types of gray iron for which Class B, Specification A 126, would be selected. Other appropriate Specifications would be A 74 and A 278, in which the lowest strength class is preferable for coating purposes.

3.7 Parting lines coincident with an enameling surface should be accessible for grind finishing.

4. Recommended Foundry Practices

4.1 The governing factors in pattern layout and shop control are elimination of discontinuities, chill, and inclusions at or near the surfaces to be coated.

4.2 Metal compositions and unnecessary increases of carbon equivalents in hypereutectic irons that give rise to coarse graphite or kish in heavy sections should be avoided. Heavy combined carbon will result in the formation of kish during the enameling fire and may cause poor adherence, spalling, or blistering, or combination thereof.

4.2.1 For lighter section castings ¼ in. (6.35 mm) thick and under, the desirable range for carbon equivalent is 4.3 to 4.5 %.Carbon equivalent is generally calculated as: C.E. = percent total carbon + 1⁄3 (percent silicon + percent phosphorus).

4.2.2 Sulfur in excess of 0.14 % and out-of-balance sulfur will cause enamel defects.

4.2.3 Manganese content of the iron must be sufficient to balance the sulfur content. A slight excess of manganese is preferred in order to assure sulfur tie-up; that is, Mn, percent = (1.7 × S, percent) + 0.3.

4.2.4 High phosphorus content of 0.70 % may be desirable for improved strength at enameling temperatures. Phosphorus in the iron has no reported association with boiling defects in the coating.

4.3 When pouring thin-walled or complex shapes to be enameled, one must consider the effect of metal composition on microstructure. White or mottled structures will not roughen adequately during cleaning, and also may introduce other problems in the coating process. Silicon content over 2.4 % and the use of heater strips may be effective, but a suitable anneal is the desirable corrective measure.

4.4 Metal having a microstructure containing massive carbides and high pearlite content will introduce enameling problems. Heat treatments employed to obtain desired mechanical properties in the casting should minimize these problems.

4.5 Where annealing is a regular part of the foundry operations, an oxidizing furnace atmosphere is highly desirable in order to produce easily removed scale and obtain decarburized enameling surfaces. Decarburized surfaces are advantageous to enameling.

4.6 Heating and cooling cycles employed in the enameling process cause transformations that affect microstructure. Appropriate metallurgical constituents used to stabilize or retard these conditions should not be incorporated until a thorough study is made of their effect on the coating results. Examples of pearlite stabilizers are tin or manganese.

4.7 Shakeout techniques must be geared to both casting warpage and potential effect on enameling results. Castings should be fully separated from the sand once shakeout is started to prevent high internal stress that would later cause casting warpage or cracking or enameling defects.

4.8 Contaminants, harmful to the coating process, should be avoided in the molding sands and cores for castings to be enameled. Carbonaceous coatings for cores and molds are reported to be particularly harmful.

5. Recommended Pre-Enameling Practices

5.1 Visual inspection methods for enameling surfaces should place emphasis on the detection and remedy of porosity, sand inclusions, and gas holes. Porosity consisting of essentially subsurface pinholes, shallow covered blows, body scars, or shrinkage near the surface may or may not be acceptable for correction, depending upon severity.

5.1.1 Non-continuous metal consisting mainly of misrun (in which metal fails to fill out the mold cavity) or cold shut (imperfect fusion of metal against metal) should not be coated where appearance requirements of the finish are involved. Mold shifts, core shifts, or improperly aligned patterns resulting in an improperly positioned casting surface are not detrimental to the coating processes unless they give rise to unequal heating rates.

5.1.2 Machined or ground surfaces and metallic-cosmetic repairs should be cleaned by appropriate methods prior to inspection.

5.1.2.1 Cosmetic repair of various-surface blemishes, using metallic or ceramic fillers, should be made subject to agreement by coater and founder, and influenced by economic and feasibility aspects.

5.1.2.2 Metal-filler repairs of blemishes after elimination by mechanical methods such as grinding should be based on the extent and condition of the repair area. Under certain circumstances repair methods such as welding, brazing, or mechanical peening may not be wholly desirable.

5.1.2.3 Ceramic-filler repair on small-subsurface holes that do not contain inclusions can usually be made with a water-based, quartz-clay-soda ash putty.

5.1.3 White fractures due to chilled iron at edges and sharp corners, and structures containing massive carbides are not readily decomposed during enameling. Such castings should be heat treated to a softened condition prior to mechanical cleaning.

5.1.3.1 Oils and greases, whether used for temporary surface preparation or resulting from machining operations,
should be removed by methods that will produce an enamel-compatible surface.

5.1.3.2 Thermal cleaning or heating the casting sufficiently to burn out organic soil is the most desirable pretreatment method prior to mechanical cleaning.

5.1.3.3 Oxide films, scale, and similar surface matter should be removed by mechanical cleaning.

5.1.3.4 Cleaning prior to the enameling process should remove foreign material and produce a sharply roughened surface without peening or contaminating it.

5.1.3.5 Two mechanical cleaning methods usually employed are sand blasting and airless grit blasting. Shot is not recommended, as it tends to peen rather than cut the surface. A third category of tumbling is rarely used. In all mechanical cleaning methods, the longer the cleaning time, the less tendency there is for boil-type defects. Grit or sand used to clean castings should be free of extraneous matter such as nonferrous metal, cutting oils, paint, dust, or other soils that tend to contaminate enameling surfaces.

5.1.4 Chemical cleaning processes used to remove organic soils should be followed by a roughening action such as blasting. Pickling is not resorted to since it gives rise to defects in enameling.

5.1.5 Heat treating employed prior to enameling if performed in an oxidizing atmosphere will minimize boiling defects and partially relieve stresses. Two general types are considered for different heat treating results:

5.1.5.1 Normalizing the casting for partial graphitization of massive combined carbon and decomposition of pearlite is one type of heat treating. Normalizing should be done in the 1625 to 1650°F (885 to 900°C) temperature range and for 1 h/in. of section with a minimum of 20 min at temperature per casting.

5.1.5.2 The other type of heat treating is subcritical anneal to partially graphitize pearlite. Subcritical annealing should be done in the 1360 to 1420°F (735 to 770°C) temperature range and for 1 h/in. of section with a minimum of 20 min per casting.

5.1.6 Enameling operations should begin on castings as soon as possible, within a week after foundry finishing.

5.1.7 Castings should be stored in a dry place. They should not be “aged.” If aged castings are to be enameled, an annealing treatment prior to enameling operations is beneficial.
Standard Test Methods for Thickness of Diffusion Coating

1. Scope

1.1 These test methods cover two procedures for measuring the thickness of diffusion coatings.

1.2 Test Method A is the determination of the dimensional-change thickness, defined as the difference in the thickness of the part before and after coating. (The terms micrometer thickness and part growth are considered synonymous with dimensional change thickness.)

1.3 Test Method B is the determination of total coating thickness, defined as the distance between the observably unaffected substrate and the exterior surface of the coating. This includes the total of all included phases, zones and layers. (The term case depth is considered to be synonymous with total coating thickness.) The total coating thickness is determined by cross-sectioning the coating, preparing a metallurgical mount and microscopically measuring the coating thickness.

1.4 The total coating thickness as determined microscopically from a cross-section will usually be greater than, or equal to, the dimensional change thickness determined by part growth. When the coating is produced primarily by reaction with the substrate, the substrate-coating interface recedes as the substrate is consumed in the reaction. In such cases the difference between the total coating thickness and the dimensional change thickness is the thickness of the substrate consumed.

1.5 Diffusion coatings are usually formed at elevated temperatures for service at elevated temperatures. This means that diffusion coatings are dynamic systems which are continually undergoing changes while in an elevated-temperature environment. It is necessary to know that certain phases are growing at the expense of others and to know the previous history of a coating to understand the significance of coating thickness data.

1.6 Values in SI units are to be regarded as the standard. Inch-pound units are provided for information only.

1.7 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

D 374 Test Methods for Thickness of Solid Electrical Insulation

E 3 Methods of Preparation of Metallographic Specimens

3. Significance and Use

3.1 A diffusion coating is one produced by causing an element or elements to react with or diffuse into, or both, the surface of a metallic substrate, thus chemically altering the substrate adjacent to the surface. To appreciate the significance of coating thickness measurements one must understand the contributions to a particular coating of solid-solution zones in the substrate and reaction products such as intermetallic compounds.

4. Test Method A

4.1 Apparatus—The instrument shall be a machinist’s type micrometer without a locking device. If calibrated in inches, it shall be constructed with a vernier reading to 0.0001 in. (0.1 mil). If calibrated in metric units, it shall be capable of reading to 0.01 mm. It shall have a ratchet or similar mechanism, such as a friction thimble, for controlling measuring pressure and shall have anvil and spindle surfaces 6.00 ± 0.03 mm (0.250 ± 0.001 in.) in diameter. It shall meet all other requirements and calibration procedure for Method A of Test Method D 374.

4.2 Procedure:

4.2.1 Clean the area selected for coating-thickness measurement of dust or other powdery materials prior to coating. Record the precise area to be measured, so that the same area can be remeasured after coating.

4.2.2 Take a minimum of two readings, using the machinist’s micrometer, in each area selected. Use the same procedure for using the micrometer as that stipulated in Test Methods D 374.

4.2.3 After the part has been coated and the surface has been cleaned of superficial powder or dirt, repeat the measurement procedure. It is necessary to ensure that the same location is remeasured.

4.2.4 The thickness of the part after coating minus the thickness of the part prior to coating divided by two, is the dimensional change thickness per coated surface.

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1 These test methods are under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and are the direct responsibility of Subcommittee B08.12 on Materials for Porcelain Enamel and Ceramic-Metal Systems.


3 Annual Book of ASTM Standards, Vol 03.01.
5. Test Method B

5.1 Apparatus:

5.1.1 Standard Metallurgical Specimen-Preparation Equipment.

5.1.2 Metallographic or Optical Microscope.

5.1.3 The equipment used shall be consistent as to type and quality as specified in Methods E 3.

5.2 Procedure:

5.2.1 Section with a fine grained cut-off wheel the area of the coated part in which the total coating thickness is to be measured. Control the clamping of the part, lubricant cooling fluid, and control the cutting rate to produce a square cut edge without chipping the coating. Exercise care to ensure that the cut is perpendicular to the surface of the coating.

5.2.2 After cleaning with an organic solvent, plate the section (electrolytically or electroless) with nickel, chromium, or other metallic plating which approximates the hardness of the coated substrate. As an alternative to plating, wrap the specimen in a metallic foil. The foil applied must give sufficient support to prevent chipping, and be in sufficient proximity to the specimen to prevent gaps and rounding of the edges during polishing.

5.2.3 Use the standard techniques detailed in Methods E 3 for mounting and polishing of the metallurgical specimen.

5.2.4 The etching procedure, if required, shall be in general accordance with Methods E 3. The specific etchant and etch time, if employed, will be by mutual agreement between the customer and vendor. The etching procedure will clearly demarcate the boundaries of the coating as well as properly differentiate included zones, if it is desirable to measure their thickness as well.

5.2.5 Measure the total coating thickness microscopically with the aid of a calibrated-filar eyepiece, or by direct measurement of a projected image on ground glass. Magnify the coating a minimum of 250× diameters, although 500× is preferable. When measuring from ground glass, measure the image of the coating to the closest 1.27 mm (0.05 in.). The exterior bounds of the coating is the average of the peaks and valleys. Visually average over a minimum length of 10 mils (0.254 mm (0.010 in.)). Determine the interior bounds of the coating as the depth to which the substrate has been visually altered by the coating. (One must appreciate that this depth is highly dependent upon the etchant employed, and mutual agreement of concerned parties should be sought.) As was the case of the exterior bounds, visually average over a 10 mil (0.254 mm (0.010 in.)) length. Measure the specific zones or phases by a mutual agreement between the purchaser and the vendor. Measure the total coating thickness at a minimum of 10 locations around the entire metallographic specimen with the measurement points separated by at least 20 mils (0.508 mm (0.020 in.)). Average the individual total coating-thickness measurements.

6. Report

6.1 The report shall include the following:

6.1.1 Thickness as mils per surface. Maximum, minimum, and average of the individual measurements made on a specific part or specimen.

6.1.2 Type of coating thickness determined and the method employed.

6.1.3 Specific area where the thickness was measured.

6.1.4 Type and condition of the coating such as “as deposited” or “after a specified term of service.”

6.1.5 If Test Method B is employed, the etching procedure. A photomicrograph showing the bounds of a typical area of the coating is suggested, and

6.1.6 If Test Method B is employed, the thickness of any phases or layers as agreed upon between the purchaser and the seller in mils per surface.

7. Precision and Bias

7.1 Test Method A (dimensional change thickness)—±0.2 mil (0.005 mm (0.0002 in.)). This test method assumes that both sides of surfaces of the coated piece have an equal coating thickness, if both surfaces are coated. The validity of this assumption will vary with different coatings and processes of application.

7.2 Test Method B (total coating thickness)—±0.1 mil (0.0001 in., 0.0025 mm).
Standard Test Method for Weight Loss (Mass Loss) of Sheet Steel During Immersion in Sulfuric Acid Solution

This standard is issued under the fixed designation C 694; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

In the manufacture of porcelain-enamelled ware, formed steel articles are pretreated to ensure enamel adherence. The pretreatment comprises, in part, of etching the steel surface with sulfuric acid solution and in depositing nickel on the steel surface from a nickelous sulfate solution. Conditions are maintained to provide a minimum amount of metal removal (weight loss) (mass loss) in the acid solution and a minimum amount of nickel deposition. These minimums are particularly critical in direct-on enameling in which the ground-coat enamel with its adherence-promoting oxides is omitted.

1. Scope

1.1 This test method covers the evaluation of the weight-loss (mass loss) characteristics of sheet steel in sulfuric acid solution.

1.2 This test method provides means of rating the effectiveness of in-plant pretreatment acid solutions in preparing steel surfaces for porcelain enameling.

1.3 The values stated in inch-pound units are to be regarded as the standard. The values in parentheses are for information only.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Terminology

2.1 Definitions:

2.1.1 quarter lines—imaginary lines parallel to the direction of rolling, positioned at a distance from the sheet mill edge equal to one quarter of the sheet width.

3. Summary of Test Method

3.1 Representative sheet-steel specimens are selected, measured, cleaned, and weighed prior to immersion for measured periods in a bath of dilute sulfuric acid that has been preconditioned by controlled solution of panels of the same sheet steel. The specimens are rinsed, dried, and reweighed after the timed exposure.

3.2 Values of weight loss (mass loss) per unit area are calculated for the four acid immersion periods and, if desired, the rate of weight loss (mass loss) per unit area per unit time is calculated.

4. Significance and Use

4.1 The results of this test method can be used to evaluate the pickle weight-loss (mass loss) characteristics of a given lot of sheet steel in dilute sulfuric acid solution, and may enable the enamel processor to select a pickling time that will provide satisfactory porcelain enamel bond.

4.2 The results of this test method can be used to evaluate the effectiveness of the enamel processor’s pretreatment system in preparing the steel for porcelain enameling, and may aid the processor in obtaining satisfactory porcelain enamel bond.

5. Apparatus

5.1 Analytical Balance, accurate to 0.01 g.

5.2 Linear Measuring Device.

5.3 Borosilicate Glass Container, having an inside diameter of about 11 1/2 in. (290 mm) and an outside depth of about 11 in. (280 mm).

5.4 Water Bath, heated, of sufficient size to immerse the glass container (5.3) to within about 1 in. (25 mm) of its top.

5.5 Glass Plate or Acid-Resistant Porcelain-Enameled Steel Sheet, sufficient to cover the container described in 5.3.

5.6 Stainless Steel Jig, for support of test specimens (see Fig. 1, Fig. 2, and Fig. 3).

6. Reagents and Materials

6.1 Distilled Water.

6.2 Isopropyl Alcohol \((\text{CH}_3\text{CH(OH)}\text{CH}_3)\).

6.3 Methyl Alcohol \((\text{CH}_3\text{OH})\).

6.4 Steel Sheet, sufficient in size to provide the panels and strips described in 6.4.1 and 6.4.2, and to provide the test specimens described in 7.1 and 7.1.1.
6.4.1 Shear two to four 4 by 6-in. (102 by 152-mm) panels from the steel sheet of 6.4. Use these panels in the preconditioning in accordance with 9.3.3 and 9.3.4.

6.4.2 Shear ten to twelve ¼ by 5-in. (6 by 127-mm) strips from the steel sheet of 6.4. Fashion these strips into hooks for hanging test specimens from the stainless steel jig.

6.5 Sulfuric Acid (H₂SO₄), American Chemical Society (ACS) reagent grade.

6.6 Trisodium Phosphate—(Na₃PO₄·12H₂O), granular, technical grade.

7. Sampling

7.1 Shear eight test specimens, each 4 by 6 in. (102 by 152 mm), from within the quarter lines of the sheet or coil.

7.1.1 Choose specimens from rust-free areas that do not contain the mill identification stamp.

7.1.2 Identify the specimens by steel die stamping.

8. Test Specimens

8.1 File edges of the eight test specimens lightly to remove shearing burrs.

8.1.1 Punch or drill a hole near one end, at the center of the specimen width.

8.1.2 Determine the width, W, and length, L, of the test specimens to the nearest 0.01 in. (nearest 1 mm).

8.1.3 Thoroughly clean the specimens with methyl alcohol. (Thereafter, handle the specimens by the edges with clean white gloves.)

8.1.4 Dry in still air.

8.1.5 Store the specimens in a desiccator until ready for weighing.

8.2 Determine the initial weight (mass), W₁, of each test specimen to the nearest 0.01 g.

8.2.1 Store the specimens in a desiccator until ready to run the test.

9. Preparation of Solutions

9.1 Cleaner—Prepare at least 19 L of 5 ± 0.5 weight (mass) percent solution using 53 g of trisodium phosphate per litre of tap water.

9.1.1 Control the cleaner temperature at 190 to 195°F (88 to 91°C).

9.2 Cleaner Rinse—At least 19 L of tap water.

9.2.1 Control the rinse temperature at 170 ± 5°F (77 ± 3°C).

9.3 Sulfuric Acid Solution—Add 765 mL of ACS reagent grade H₂SO₄ to 16 900 mL of distilled water in the glass container described in 5.3.

9.3.1 Immerse the acid container to within about 1 in. (25.4 mm) of its top in the heated water bath described in 5.4. Do not heat the acid with an immersion heater.

9.3.2 Control the acid temperature at 155 ± 1°F (68 ± 1°C).
9.3.3 Process the two to four panels in 6.4.1 through the cleaner (9.1) and cleaner rinse (9.2) so that the panels are thoroughly cleaned, as evidenced by freedom from water breaks.

9.3.4 Immerse the two to four panels in the acid solution to dissolve a total of 15 to 30 g of iron. Do not add iron powder or chemicals such as ferrous sulfate (FeSO$_4$·7H$_2$O) to the acid to obtain the required dissolved iron.

9.3.5 To avoid evaporation, place the glass plate or acid-resistant porcelain-enameded steel sheet on the acid container when the acid is not in use.

9.4 Acid Rinse—At least 19 L of tap water.

9.4.1 Adjust the rinse to a pH of 2.5 to 3.0 with H$_2$SO$_4$.

9.4.2 Maintain the acid rinse at room temperature.

9.5 Final Rinse—At least 19 L of tap water.

9.5.1 Maintain the final rinse at room temperature.

9.6 Isopropyl Alcohol Rinse—Sufficient quantity for complete immersion of two panels.

9.6.1 Maintain the alcohol rinse at room temperature.

10. Procedure

10.1 Suspend the eight specimens on the stainless steel jig (Fig. 1) using hooks made of metal strips (6.4.2).

10.2 Immerse the specimens in the cleaner (9.1) for 10 min.

10.2.1 Discard the cleaner in accordance with 10.3.2.

10.3 Transfer the eight specimens to the cleaner rinse (9.2), and immerse the specimens for five 15-s periods (momentarily remove the specimens from the rinse after each immersion period).

10.3.1 If water breaks are observed on the specimens after the last immersion period, repeat 10.2 and 10.3.

10.3.2 If water breaks are observed after repeating 10.2 and 10.3 three times, prepare a new cleaner solution and repeat 10.2-10.3.1.

10.3.3 Discard the cleaner rinse after processing each group of eight specimens.

10.4 Transfer the eight specimens to the H$_2$SO$_4$ solution taking care not to agitate the solution or the specimens (Fig. 2).

10.4.1 Discard the acid solution after processing each group of eight specimens.

10.5 Transfer a set of two specimens from the acid solution (Fig. 3) to the acid rinse at successive 5 min ± 5-s intervals.

10.5.1 Agitate each set of two specimens for 1 min.

10.5.2 Discard the rinse after processing each group of eight specimens.
10.6 Transfer each set of specimens to the final rinse.
10.6.1 Agitate the specimens for 30 s.
10.6.2 Discard the final rinse after processing each group of eight specimens.
10.7 Transfer the specimens to the isopropyl alcohol.
10.7.1 Agitate the specimens for 30 s.
10.8 Dry the specimens in still air.
10.9 Place the specimens in the desiccator until they are ready for weighing.
10.10 Determine the final weight (mass), \( W_f \), of each specimen to the nearest 0.01 g.

11. Calculation

11.1 Calculate the weight loss (mass loss) in grams per square feet of surface, \( X \), using Eq 1:

\[
X = 72(W_i - W_f)(L \times W)
\] (1)

where:
\( W_i \) = initial panel weight (mass), g,
\( W_f \) = final panel weight (mass), g,
\( L \) = length of panel, in., and
\( W \) = width of panel, in.

11.2 Calculate the weight loss (mass loss) in grams per square decimetre of surface, \( Y \), using Eq 2:

\[
Y = 5(W_i - W_f) \times 10^3/(L \times W)
\] (2)

where:
\( W_i \) = initial panel weight (mass), g,
\( W_f \) = final panel weight (mass), g,
\( L \) = length of panel, mm, and
\( W \) = width of panel, mm.

12. Report

12.1 The report shall include the following:
12.1.1 Calculated weight loss (mass loss) per unit area and immersion time for each of the eight panels,
12.1.2 Average weight loss (mass loss) per unit area for each set of two panels at each immersion time, and
12.1.3 Amount of iron dissolved in the acid solution (9.3.4).

13. Precision and Bias

13.1 Precision—The 95 % reproducibility limits showing the difference between two test results is 3.99 ± 0.255 g/ft\(^2\) (55.42 ± 3.54 g/m\(^2\)) for repeatability and 3.99 ± 0.581 g/ft\(^2\) (55.42 ± 8.07 g/m\(^2\)) for reproducibility, where 3.99 g/ft\(^2\) (55.42 g/m\(^2\)) is the weight (mass) loss.

13.2 Bias—The major known sources of bias in this test method are: (1) the amount of iron dissolved in the sulfuric acid solution prior to the testing of the samples and its source, and (2) the requirement that the surface of the sheet steel samples be clean and show no water break before they are

<table>
<thead>
<tr>
<th>in.</th>
<th>(\frac{1}{4})</th>
<th>(\frac{5}{16})</th>
<th>(\frac{3}{8})</th>
<th>(\frac{1}{2})</th>
<th>2(\frac{1}{2})</th>
<th>3</th>
<th>5</th>
<th>6(\frac{1}{2})</th>
</tr>
</thead>
<tbody>
<tr>
<td>(mm)</td>
<td>(6.4)</td>
<td>(8)</td>
<td>(16)</td>
<td>(19)</td>
<td>(63.5)</td>
<td>(76)</td>
<td>(127)</td>
<td>(165)</td>
</tr>
</tbody>
</table>
immersed in the sulfuric acid. Both of these sources of bias have been pointed out in this test method and the procedure is designed to minimize them.

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Standard Test Methods for Spalling Resistance of Porcelain Enameled Aluminum

This standard is issued under the fixed designation C 703; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover accelerated determination of the resistance of porcelain enamel coatings on aluminum alloys to spalling from exposure to moisture or weathering. Test Method A, using a 5% solution of ammonium chloride, requires 96-h immersion while Test Method B, using a 1% solution of antimony trichloride, is completed after 20 h of immersion. The spalling tendency is evaluated by the same criteria in both methods. While either method is suitable for magnesium silicon alloys, such as 6061, Test Method B is preferred for simple alloys or commercially pure aluminum, such as 1100.

1.2 The test methods appear in the following order:

<table>
<thead>
<tr>
<th>Sections</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test Method A—Ammonium Chloride</td>
</tr>
<tr>
<td>Test Method B—Antimony Trichloride</td>
</tr>
</tbody>
</table>

2. Terminology

2.1 Definitions:

2.1.1 spalling—a defect characterized by separation of the porcelain enamel from the aluminum base metal without apparent external cause. Spalling can result from the use of improper alloys or enamel formulations, incorrect pretreatment of the base metal, or faulty application and firing procedures.

3. Significance

3.1 It is difficult to overemphasize the importance of the spall test. Porcelain enameled aluminum that fails this test will probably spall in service if subjected to moisture or weathering.

TEST METHOD A—AMMONIUM CHLORIDE

4. Apparatus

4.1 Container, glass or plastic, large enough to immerse the test area of the specimen completely and hold a minimum of 3 mL of solution per square centimetre of the immersed surface.

5. Reagent

5.1 Ammonium Chloride Solution (5%)—The test solution, freshly prepared, shall consist of 5 parts, by mass, of ammonium chloride (NH₄Cl) (technical grade is adequate) dissolved in 95 parts, by mass, of water. Deionized or distilled water is preferred, but in the case of very large production pieces, tap water may be used. Sufficient solution shall be prepared to permit complete immersion of the specimens.

6. Test Specimens

6.1 Full-size production pieces should be tested. When this is not practical, specimens approximately 4 by 6 in. (102 by 152 mm), cut from production parts should be tested. At least one representative specimen from each “job” or each 1000 ft² (93 m²) of production, whichever is applicable, should be spall tested. Spall-tested pieces should not be shipped.

6.2 Test production parts in as-produced condition.

7. Procedure

7.1 Immerse the test specimens completely in the NH₄Cl solution at room temperature. Large production pieces may be immersed in a container made by lining a packing box with polyethylene plastic sheeting.

7.2 Make a visual inspection after 96 h of immersion.

NOTE 1—Variations of this test may be used for studies of processing variables. These include scored or deformed samples, 24 h inspection, and multiple cycles of 96 h each.

8. Evaluation

8.1 Any of the following types of spall that develop within 96 h shall constitute failure:

8.1.1 Any spall area (revealing bare metal) extending 1/8 in. (3 mm), or more, from an edge and more than 1 in. (25 mm) in length.

8.1.2 Any spall area on the interior surface (not touching an edge) that is more than 1/8 in. (3 mm) to its maximum dimension, or

8.1.3 More than six visible spall spots per 1 ft² (929 cm²) on the interior surface.

9. Report

9.1 The report shall include the following:

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9.1.1 Evaluation of the specimen as passing or failing the immersion test in 96 h.
9.1.2 Complete identification of the specimen, including the basis metal, metal pretreatment, enamel slip formulation, firing time and temperature, date, and any other pertinent processing information, and
9.1.3 Size, description of part, number of specimens tested, and ratio of parts tested to parts produced.

TEST METHOD B—ANTIMONY TRICHLORIDE

10. Apparatus

10.1 Container, glass or plastic, large enough to immerse all or part of the test specimen and hold a minimum of 3 mL of solution per square centimetre of specimen surface.
10.2 Steel Rod or Mandrel of ½-in. (12.7-mm) diameter.

11. Reagent

11.1 Antimony Trichloride Solution (1 %)—The test solution, freshly prepared, shall consist of 1 part, by mass, of antimony trichloride (SbCl₃) (technical grade is adequate) dissolved in 99 parts, by mass, of water (10 g/L). Tap water may be used. Stir the solution thoroughly to disperse the antimony trichloride.
11.2 Discard the test solution when it becomes 3 days old or when the 1 gal/195 in.² (3 mL/cm²) limit is reached, whichever occurs first.

12. Test Specimens

12.1 When practical, full-size production pieces may be tested; otherwise, specimens approximately 4 by 6 in. (102 by 152 mm), cut from production parts or a 4-in. (102-mm) wide cross-sectioned cut sample from a production piece should be tested. It is necessary that the metal-enamel interface is exposed to the test solution. Cut edges or the cracks in the enamel caused by bending over a mandrel usually accomplish this. If not, the interface should be exposed by scoring the enamel. At least one representative specimen from each “job” or each 1000 ft² (93 m²) of production, whichever is applicable, should be spall tested.
12.2 Test production parts in as-produced condition.
12.3 Wherever practical (such as porcelain enameled sheet material) specimens cut from production pieces should be bent to a 45° angle over a ½-in. (12.7-mm) diameter rod or mandrel.

13. Procedure

13.1 Immerse as much of the test specimen as practical in the SbCl₃ solution at room temperature. If a sample has been bent over a mandrel, the bent portion must be immersed in the solution.
13.2 After 20 h of immersion, rinse the sample in water and scrub with a sponge to remove loose glass flakes (particularly on the bent areas).

14. Evaluation

14.1 On flat or non-deformed areas, any of the following types of spall that develop within 20 h shall constitute failure:
14.1.1 Any spall area (revealing bare metal) extending ⅛ in. (3 mm), or more, in from an edge and more than 1 in. (25 mm) in length.
14.1.2 Any spall area on the interior surface (not touching an edge) that is more than ⅛ in. (3 mm) in its maximum dimension, or
14.1.3 More than six visible spall spots per 1 ft² (929 cm²) on the interior surface. Disregard pinholes up to ⅛-in. (1.5-mm) diameter.
14.2 The evaluation of spall on the formed (bent) enamel areas has not been standardized for all aluminum alloys used in sheet form. While spalling on bends indicates a potential field failure of production pieces, absence of spalling on bends may not be indicative of good porcelain adherence of all aluminum alloys in sheet form. For 1100 clad alloy, the following spall developing in 20 h shall constitute failure:
14.2.1 Any spall area on the bent portion (excluding the edges) that is more than ⅛ in. (3 mm) wide or more than ½ in. (12.7 mm) in length.

15. Report

15.1 The report shall include the following:
15.1.1 Evaluation of the specimens as passing or failing the immersion test in 20 h, for the bent or deformed and flat areas, respectively,
15.1.2 Complete identification of the specimen, including the basis metal, metal pretreatment, enamel slip formulation, firing time and temperature, date, and any other pertinent processing information, and
15.1.3 Size, description of parts, number of specimens tested, and ratio of parts tested to parts produced.

16. Precision and Bias

16.1 No justifiable statements can be made regarding the precision and bias of these test methods because it is designed for application to full-size production parts, with the result that variables due to design, metal composition, fabrication, and metal processing, as well as porcelain enameling, are introduced into the results.
Standard Test Method for Nickel on Steel for Porcelain Enameling by Photometric Analysis¹

This standard is issued under the fixed designation C 715; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the amount of nickel deposited on sheet steel during its preparation for porcelain enameling. It is a photometric method commonly used on production parts and is suitable for determining the heavier nickel deposits that may be obtained during the processing of steel for one-coat enameling.

NOTES
1—An alternative X-ray emission spectrometry method is Test Method C 810.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
C 810 Test Method for Nickel on Steel for Porcelain Enameling by X-Ray Emission Spectrometry.²
E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals.⁴

3. Significance and Use

3.1 This test method is primarily used to control the nickel dipping operation to ensure that the desired level of nickel deposition is attained. It is also used to prepare test plates used for calibration in Test Method C 810.

4. Apparatus

4.1 Photoelectric Photometer, conforming to Practice E 60.
4.2 Weighted Rubber Ring Assembly, required to confine stripping agents to a definite area, consisting of a molded rubber ring and a metal outer ring. The rubber ring shall have an inside diameter of 1.35 in. (34.3 mm) in order to encircle an area of 0.01 ft² (0.000929 m²), a wall configuration as shown in Fig. 1, and the lower portion beveled at a 45° angle (0.78 rad) to reduce the contact area and ensure a better seal. The metal outer ring shall weigh about 3.5 lb (1.5 kg), suitably machined to fit over the top of the rubber ring as shown in Fig. 1.

4.2.1 The exact area covered by the rubber ring will gradually increase as the rubber ring itself is consumed by the acid reagent used. In the most accurate analysis, the area etched by the rubber ring shall be calculated occasionally, factored against the prescribed area, and that number applied to the reading obtained from the graph.

4.3 Aspirator, consisting of a calibrated 500-mL flask, equipped with a twohole stopper, an aspirator bulb, and a suction tube formed from 0.079-in. (2-mm) inside diameter capillary glass tubing.

5. Reagents and Materials

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.⁵ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Purity of Water—Use distilled or deionized water.
5.3 Ammonium Hydroxide (sp gr 0.90)—Concentrated ammonium hydroxide (NH₄OH).
5.4 Ammonium Persulfate—(NH₄)₂S₂O₈.
5.5 Dimethylglyoxime—Prepare a 1 % solution of dimethylglyoxime in methyl alcohol or a 2.62 % solution of sodium dimethylglyoximate in water (store in a polyethylene bottle).
5.6 Hydrochloric Acid (1+5)—Dilute 1 vol of concentrated hydrochloric acid (HCl, sp gr 1.19) with 5 vol of water.
5.7 Nickel Sulfate, Standard Solution—Dissolve 0.448 g of nickel sulfate (NiSO₄·6H₂O) in water. When the material is completely dissolved, cautiously add 10 mL of concentrated H₂SO₄ and transfer the solution to the 1000-mL flask. When

¹ This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.12 on Materials for Porcelain Enamel and Ceramic-Metal Systems.
³ Annual Book of ASTM Standards, Vol 02.05.
⁴ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Annual Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.
8. Determinations of Nickel Coating Masses

8.1 Sampling a Nickel-Coated Steel Surface

8.1.1 Place the weighted, rubber ring assembly on the nickel-coated metal surface. Add 3 mL of warm (approximately 120°F (50°C)) HNO₃ (1+1). Allow the foaming reaction to proceed for about 10 s for light nickel coatings and about 15 s for heavier nickel coatings. If the warm acid does not react, scratch the steel surface or try another spot. After the acid has foamed for the prescribed time, add 5 mL of HCl (1+5) to stop the foaming reaction. Withdraw the solution from the steel surface with the aspirator into the calibrated flask. Rinse the test area twice with water and retain the washings in the flask.

8.1.2 Alternative methods for determining nickel are described in Test Methods E 30, Sections 62 to 70 for the gravimetric method and Sections 71 to 73 for the volumetric method.

8.2 Analytical Procedure:

8.2.1 Add chemicals in the order given below to the solution in the flask and mix thoroughly after each addition:

<table>
<thead>
<tr>
<th>Additions for 23-mm Optical Path Cell</th>
<th>Amount in Order of Use</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonium hydroxide (sp gr 0.90)</td>
<td>50 mL</td>
</tr>
<tr>
<td>Ammonium persulfate</td>
<td>4 g</td>
</tr>
<tr>
<td>Dimethylglyoxime</td>
<td>10 mL</td>
</tr>
<tr>
<td>Dilute with water to</td>
<td>500 mL</td>
</tr>
</tbody>
</table>

^ Cells with other optical path lengths are available.

8.2.2 Filter a portion of the solution. Discard the first 10 to 20 mL from the filter and collect a sufficient amount of filtrate in the absorption cell for testing. Just before testing, set the wavelength as determined in accordance with Section 7 and adjust the instrument to 100 % transmission with a cell that contains only water. Place the cell containing the test solution in the photometer and read the percent transmission.

9. Calculation and Report

9.1 Refer to the graph developed in Section 7. Determine the mass of nickel that corresponds to the percent transmission reading shown by the photometer. Report the results in grams of nickel per square metre of steel surface.

10. Precision and Bias

10.1 The precision and bias of this test method is believed to be within 0.000929 g/ft² (0.1 g/m²). Exact values are difficult to obtain because of sample inhomogeneity, and the effect of the acid etch on the rubber ring diameter (see 4.2.1).
Standard Test Method for Continuity of Porcelain Enamel Coatings

This standard is issued under the fixed designation C 743; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 Porcelain enamel coatings are applied to metals to provide protection from corrosion as well as to enhance their appearance. This test method permits the easy detection of discontinuities and areas of light coverage, not readily seen by visual inspection, which limit the protection to the substrate. Somewhat similar tests applicable to the thicker glass coatings used for chemical reaction vessels are found in Test Methods C 536 and C 537.

1.2 Values stated in SI units are to be regarded as the standard. Inch-pound units are provided for information only.

1.3 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

C 536 Test Method for Continuity of Coatings in Glassed Steel Equipment by Electrical Testing
C 537 Test Method for Reliability of Glass Coatings on Glassed Steel Reaction Equipment by High Voltage

3. Terminology

3.1 air-gap voltage—the voltage needed to arc through a defect that is open to both the surface of the porcelain enamel and the metal substrate. The length of the air gap is estimated by measuring the enamel thickness.

3.2 overvoltage—the difference between an applied test voltage and the air-gap voltage for the porcelain enamel being tested.

3.3 RMS voltage—the root mean square voltage, or “average” voltage, shown on ac test meters.

3.4 peak voltage—for ac meters, the voltage that will discharge across the air gap or across a defect. The peak voltage is the RMS voltage multiplied by 1.414.

4. Apparatus

4.1 A nondestructive coating thickness gage with a range of at least 0 to 0.5 mm in 0.03-mm increments (0 to 20 mils in 1.0-mil increments).

4.2 Either an ac or a dc high-voltage discharge test instrument with a continuously variable output voltage over the range of at least 0 to 4 kV as indicated on a built-in voltmeter. The instrument shall be capable of being set to 0.1 kV and shall have an insulated cable and probe and a means for indicating (light, meter, or bell) a voltage discharge. Before use in this test, the high-voltage discharge test instrument shall be calibrated in accordance with the procedure set forth in Annex A1.

4.3 A test probe constructed of 100 mesh wire gauze as illustrated in Fig. 1.

5. Test Specimens

5.1 Porcelain enameled specimens of any size may be used in this test. The enamel shall be removed from an edge or the back of the specimen to provide an electrical ground for the test instrument. If the probe is wider than the specimen, the edges of the specimen shall be masked with electrical insulating tape to prevent discharges from occurring at the edges where porcelain coverage may be light.

6. Conditioning

6.1 Before testing, wash the specimens with a 1 weight %, solution of trisodium phosphate on a soft cellulose sponge, rinse with tap water, and then rinse with distilled water. Then dry the specimens thoroughly in air.

7. Procedure

7.1 Measuring the Enamel Thickness—Measure the enamel thickness at a minimum of three locations on the specimen. Make these measurements to the nearest 0.03 mm (1.0 mil) in accordance with the operating instructions for the thickness gage. Use the maximum value obtained as the enamel thickness.

7.2 Selecting the Test Voltage—The test voltage to be used...
depends upon the purpose for conducting the test. The test can be used to determine (1) the presence of defects open to both the enamel surface and the base metal, (2) the location of areas of light coverage in addition to defects open to both the enamel surface and the base metal, or (3) compliance with a specification. According to the user’s purpose for conducting the test, the following three instructions correspond respectively to (1), (2), or (3) above:

7.2.1 Test Method A—For Open Defects:
7.2.1.1 Determine the air-gap voltage that corresponds to the enamel thickness from the calibration curve described in the annex. This value is the test voltage.
7.2.1.2 Probe the specimen in accordance with 7.3.

7.2.2 Test Method B—For Light Coverage and Open Defects:
7.2.2.1 Determine the air-gap voltage that corresponds to the enamel thickness from the calibration curve described in the annex.
7.2.2.2 Arbitrarily select the overvoltage to be used. (Increasingly smaller defects are located with this test as the overvoltage is increased. Therefore, confidence that ware passing this test will have a long, corrosion-free service life is increased as the overvoltage is increased.)
7.2.2.3 Add the selected overvoltage to the air-gap voltage. This value is the test voltage.
7.2.2.4 Probe the specimen in accordance with 7.3.

7.2.3 Test Method C—For Compliance with a Specification—Specifications for continuity of coating will be in terms of d-c overvoltage or peak a-c overvoltage (these are identical). Determination of the test voltage depends on whether a d-c or an a-c test instrument is used.
7.2.3.1 DC Test Instruments—Determine the test voltage in accordance with 7.2.2, substituting the specified overvoltage for the selected overvoltage as in 7.2.2.2 and 7.2.2.3.
7.2.3.2 AC Test Instruments—Divide the specified overvoltage by 1.414, then determine the test voltage as in 7.2.2, substituting the specified overvoltage divided by 1.414 for the selected overvoltage as in 7.2.2.2 and 7.2.2.3.
7.2.3.3 Probe the specimen as shown in 7.3.

7.3 Probing the Specimens:
7.3.1 Connect the ground wire of test instrument to the base metal of the specimen.
7.3.2 Turn on the test instrument.
7.3.3 Set the output voltage (as indicated on the built-in voltmeter) to the value selected in 7.2.
7.3.4 Let the voltage stabilize for 15 s.
7.3.5 Hold the probe with a light pressure at approximately 30° to the surface of the specimen.
7.3.6 Probe the specimen at a rate of 3 to 4 in./s (75 to 100 mm/s).

7.4 Locating Discontinuities:
7.4.1 When the probe passes over a discontinuity, a spark will arc across the discontinuity to the ground provided by the base metal. This spark can be seen and heard. In addition, most instruments are equipped with an auxiliary light that flashes when a discontinuity is located. Some instruments also have an audible signal or digital counter to aid in the detection of discontinuities.
7.4.2 When a discontinuity is located, a failure in the coating is present.

8. Report
8.1 The report shall include the following:
8.1.1 Title of test, ASTM designation, and issue.
8.1.2 Enamel thickness,
8.1.3 Air-gap voltage,
8.1.4 Method of selecting test voltage (Test Method A, B, or C),
8.1.5 Overvoltage used,
8.1.6 Name and model of test equipment,
8.1.7 Whether or not discontinuities were present,
8.1.8 Type of specimen tested,
8.1.9 Number of specimens tested, and
8.1.10 Type of enamel tested.

9. Precision and Bias

9.1 No statement is made about either the precision or bias of this test method since the result merely states whether there is conformance to the criteria for success specified in the procedure.

ANNEX
(Mandatory Information)

A1. CALIBRATING THE TEST INSTRUMENTS

A1.1 Even though each test instrument is equipped with a built-in voltmeter, the discharge voltage across a given air gap will vary from instrument to instrument. Therefore, each instrument must be calibrated by determining its air-gap voltage curve.

A1.2 Specimens Required—Three specimens of each of 15 enamels are required to determine the air-gap voltage for the test instrument. These 15 enamels shall spread over the range of thicknesses from 0.1 to 0.5 mm (4 through 18 mils) as uniformly as possible. These specimens shall be prepared as outlined in 4.1 and 5.1.

A1.3 Procedure:
A1.3.1 Puncturing the Enamel Coating:
A1.3.1.1 Connect the ground wire of the test instrument to the base metal of the specimen.
A1.3.1.2 Turn on the test instrument.
A1.3.1.3 Raise the output voltage of the test instrument to its maximum and allow it to stabilize for 15 s.
A1.3.1.4 Hold the probe with a light pressure at approximately 30° to the surface of the specimen.
A1.3.1.5 Probe the specimen at a rate of 75 to 100 cm/s (3 to 4 in./s).
A1.3.1.6 Discard all specimens that are not punctured by this treatment.
A1.3.2 Reprobing the Specimens:
A1.3.2.1 Reprobe the specimens that were punctured by the high-voltage probe in a manner similar to that outlined in A1.3.1.1 through A1.3.1.4, except that the test voltage shall be 100 V initially and shall be increased in increments of 100 V until a discharge occurs. A15-s stabilization period shall be observed for each increase in voltage before reprobing the test specimens. This procedure may be expedited by starting the reprobing of duplicate specimens at 300 Volts lower than the breakdown voltage noted for the first reprobed specimen, then increasing the voltage by increments of 100 V. The discharge voltage determined in this manner is defined as the air-gap voltage.

A1.4 Measuring the Enamel Thickness—Measure the enamel thickness at three locations on the surface of the specimen. Make these measurements close to the area where the discharge occurred. Make these measurements to the nearest 0.025 mm (1.0 mil) in accordance with the operating instructions for the thickness gage. Average the three measurements to give the average enamel thickness which is assumed equivalent to the length of the air gap.

A1.5 Calculations—Calculate the average enamel thickness and air-gap voltage for each specimen. Plot these averages and fit the least squares line (of the form $y = a + bx$) through these averages as follows:

$$ a = \frac{\sum y - \sum x \sum xy}{N \sum x^2 - (\sum x)^2} $$

$$ b = \frac{N \sum xy - \sum x \sum y}{N \sum x^2 - (\sum x)^2} $$

where:

- $a$ = intercept,
- $b$ = slope,
- $N$ = number of specimens,
- $x$ = average thickness of an enamel, mils, and average air gap voltage of an enamel, kV.

The resulting curve is the calibration curve.

Note A1.1—The equations given here are for hand computation or desk calculator use only. If the least squares line is fitted by digital computer, any accurate library program may be used.
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Designation: C 756 – 87 (Reapproved 1999)

Standard Test Method for Cleanability of Surface Finishes

This standard is issued under the fixed designation C 756; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the numerical evaluation of the ease or difficulty of cleaning soiled surface finishes. This test method is applicable to all surface finishes not affected by water.

1.2 Values given in SI units are to be regarded as the standard. Inch-pound units are provided for information only.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

C 282 Test Method for Acid Resistance of Porcelain Enamels (Citric Acid Spot Test)

C 614 Test Method for Alkali Resistance of Porcelain Enamels

3. Summary of Test Method

3.1 The test method consists of applying an exact amount of a fluorescent water-soluble soiling agent to a specimen surface and then cleaning the surface with a reproducible machine-wiping technique. The soil remaining on the specimen after wiping is extracted with a water solvent and the fluorescence of the solution measured. A standard reference surface is treated in a similar manner. The cleanability index of the surface under test is expressed as the ratio of the fluorescence of the solutions extracted from the test surface and from the standard reference surface. Cleanability indexes greater than 1.0 indicate that the test surface is more difficult to clean than the standard reference surface, whereas indexes less than 1.0 indicate that the test surface is more easily cleanable than the standard of comparison.

3.2 The soiling agent used consists of polyethylene glycol, a black dye, and a fluorescent tracer, each of which is readily water soluble.

4. Significance and Use

4.1 This test method was developed to guide the user in selecting a finish coating or material that is resistant to soiling in a particular application.

4.2 The numerical values derived by this test method enables the user to rank finish coatings and materials in regard to soil retention or ease of soil removal.

5. Apparatus

5.1 Motor-Driven Lapping Plate, 203-mm (8-in.) diameter, speed 163 r/min.

5.2 Automatic Polishing Unit, 11-s cycle, adjustable to 48-mm (1 3/4-in.) stroke.

5.3 Hypodermic Syringe, glass, 2-mL capacity, without needle.

5.4 Repeating Pipet, 0.025 mL (25 µl) capacity.

5.5 Repeating Pipet, 10-mL capacity.

5.6 Desiccator approximately 254 mm (10 in.) diameter.

5.7 Cleaning Head, brass, 57 mm (2 1/4 in.), with worm-driven hose clamp for attachment of cleaning tissues (Fig. 1).

5.8 Soiling Head, brass, 25 mm (1 in.) diameter, with 25-mm (1-in.) diameter facing of polytetrafluoroethylene attached to a water-proof household cement (Fig. 1).

5.9 Extraction Cell, fitted with a fluorosilicone O-ring, size 3.2 by 57 mm (1/8 by 2 1/4 in.) (Fig. 2).

5.10 Fluorometer, for measuring the fluorescence of solutions.

5.11 0.9 Neutral Density Filter, 50.8 by 50.8 mm (2 by 2 in.) glass mounted (transmission 12.5 %).

5.12 Beakers, borosilicate, 100-mL capacity.

1 Suitable lapping plates are available from Buehler Ltd., 2120 Greenwood St., Evanston, IL 60204, Struers, Inc., 20102 Progress Drive, Cleveland, OH, 44136; or other Metallurgical Supply Sources.

2 An Olsen “S.M.” Automatic Polisher has been found suitable and is available under the code name OLPOL from Struers, Inc., 20102 Progress Drive, Cleveland, OH 44136.

3 O-rings must be fluorosilicone polymer; consult Precision Associates, 742 N. Washington Ave., Minneapolis, MN 55401; Parker Seal Co., 10567 W. Jefferson Blvd., Culver City, CA 90230; & B. W. Rogers (Parker Dis.) 1000 Brookpark Rd., Cleveland, OH 44109. Also see other sources in Thomas Register under SEALS: “O” RINGS. A suitable device for clamping the cell to a specimen is shown in Fig. 3 and Fig. 5.

6. Reagents and Materials

6.1 Cleaning Tissues, approximately 127 by 229 mm (5 by 9 in.).

6.2 Potassium Carbonate.

6.3 Polyethylene Glycol, 400.

6.4 Uranine, water-soluble, USP.

6.5 Keco Acid Black, B(F101).

6.6 Ethyl Alcohol.

6.7 Mild Household Detergent, liquid.

7. Test Specimens

7.1 The preferred specimen size is 114 mm (4 1⁄2 in.) square, but any other size or shape may be used provided it can be rotated on the lapping wheel and the smaller dimension is between 102 and 127 mm (4 and 5 in.). Sharp or jagged edges shall be filed or honed to prevent snagging of the cleaning tissues during the mechanical cleaning process.

7.2 Twelve specimens are required to evaluate the cleanability index, that is, six of the candidate surface, and six of a porcelain enamel standard reference surface for cleanability. Porcelain enamel specimens are not damaged by the cleanability test and may be cleaned and reused many times. Specimens have been reused as many as 50 times without damage or solid buildup.

8. Preparation of Standard Soil

8.1 The soiling agent shall consist of the following:

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Weight %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyethylene glycol</td>
<td>98</td>
</tr>
<tr>
<td>Keco Acid Black B</td>
<td>1</td>
</tr>
<tr>
<td>Uranine, water-soluble</td>
<td>1</td>
</tr>
</tbody>
</table>

8.2 The urine shall be added to the polyethylene glycol in food blender and mixed by alternate periods of mixing and standing until the crystalline urine appears to be completely dissolved in the glycol. The black dye which obscures all else shall be added last and thoroughly mixed in the blender. This soiling agent shall be stored in a tightly stoppered glass bottle at least overnight before use.

9. Procedure

9.1 Specimen Pretreatment—Scrub the specimens with a cellulose sponge wet with a 1% solution of a mild household detergent at room temperature. Rinse in turn with tap water, distilled water, and ethyl alcohol, and allow to dry in a near-vertical position at room temperature. Store the washed and dried specimens overnight in a desiccator charged with a saturated solution of potassium carbonate.

9.2 Conditioning of Cleaning Tissues—Cut a supply of tissues ample for use in specimen cleaning treatment in the following paragraph into approximately 102-mm (4-in.)
squares and store overnight, before use, in a desiccator charged with a saturated solution of potassium carbonate (relative humidity approximately 45%). Allow the tissues to remain in the desiccator until just prior to use.

9.3 Soiling and Cleaning Treatments:

9.3.1 Lay out six specimens face up on a table. Homogenize the soiling agent by up-ending several times before use. Fill the hypodermic syringe with no needle attached with the soiling agent by withdrawing the plunger. Expel this charge of soil back into the soil bottle. Then fill the syringe again. Wipe the tip of the syringe with a cleaning tissue and expel several drops into the soil bottle. Then, without rewiping the tip, hold the syringe vertically over a specimen and discharge one drop near the center of each of the six specimens. Center the first specimen on the lapping wheel and hold it in place by means of adhesive tape across the specimen corners. Place the polytetrafluoroethylene-faced soiling head on the soil, and engage the spindle (Fig. 4) of the polishing unit. Operate the lapping wheel and the polishing unit for 1 min to distribute the soil over the central portion of the specimen (Fig. 5). Remove the soiling head and clean it with tissue for reuse. Adjust the spindle so that it rests over the center of the soil spot. Prepare two cleaning heads 57 mm (2 1/4 in.) diameter) by covering with four thicknesses of cleaning tissues clamped smoothly in place. With the repeating pipet expel exactly 0.021 mL of distilled water at the center of the tissue on the first cleaning head.

9.3.2 Immediately place the cleaning head with tissue side against the soiled specimen, engage the spindle, and operate for 22 s (so that it will stop as well as start at the center of the specimen). Remove the cleaning head without sliding motion. Repeat the cleaning operation with the second tissue-covered head, using the same amount of distilled water and for the same 22-s period.

9.3.3 Store the first soiled and cleaned specimen in a near vertical position in a rack while applying exactly the same series of soiling and cleaning treatments to the five remaining specimens.

9.4 Water Extraction of the Soil Retained—Remove the small, often nonvisible, amounts of soil from the central portion of the soiled and cleaned specimens with 10 mL of distilled water in the O-ring sealed extraction cell by the following sequence of operations within 30 min after soiling; place the first soiled and cleaned specimen, face up, in the center of the cell clamping frame. Place a clean and dry extraction cell (Fig. 2) on the center of the specimen so that the O-ring defines the area to be extracted (Fig. 5). Clamp the cell in the frame with a light pressure from the wing nuts; introduce 10 mL of distilled water with the 10-mL repeating pipet, swirl...
the water in the cell momentarily and let stand for 3 min, give
the solution in the cell another swirl and pour out into a clean
and dry 100 mL beaker; transfer a portion of the extract to a
clean, dry cuvette and place the cuvette in a rack until the other
five extracts are ready for measurement in the fluorometer.

9.5 Measurement of Fluorescence—Operate the fluorometer
according to the manufacturer’s instructions. Measure and
record the fluorescence of a distilled water blank. Measure and
record in turn the fluorescence of the solutions extracted from
the specimens.

NOTE 2—If concentrated solutions give off-scale readings, a 0.9
neutral-density filter should be inserted between the fluorescing solution
and the photomultiplier. This filter transmits only 12.5% of the light
emitted by the solution, reducing the fluorescence readings and bringing
them on scale. Redetermine the blank reading with the filter in place and
multiply the reduced reading by 8.0 which is 1 divided by the 12.5% transmission of the neutral density filter.

9.6 Standard Reference Surface—Apply the test procedure
outlined in 7.1 through 7.5 to the Standard Reference Surfaces
at least once during each testing day to obtain the average
fluorescence of solutions extracted from these specimens.

9.7 Preferred Methods of Equipment Cleaning:
9.7.1 The successful use of the fluorimetric analytical tech-
niques employed in this procedure demands that a scrupulous
level of cleanliness be maintained throughout. An oily thumb-
print on the glass cuvette containing the solution to be
measured may be more fluorescent than the unknown.

9.7.2 Wash the glass beakers and the extraction cells before
and after use by brushing in a warm detergent solution; rinse
copiously with flowing tap water and then with distilled water.
The glass beakers may be oven dried. The extraction cells,
without O-ring removal, may be wiped dry with clean tissue.
Clean the syringe used for dispensing uniform amounts of soil
on the specimens immediately following use with tap water,
distilled water and alcohol, and dry the parts with tissue. Give
the glass cuvettes used for measuring fluorescent solutions
several rinses with tap water and then alcohol, and allow to
drain in an inverted position in a holding rack. Discard the
tissue for wiping glassware and other equipment after one use.

10. Calculation of a Cleanability Index
10.1 Subtract the measured fluorescence value for the dis-
tilled water blank from the measured fluorescence value for
each of the six test surfaces and the six standard reference
surfaces. This will result in corrected fluorescence values for
the six test surfaces and for the six standard reference surfaces.

10.2 Calculate the average of the corrected fluorescence
values for the six test surfaces and for the six standard
reference surfaces.

---

**FIG. 3 Device for Clamping Extraction Cell to Specimen**

<table>
<thead>
<tr>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
<th>G</th>
<th>H</th>
<th>I</th>
<th>J</th>
<th>K</th>
<th>L</th>
<th>M</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>in.</td>
<td>1/16</td>
<td>2 1/4</td>
<td>1 1/4</td>
<td>1 1/4</td>
<td>1 7/8</td>
<td>5 1/4</td>
<td>6 1/4</td>
<td>3 1/4</td>
<td>1 1/4</td>
<td>4 1/4</td>
<td>4 1/4</td>
<td>4 1/4</td>
<td>5</td>
</tr>
<tr>
<td>(mm)</td>
<td>(1.6)</td>
<td>(64)</td>
<td>(35)</td>
<td>(6.4)</td>
<td>(47)</td>
<td>(8)</td>
<td>(127)</td>
<td>(152)</td>
<td>(32)</td>
<td>(83)</td>
<td>(6.4)</td>
<td>(117)</td>
<td>(117)</td>
</tr>
</tbody>
</table>
10.3 Calculate the coefficient of variation of the corrected fluorescence values for the six test surfaces and for the six standard reference surfaces.

10.4 Repeat the cleanability determinations on all six specimens (either the test specimens or the six standard reference specimens) if the coefficient of variation is greater than 20%. 

Metric Equivalents

<table>
<thead>
<tr>
<th>Metric</th>
<th>in.</th>
<th>(mm.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>10</td>
<td>254</td>
</tr>
<tr>
<td>B</td>
<td>1/8</td>
<td>13</td>
</tr>
<tr>
<td>C</td>
<td>1/4</td>
<td>6.4</td>
</tr>
</tbody>
</table>

FIG. 4 Spindle for Polisher

FIG. 5 Equipment Used in Soiling and Cleaning Specimens
10.5 The cleanability index is calculated as follows:

\[ CI = \frac{Ft}{Fr} \]  
(1)

where:
- \( CI \) = the cleanability index of the surface being tested,
- \( Ft \) = the average corrected fluorescence of the test surfaces as calculated in 10.2, and
- \( Fr \) = the average corrected fluorescence of the standard reference surfaces as calculated in 10.3.

10.6 The method of calculation is shown in the Appendixes.

11. Precision and Bias

11.1 The precision of this test method, when performed by the same operator in the same laboratory, should be sufficient so that the cleanability determination on six standard references should show a coefficient of variation no greater than 20%. No standard is available by which the bias of the test method can be determined.

APPENDIXES

(Nonmandatory Information)

X1. INITIAL CALCULATIONS

X1.1 The four calculations necessary on the data for the standard reference surfaces and the test surfaces before the cleanability index can be determined are:

X1.1.1 Correcting fluorescence measurements:

Test Surface
\[ CFT = FTS - FB \]  
(X1.1)

where:
- \( CFT \) = corrected fluorescence measurement for the test surface,
- \( FTS \) = measured fluorescence of the test surface, and
- \( FB \) = measured fluorescence of the distilled water blank.

Standard Reference Surface
\[ CFS = FSR - FB \]  
(X1.2)

where:
- \( CFS \) = corrected fluorescence measurement for the standard reference surface,
- \( FSR \) = measured fluorescence of the standard reference surface, and
- \( FB \) = measured fluorescence of the distilled water blank.

X1.1.2 Determining average corrected fluorescence:

Test Surface
\[ Ft = \frac{\Sigma CFT}{N} \]  
(X1.3)

where:
- \( Ft \) = average corrected fluorescence of the test surfaces,
- \( \Sigma CFT \) = sum of the corrected fluorescence measurements for the test surfaces, and
- \( N \) = number of test surfaces (6).

Standard Reference Surface
\[ Fr = \frac{\Sigma CFS}{N} \]  
(X1.4)

where:
- \( Fr \) = average corrected fluorescence of the standard surfaces,
- \( \Sigma CFS \) = sum of the corrected fluorescence measurements for the standard reference surfaces, and
- \( N \) = number of standard reference surfaces (6).

X1.1.3 Determining standard deviation:

Test Surface
\[ STS = \sqrt{\frac{\Sigma CFT^2 - (\Sigma CFT)^2/N}{N-1}} \]  
(X1.5)

where:
- \( STS \) = standard deviation of the corrected fluorescence measurements for the test surface,
- \( \Sigma CFT^2 \) = sum of the squares of the corrected fluorescence measurements of the test surfaces,
- \( (\Sigma CFT)^2 \) = square of the sums of the corrected fluorescence measurements for the test surfaces, and
- \( N \) = number of test surfaces (6).

Standard Reference Surface
\[ SRS = \sqrt{\frac{\Sigma CFS^2 - (\Sigma CFS)^2/N}{N-1}} \]  
(X1.6)

where:
- \( SRS \) = standard deviation of the corrected fluorescence measurements for the test surface,
- \( \Sigma CFS^2 \) = sum of the squares of the corrected fluorescence measurements of the standard reference surfaces,
- \( (\Sigma CFS)^2 \) = square of the sums of the corrected fluorescence measurements for the standard reference surfaces, and
- \( N \) = number of standard reference surfaces (6).

X1.1.4 Determining coefficient of variation:

Test Surface
\[ CVT = \frac{STS}{Fr} \times 100 \]  
(X1.7)

where:
- \( CVT \) = coefficient of variation for the test surfaces,
- \( STS \) = standard deviation for the test surfaces, and...
**X2. CLEANABILITY INDEX CALCULATIONS**

X2.1 Once the four calculations in Appendix X1 have been made, the cleanability index may be determined by the following:

\[
CI = \frac{F_t}{F_r}
\]  

(X2.1)

where:

**X3. SAMPLE CALCULATION OF CLEANABILITY INDEX**

X3.1 *The Measured Values:*

<table>
<thead>
<tr>
<th>Material</th>
<th>Measured Fluorescence Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test surface</td>
<td></td>
</tr>
<tr>
<td>Specimen number</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>46.5</td>
</tr>
<tr>
<td>2</td>
<td>38.0</td>
</tr>
<tr>
<td>3</td>
<td>43.0</td>
</tr>
<tr>
<td>4</td>
<td>40.0</td>
</tr>
<tr>
<td>5</td>
<td>52.0</td>
</tr>
<tr>
<td>6</td>
<td>39.1</td>
</tr>
<tr>
<td>Standard reference surface</td>
<td></td>
</tr>
<tr>
<td>Specimen number</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>32.5</td>
</tr>
<tr>
<td>2</td>
<td>37.5</td>
</tr>
<tr>
<td>3</td>
<td>30.5</td>
</tr>
<tr>
<td>4</td>
<td>36.5</td>
</tr>
<tr>
<td>5</td>
<td>39.0</td>
</tr>
<tr>
<td>6</td>
<td>36.0</td>
</tr>
<tr>
<td>Distilled water blank</td>
<td>2.0</td>
</tr>
</tbody>
</table>

X3.2 Subtract the measured fluorescence value for the distilled water blank from the measured fluorescence values of the test surface and the standard reference surface.

<table>
<thead>
<tr>
<th>Test Surface</th>
<th>Corrected Fluorescence Value</th>
<th>Standard Reference Surface</th>
</tr>
</thead>
<tbody>
<tr>
<td>Measured</td>
<td>Corrected</td>
<td>Measured</td>
</tr>
<tr>
<td>Value</td>
<td>Fluorescence Value</td>
<td>Fluorescence Value</td>
</tr>
<tr>
<td>46.5</td>
<td>44.5</td>
<td>32.5</td>
</tr>
<tr>
<td>38.0</td>
<td>36.0</td>
<td>37.5</td>
</tr>
<tr>
<td>43.5</td>
<td>41.5</td>
<td>30.5</td>
</tr>
<tr>
<td>40.0</td>
<td>38.0</td>
<td>34.5</td>
</tr>
<tr>
<td>52.0</td>
<td>50.0</td>
<td>39.0</td>
</tr>
<tr>
<td>39.1</td>
<td>37.1</td>
<td>36.0</td>
</tr>
</tbody>
</table>

X3.3 To calculate the average corrected fluorescence values:

X3.3.1 Compute the sum of the six corrected fluorescence values for both the test surfaces and the standard reference surfaces.

<table>
<thead>
<tr>
<th>Test Surface</th>
<th>Standard Reference Surface</th>
</tr>
</thead>
<tbody>
<tr>
<td>44.5</td>
<td>30.5</td>
</tr>
<tr>
<td>36.0</td>
<td>35.5</td>
</tr>
</tbody>
</table>

X3.4 To calculate the standard deviation of the corrected fluorescence values:

X3.4.1 Compute the sum of the squares of the six corrected fluorescence values for the test surfaces and the standard reference surfaces.

X3.4.2 Square the sums computed in X3.3.1 for the six test surfaces and the six standard reference surfaces and divide each sum by 6.

\[
(247.1)^2/6 = 61.05841666 \approx 10.17640
\]

**X3.4.3** Subtract the value obtained in X3.4.2 from the value obtained in X3.4.1 for both the test surfaces and the standard reference surfaces.
X3.4.4 Divide the value obtained in X3.4.3 by 5 for both the test surfaces and the standard reference surfaces.

Test Surface | Standard Reference Surface
---|---
142.51 | 51.33

X3.4.5 Take the square root of the values obtained in X3.4.4 for the test surfaces and the standard reference surfaces.

\[ \sqrt{28.50} = 5.34 \quad \sqrt{10.27} = 3.20 \]

X3.5 To calculate the coefficient of variation of the corrected fluorescence values:

X3.5.1 Divide the values obtained in X3.4.5 by those obtained in X3.3.2 for both the test surfaces and the standard reference surfaces and multiply the product by 100.

Test Surface (X3.2) | Standard Reference Surface
---|---
5.34/41.18 = 13.0 % | 3.20/33.33 = 9.6 %

X3.5.2 If the values in X3.5.1 are under 20 % proceed with the calculation of the Cleanability Index. X3.6 If the values in X3.5.1 are over 20 %, repeat the cleanability determinations on all six surfaces where indicated.

X3.6 Calculation of Cleanability Index—Calculate the cleanability index by dividing the average corrected fluorescence value of the test surfaces (X3.3.2) by the average corrected fluorescence values of the standard reference surface (also in X3.3.2).

\[ \text{Cleanability index} = \frac{41.18}{33.33} = 1.24 \]
Standard Test Method for Yield Strength of Enameling Steels After Straining and Firing

1

This standard is issued under the fixed designation C 774; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers determination of the yield strength of steel specimens after simulated forming and enamel firing operations.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
   A 370 Test Methods and Definitions for Mechanical Testing of Steel Products
   E 8 Test Methods for Tension Testing of Metallic Materials

3. Definition

3.1 quarter lines—imaginary lines parallel to the direction of rolling, positioned at a distance from the sheet mill edge equal to one quarter of the sheet width.

4. Summary of Test Method

4.1 Representative sheet-steel specimens are selected, strained in tension to simulate forming, heat treated to simulate enamel firing, and tension tested for determination of yield strength.

5. Significance and Use

5.1 In the manufacture of porcelain enameled ware, sheet steel is subjected to forming operations and subsequently to firing of the enamel at temperatures, typically, of 1400 to 1550°F (760 to 844°C). Some steels used for porcelain enameling are subject to grain growth in critically strained areas resulting in loss of strength.

5.2 This may lead to easy deformation of the steel and damage to the porcelain enamel coating.

5.3 This test method may be correlated with transit or use tests to evaluate the suitability of steel for porcelain enameled ware.

6. Apparatus

6.1 Shear for cutting blanks.
   6.2 Equipment for cutting or machining tension specimens.
   6.3 Gage, 2-in. (50.8-mm), for marking tension specimens.
   6.4 Tension-Testing Equipment, as described in Test Method A 370.
   6.5 Specimen-Supporting Rack, slotted to support specimens on edge spaced 1 in. (25.4 mm) apart.
   6.6 Furnace, capable of heating the test specimens and their supporting rack to the firing temperature in 2 min.

7. Reagents and Materials

7.1 Sheet Steel, sufficient in size to provide the test specimens, described in Section 8.
   7.2 Solvent, such as acetone or isopropyl alcohol.
   7.3 Hydrochloric Acid (1+1)—Dilute 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 1 volume of water.

8. Sampling

8.1 Obtain ten test samples, each approximately 1 by 9 in. (25 by 229 mm), from within the quarter lines of the sheet sample, the long dimension being in the direction of rolling.

9. Procedure

9.1 Remove burrs from the cut edges of the test samples.
   9.2 Mark 2-in. (50.8-mm) gage lengths in the middle of the test samples and strain in tension. Maintain a minimum of 1 in. (25.4 mm) of length between the grips of the tension-testing machine and the gage marks.
   9.3 Elongate duplicate test samples to nominal strain levels of 0, 8, 12, 16, and 20 %. Measure the elongation with extensometers, dividers, or other means.
   9.4 Remove the test samples from the tension machine and measure actual strain to the nearest 0.01 in. (0.25 mm).
   9.5 Clean the strained test samples with the solvent and fire in air atmosphere at 1450 ± 10°F (788 ± 5°C) for 4 min. Test samples may be hung from hooks or supported on edge spaced
1 in. (25.4 mm) apart (if the latter, be sure to support in a manner to prevent sagging during firing). The test samples should reach the firing temperature within $2.0 \pm 0.5$ min.

9.6 Remove the samples from the furnace and air cool.

9.7 Descale the fired samples in a HCl (1+1) solution containing a few drops of an inhibitor, such as formaldehyde, at a temperature of 70 to 100°F (21 to 38°C). Promptly rinse the test samples and dry.

9.8 Machine the descaled test samples to make standard rectangular tension tests specimens in accordance with Test Methods and Definitions A 370. The specimens shall have a minimum length of 8 in. (204 mm) and the sides of the reduced section shall be parallel.

9.9 Measure the thickness and width of each specimen, and test for yield strength as described in Test Methods E 8 at a crosshead speed of 0.10 in. (2.5 mm)/min. Determine the yield strength at the lower point or by the 0.5 % extension under load.

10. **Report**

10.1 Report the yield strengths determined for the duplicate specimens at each of the five strain levels.

10.2 Report the actual strain imparted to each specimen.

11. **Precision and Bias**

11.1 The precision and bias of yield strength and strain in this test method would be identical to the precision and bias of Test Methods E 8. Refer to this standard for up-to-date precision and bias.
Standard Test Method for Nickel on Steel for Porcelain Enameling by X-Ray Emission Spectrometry

This standard is issued under the fixed designation C 810; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of the amount of nickel deposited on sheet steel during its preparation for porcelain enameling. It is an X-ray emission method used for testing sample panels or certain commercial parts.

Note 1—An alternative wet chemical method is Test Method C 715.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For a specific hazards statement, see Section 7.

2. Referenced Documents

2.1 ASTM Standards:
   C 715 Test Method for Nickel on Steel for Porcelain Enameling by Photometric Analysis

3. Summary of Test Method

3.1 Steel samples coated with a light nickel deposit are inserted in the sample position of an X-ray spectrometer. The count rate for nickel is measured and converted by means of a calibration curve to g/m² (g/ft²).

Note 2—1 m² = 10.75 ft². Industry usage is typically in mixed units, grams per square foot. For example, 0.10 g/ft² equals a little more than 1 g/m².

4. Significance and Use

4.1 This test method is an accurate and rapid means for measuring nickel deposits on steel sample plates and such parts that can be fitted into the X-ray spectrometer. Its accuracy extends over a wide range of nickel deposits.

5. Interferences

5.1 There are no interferences from other elements present. However, low values can result from absorption of the X rays by overlaying material. Grease on the sample or rust due to storage in humid areas are examples of such material. Low results are also obtained on de-enamelled samples because the nickel deposit is converted to a nickel iron alloy at enameling temperatures. The presence of the iron in the alloy layer absorbs some of the X radiation and accounts for the lower result.

6. Apparatus

6.1 Suitable X-Ray Emission Spectrometer complete with 50-kV power supply goniometer, detector with pressure-regulated gas flow attachments, scaler-counter, lithium fluoride analyzing crystal, and 0.02° Soller slit collimator is required. About a 1-in. (25.4-mm) diameter area of the sample is irradiated.

6.2 Special Sample Holder (Fig. 1), to permit insertion of a 2 by 2-in. (51 by 51-mm) flat corner of a large flat sample. Alternatively, the standard sample holder supplied with the equipment may be used, but the sample must be cut to 1.5 by 1.25 in. (38 by 32-mm).

6.3 Steel Sheets with various amounts of nickel deposits are required for calibration and standardization.

6.4 Nickel-Base Alloy Sample, such as 18-8 stainless steel, for routine calibration.

7. Hazards

7.1 Equipment should be periodically checked for radiation leaks to ensure against exposure to X radiation.

8. Calibration and Standardization

8.1 Prepare approximately 18 standard plates by cleaning and pickling 4 by 6 in. (102 by 152 mm) commercial enameling iron stock (any steel used in commercial enameling operations may be used) and applying nickel in the conventional manner for varying treatment times to give a range of nickel deposition from 0 to 3 g/m² (0 to 0.4 g/ft²).

---


2. Annual Book of ASTM Standards, Vol 02.05.

3. Available from the following: (1) Philips Electronic Instruments, 750 S. Fulton Ave., Mount Vernon, NY 10550; (2) Siemens Corp., Medical Industrial Div., 186 Wood Ave., South, Iselin, NJ 08831; and (3) Diano Corp., X-ray Div., 2 Lowell Ave., Winchester, MA 01890.
8.2 Prepare a parer mask which, when placed over each plate, will show the areas measured by the X-ray spectrometer. The mask is used later to indicate the same areas for wet determination of nickel deposition by Test Method C 715. In this way, comparative data are obtainable by both measurement methods on the same areas of the standard plates.

8.3 The spectrometer is set to the K-alpha line of nickel at 1.66 Å and the X-ray intensity in counts/s is scaled for each measurement. A reference plate of Type 321 stainless steel (Cr 18, Ni 10) is measured before and after each measurement on the standard plates. The two counts on the reference plate are averaged.

8.4 The numbered standard plates are measured on one side at two areas, top and bottom, and are corrected for instrument drift by relating the count rates on the standard plates to a fixed average count rate on the reference plate.

8.5 On the basis of X-ray count data, ten standard plates are selected for the best distribution of counts covering the full range of nickel on the plates. Nickel deposition on the ten selected plates is then determined at one area on each plate by Test Method C 715. The wet results are then plotted versus X-ray count rate on linear graph paper and a smooth curve drawn through the plotted points. The curve may be used to prepare a chart which lists count ranges for each increment of 0.1 g/m² (0.01 g/ft²) nickel.
9. Procedure

9.1 Standardization of Equipment—Insert the reference standard (6.4) which has a known X-ray count determined when equipment was standardized, in the special sample holder. After the equipment is warm, set the voltage to obtain X-ray counts characteristic of the reference standard.

9.2 Nickel Determination:

9.2.1 Insert a 4 by 6-in. (10 by 152-mm) sample in the special sample holder (or cut a piece to fit a regular holder).

9.2.2 Using an X-ray tube with suitable target, adjust the power supply to provide 50 kV at 25 mA and set the scaler for a 10-s count. Align the spectrogoniometer to 48.65°, 2-θ angle for nickel determination using a lithium fluoride crystal. Start the apparatus and record the count from the counter scales.

9.2.3 Repeat the determination on a minimum of two areas on each side of each sample. Average the counts so recorded and read the nickel concentration from the standard plot.

9.2.4 Specific details of operation of the X-ray apparatus are not included herein due to the complexity of such equipment and the slight variations in procedure between different types of apparatus. These details can be provided by the manufacturer.

9.3 Verification of Standard Equipment—After completing determinations of unknown samples, recheck the machine calibration by reinserting the reference standard and checking the reading.

10. Report

10.1 Convert X-ray counts to grams per square metre by using the calibration curve.

Note 3: The result in grams per square metre can be converted to grams per square foot by dividing by 11.

11. Precision and Bias

11.1 The precision and bias of this test method is being established.
INTRODUCTION

Many methods and tests have been used in the industry to determine the relative compressive stress of porcelain enamel ground coats and cover coats. Three methods have been most commonly used. They are loaded-beam, warp, and split-ring methods. In the loaded-beam method the measure of stress is the load required to bring a specimen back to a plane after it has been bowed by controlled application of the enamel under test to one side of the specimen. The degree of the deflection of a specimen from a plane caused by controlled application of enamel to one side is the indicating measure of stress in the warp test. The indicating measurement in the split-ring test is the force required to just open a split ring after controlled application of test enamel to the outside surface of a ring.

1. Scope

1.1 This test method covers the measurement of the compressive stresses (Note 1) developed by fired porcelain enamels using the loaded-beam method.

Note 1—Although some may interpret the calculations that are used in this test method as indicating compressive load, it is commonly referred to as compressive stress within the porcelain enamel industry.

1.2 This test method is limited to the use of the loaded-beam method. However, this method includes charts (Fig. 1 and Fig. 2) that provide for conversion of loaded-beam test results to warp and ring stress values.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

A 424 Specification for Steel, Sheet, for Porcelain Enameling

3. Terminology

3.1 Description of Term Specific to This Standard

3.2 compressive stress or stress—a term used to designate the degree of compression that exists in a porcelain enamel on steel after processing.

4. Significance and Use

4.1 All porcelain enamel coatings or glass coatings are by necessity under some degree of compression at room temperature. The desired degree of compression or stress depends upon the type of ware and the end use of the item. Some method of determining relative compressive stress of enamels is necessary to establish the suitability of an enamel for a proposed application.

5. Apparatus

5.1 Furnace, suitable for simultaneous firing of at least six 1 by 12-in. (25 by 305-mm) specimens in vertical hanging position.

5.2 Firing Rack, suitable for furnace.

5.3 Loaded-Beam Test Apparatus (see Fig. 3 and Fig. 4).

5.4 Metric Weights, slotted, 1 to 500 g.

5.5 Laboratory Balance, accurate to 10 mg with necessary weights (1 to 100 g).

5.6 Brushing Template, 2/16 by 10 3/16 in. (52 by 278 mm) (see Fig. 5 for one example of achieving the desired end result).

5.7 Stencil Brush.

5.8 Fluorescent Light Fixture with diffusion panel and adjustable support.
6. Test Specimens

6.1 Six specimens are required for each enamel tested. Specimens 1 by 12 in. (25 by 305 mm) shall be cut by shearing from flat sheets of 20-gage Commercial Quality Type II enameling iron (see Specification A 424) with thickness $0.036 \pm 0.002$ in. (0.91 $\pm$ 0.05 mm). The 12-in. dimension should be cut perpendicular to the direction of rolling of sheet. Care should be exercised to prevent formation of burrs during shearing. Burrs that are formed may be removed by carefully filing edges or by lightly touching edges to a sanding belt. Since opposing sides of the sheet may produce slightly different results, one side of the sheet should be scribed in such a manner that the scribe mark will appear about 1 in. from one end of the resulting strips after shearing. A $\frac{3}{8}$-in. (6.35-mm) diameter hole should be punched in one end of the specimen for hanging purposes during drying and firing. The center of the hole should be on the center line $\frac{3}{4}$ in. (11 mm) from the end of the specimen. Specimens should be numbered for identification during the testing procedure. They may be permanently identified by metal stamping on the scribed side while being supported on an anvil (see Fig. 6).

7. Procedure

7.1 Application of Ground Coat to Test Strips:

7.1.1 Subject the specimens to a pickle procedure normal for ground coat application. Pickle in a suspended vertical position to prevent distortion.

7.1.2 Measure and record metal thickness of the test specimens.

7.1.3 Apply a medium high-temperature ground coat (1520 to 1540°F (825 to 840°C)) by spraying. With scribed sides up, first spray the back sides of strips to give 1.5 g dry weight per strip. Dry and cool. Reverse the strips and spray a like amount on the face side. Dry and cool. Use a relatively dry spray to avoid formation of beads on the edges of the strips (Note 2).

7.1.4 Fire the strips in a vertical hanging position at the optimum time and temperature for the ground coat. The preferred procedure is to fire multiples of six simultaneously. Cool in a suspended position, avoiding drafts or forced cooling.

7.1.5 Weigh and record the weight of each strip to the nearest 0.01 g.

7.1.6 Place each strip on the test apparatus with the scribed side down.

7.1.7 Measure and record the ground coat residual load, which is the weight in grams required to cause the strip to just rest on the three knife edges of the fixture. Discard any strips having zero or negative stress (reverse bow) or having positive load of more than 50 g.

NOTE 2—More consistently uniform and usable specimens will result if a spraying technique is developed whereby a few hundredths of a gram less enamel is applied to the back than to the face. This practice reduces the possibility of developing a concavity in the strip as viewed from the face side.

7.2 Application of Test Enamel:
7.2.1 Apply test enamel to the face side of six ground-coated specimens by spraying. Apply the enamel to yield a dry weight of 30 g/ft² (323 g/m², or 2½ g per strip). Use a relatively dry spray to avoid formation of beads along edges. After drying and cooling, use a template to brush back 111/16 in. (43 mm) from each end of strip, leaving 8 5/8 in. (179 mm) of cover coat on the strip. Wipe the edges and back of strips with a felt pad and dust with a soft-bristled brush or wool duster.

7.2.2 Fire panels, again in a vertical hanging position, at optimum temperature and time for the test enamel as cover coat. Cool in a vertical position, avoiding forced cooling. Rapid cooling produces greater residual compressive stresses.

7.2.3 Weigh each strip to the nearest 0.01 g and record.

7.3 Measurement of Load:

7.3.1 Center each coated strip individually over the three knife edges of the fixture with face side upward and punched hole to left. Stops or blocks may be of aid in centering the strips on knife edges.

7.3.2 Centrally load the strip with slotted weights over the center knife edge until the strip just touches the central knife edge. A strong diffuse light source behind the test fixture facilitates observation of the end point.

7.3.3 Measure and record the uncorrected compressive load of each of the cover-coated test strips. The load, in grams, required to cause the strip to just touch the central knife edge is the uncorrected compressive load.

7.3.4 Corrected load is uncorrected compressive load minus residual ground coat load (see 7.1.7).

8. Calculation

8.1 Calculate the corrected load, \( L \), by subtracting the residual ground coat load from the uncorrected compressive load.

8.2 Calculate the compressive stress, \( S_c \), for each test specimen as follows:

\[
S_c = L \times \frac{1.80}{W} \times \frac{0.036}{T_i} \tag{1}
\]

or

\[
S_c = L \times \frac{1.80}{W} \times \frac{0.914}{T_m} \tag{2}
\]

where:

\( S_c \) = compressive stress of the specimen, gf/in. (or gf/mm),

\( L \) = corrected load for cover-coated strip, gf,

1.80 = calculated fired weight of enamel on 8 5/8-in. (219-mm) length of strip equivalent to 30 g/ft² (323 g/m²) (dry),

\( W \) = actual fired weight of test enamel on strip to the nearest 0.01 g,

\( T_i \) = actual metal thickness, in., and

\( T_m \) = actual metal thickness, mm.

8.3 Determine the average of the six compressive stress values and report as the compressive stress of the tested enamel.

8.4 If the end use of the test data requires a statistical comparison of samples, the standard deviation and statistical error of the six specimens comprising one lot may be calculated by the root-mean-square method. The compressive stress of the enamel may then be reported as the numerical average of the test values together with their statistical error (see Appendix X1 for an example of data treatment).

9. Conversion of Test Results

9.1 Conversion charts may be used to make approximate conversions of results of the loaded-beam test method in terms
of other test methods (or vice versa if necessary). Fig. 1 provides for conversion of loaded-beam test results to warp values. Fig. 2 may be used to convert loaded-beam results to ring-stress values.

10. Precision and Accuracy

10.1 The precision and accuracy of this test method are being developed.
FIG. 4 Loaded-Beam Test Apparatus

FIG. 5 Brushing Template

MATERIAL: 304 S.S. OR STENCIL PAPER

Note 1—See Fig. 3 for metric equivalents.

FIG. 5 Brushing Template
APPENDIX

(Nonmandatory Information)

XI. EXAMPLE OF DATA TREATMENT

X1.1 Table X1.1 provides sample values for six specimens to illustrate the application of formulas necessary for the measurement of compressive stress.

X1.2 The following compressive stress values for individual specimens are calculated by applying the formula: 

\[ S_c = L \times \left( \frac{1.80}{W} \right) \times \left( \frac{0.036}{T_i} \right) \]  

(see Section 8):

<table>
<thead>
<tr>
<th>Specimen No.</th>
<th>Compressive Stress (S_c)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>525</td>
</tr>
<tr>
<td>2</td>
<td>524</td>
</tr>
<tr>
<td>3</td>
<td>496</td>
</tr>
<tr>
<td>4</td>
<td>510</td>
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<tr>
<td>5</td>
<td>488</td>
</tr>
<tr>
<td>6</td>
<td>481</td>
</tr>
</tbody>
</table>

Mean 504

X1.3 Calculate the statistical error of the determination as follows:

\[ e = 1.05 s \]  

(X1.1)

where:

\( e \) = statistical error of the mean value for the samples at the 95% confidence level, and

\( s \) = standard deviation of the six individual values from the average of all six.

NOTE X1.1—The factor 1.05 applies only when the number of specimens is six and the confidence level is 95%. For larger or smaller numbers of specimens, use the “a” values for that number shown in Table 2, Part 2, of STP 15 D.\(^3\)

X1.4 Calculate the standard deviation, \( s \), as follows:

\[ s = \sqrt{\frac{\sum S_c^2 - \left( \frac{\sum S_c}{n} \right)^2}{n - 1}} \]  

(X1.2)

where:

\( n \) = number of specimens,

\( \sum S_c^2 \) = sum of the squares of the stress values of the six individual specimens, and

\( \left( \sum S_c \right)^2 \) = square of the sum of the six stress values.

X1.5 The following calculations are involved in applying Eq X1.2:

\[ \sum S_c = \frac{3 024}{6} \quad \frac{1 525 822}{6} = \sum S_c^2 \]  

Mean \( S_c = \frac{504}{6} \)

\[ s = \sqrt{\frac{1 525 822 - \frac{9 144 576}{6}}{5}} \]  

(X1.3)

\[ s = 18.6 \]

X1.6 Applying Eq X1.1, \( e = 1.05 \times 18.6 = 19.5 \).

X1.7 Compressive stress value for example Enamel A thus becomes 504 ± 19.5.

---


---

**FIG. 6** Detail of Test Specimen

**NOTE 1**—Wipe all bisque cover coat enamel from sides, ends, and back of ground coated test sample before firing cover coat.

**NOTE 2**—See Fig. 3 for metric equivalents.
<table>
<thead>
<tr>
<th>Enamel</th>
<th>Specimen No.</th>
<th>Application (g), ( W )</th>
<th>Residual Load</th>
<th>Final Load</th>
<th>Corrected Load, ( L )</th>
<th>Metal Thickness, ( T_i )</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1</td>
<td>1.76</td>
<td>1</td>
<td>486</td>
<td>485</td>
<td>0.034</td>
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<tr>
<td></td>
<td>2</td>
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</tr>
<tr>
<td></td>
<td>3</td>
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<td>7</td>
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<td>468</td>
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<td>1.75</td>
<td>5</td>
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</table>
Standard Test Method for Lead and Cadmium Release from Porcelain Enamel Surfaces

1

This standard is issued under the fixed designation C 872; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the precise determination of lead and cadmium extracted by acetic acid from porcelain enamel surfaces.

1.2 Values stated in SI units are to be regarded as the standard. Inch-pound units are given for information only.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

C 738 Test Method for Lead and Cadmium Extracted from Glazed Ceramic Surfaces

D 1193 Specification for Reagent Water

3. Summary of Test Method

3.1 The lead and cadmium extracted from the article under test by acetic acid at 20 to 24°C (68 to 75°F) after 24 h of leaching are measured by atomic absorption spectrophotometry using a specific hollow cathode lamp for lead and cadmium respectively.

4. Significance and Use

4.1 The determination of lead and cadmium release from porcelain enamel surfaces was formerly of interest only to manufacturers of porcelain enamel cookware and similar food service products. Food contact surfaces of these container-type products have been evaluated using a test procedure similar to Test Method C 738. Recently, however, there has been a need to measure lead and cadmium release from flat or curved porcelain enamel surfaces that are not capable of being evaluated by a test similar to Test Method C 738.

5. Interferences

5.1 Since a specific hollow cathode lamp for lead and cadmium is used, there are no interferences.

6. Apparatus

6.1 Atomic Absorption Spectrophotometer, equipped with a 102-mm (4-in.) single slot or Boling burner head and digital concentration readout attachment (DCR) if available. This instrument should have a sensitivity of about 0.5 mg/L of lead for 1% absorption and a sensitivity of about 0.03 mg/L of cadmium for 1% absorption. The operating conditions as specified in the instrument manufacturer’s analytical methods manual shall be used.

6.2 Hollow Cathode Lead Lamp, with wavelength set at 283.3 or 217.0 nm.

6.3 Hollow Cathode Cadmium Lamp, with wavelength set at 228.8 nm.

6.4 Glassware of chemically resistant borosilicate glass, to make reagents and solutions.

6.5 Test Cell, suitable for the containment of the leaching solution on a flat porcelain enamel surface. A cell that has proved suitable for this purpose is shown in Fig. 1.

7. Reagents

7.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean distilled water. (See Specification D 1193.)
7.3 Acetic Acid (4 % by volume)—Mix 1 volume of glacial acetic acid with 24 volumes of water.

Note 2—A reagent blank shall be run each time a 4 % acid solution is prepared.

7.4 Detergent Rinse—Add 15 g of suitable alkaline detergent to 1 gal (3.79 L) of lukewarm tap water.

7.5 Lead Nitrate Solution (1000 mg Pb/L)—Dissolve 1.598 g of lead nitrate (Pb(NO₃)₂) in 4 % acetic acid and dilute to 1 L with 4 % acetic acid. Commercially available standard lead solutions may also be used.

7.6 Hydrochloric Acid (1 % by weight)—Mix 1 volume of concentrated hydrochloric acid (HCl sp gr 1.19) with 37 volumes of water.

7.7 Cadmium (1000 mg Cd/L)—Dissolve 0.500 g of cadmium metal in 250 mL of hot 1 % HCl (see 7.6), cool, and dilute to 500 mL with 1 % HCl. Commercially available standard cadmium solutions may also be used.

8. Samples

8.1 Test Specimens—Specimens may be cut from production parts or may be prepared on metal blanks under production conditions. Tests may be made on finished parts where flat horizontal surfaces are available.

8.2 Size—The size of the test area shall be approximately 26 cm² (4 in.²).

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Note: A suitable detergent is Calgonite, manufactured by the Calgon Corp., Box 1436, Pittsburgh, PA 15230, and is available in most supermarkets.
9. Procedure

9.1 Preparation of Sample—Take, at random, three identical units and cleanse each with the detergent rinse. Then rinse with tap water followed by distilled water. Dry the specimens and fit into a suitable test cell similar to that shown in Fig. 1, or place a weighted cell onto a flat surface of a production part. Fill each unit with 4 % acetic acid, with a maximum of 40 mL for each 6.45 cm² (1 in.²) of exposed surface. Record the volume of acid for each unit in the sample. Cover each unit with clear, colorless glass plate to prevent evaporation of the solution, avoiding contact between the cover and surface of the leaching solution, and expose to normal laboratory light for 8 to 10 h during the leaching period. Let the solution stand for 24 h at room temperature (20 to 24°C (68 to 75°F)).

9.2 Preparation of Standards:

9.2.1 Lead Standards—Dilute lead nitrate solution (see 7.5) with acetic acid (see 7.3) to obtain working standards having final concentrations of 0, 5, 10, 15, and 20 mg Pb/L.

9.2.2 Cadmium Standards—Dilute cadmium stock solution (see 7.7) with acetic acid (see 7.3) to obtain working standards having final concentrations of 0.0, 0.3, 0.5, 1, 1.5, and 2.0 µg Cd/mL.

9.3 Determination of Lead by Atomic Absorption—Stir the sample (leaching) solution and pour off a portion into a clean flask. Using the atomic absorption spectrophotometer (6.1) and hollow cathode lamp (6.2), concomitantly determine the absorbance of the lead working standards (9.2.1) and sample (leaching) solutions, diluting the latter with 4 % acetic acid if required (if solution contains over 20 mg/L). Concentrate samples containing less than 1 ppm lead by accurately transferring a minimum of 50.0 mL of solution to a 250-mL beaker and evaporating to dryness on a steam bath. Dissolve the residue in 4 % acetic acid by adding exactly 0.1 volume of solution taken for concentration, cover with a watch glass, and swirl to complete dissolution. Prepare a standard curve of absorbance versus concentration (mg/L). Determine the lead content (mg Pb/L) of sample (leaching) solution from the standard curve.

NOTE 3—If a digital concentration readout is used, the standard curve is not necessary. However, standards bracketing the solution under test should be used.

9.4 Determination of Cadmium by Atomic Absorption Spectrophotometry—Proceed as in 9.3 using the cadmium hollow-cathode lamp (see 6.3) and cadmium standards (see 9.2.2). If the sample (leaching) solutions contain more than 2 mg Cd/L, dilute with 4 % acetic acid. Concentrate samples containing less than 0.1 mg/L as in 9.3.

10. Report

10.1 Report the type of units tested, the volume of acid used, and the lead and cadmium, respectively, leached in micrograms per millilitre for each unit tested.

NOTE 4—As indicated in Section 1, this procedure covers the extraction and measurement of lead and cadmium. It is general in that it does not recommend specific sample unit types. For special end uses, as for example, process control or interlaboratory testing, a specific size and type of sample unit should be used.

11. Precision and Bias

11.1 Precision of the analytical method for a single or multiple operator within a single laboratory is within the sensitivity of the atomic absorption spectrophotometer.

11.2 The precision and bias between laboratories is dependent upon the ability to obtain representative samples of the statistical universe being sampled.
Standard Practices for Preparation of Aluminum and Aluminum-Alloy Surfaces for Painting

This standard is issued under the fixed designation D 1730; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These practices cover four types of treatment for preparation of aluminum and aluminum-alloy surfaces for painting, as follows:

1.1.1 Type A—Solvent Cleaning.
1.1.2 Type B—Chemical Treatments.
1.1.3 Type C—Anodic Treatments.
1.1.4 Type D—Mechanical Treatments. These four types cover a number of procedures, as described herein.

1.2 Variations in surface treatment produce end conditions which differ, and which do not necessarily yield identical results when paints are applied. Service conditions will dictate the type of surface preparation that should be selected, although the quality produced by any individual method may vary with different alloys.

1.3 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:
D 609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings and Related Coating Products

2.2 Military Standards:
MIL-C-5541 Chemical Conversion Coatings on Aluminum and Aluminum Alloys

3. Type A—Solvent Cleaning

3.1 Solvent cleaning does not disturb the natural oxide film on the metal and may prove adequate for some applications, such as ambient indoor or very mild service conditions. Three methods may be employed, as follows:

3.1.1 Method 1, Manual Swabbing or Dip-Washing, with a solvent such as mineral spirits or high-flask solvent naphtha. With this method it is extremely difficult to prevent accumulation of contaminants on the swab or in the solvent. This method is only recommended when other treatments are impractical.

3.1.2 Method 2, Solvent Spray Cleaning, in accordance with Method A, Procedure 1 of Practice D 609.

3.1.3 Method 3, Vapor Degreasing, in special equipment employing trichloroethylene vapor, in accordance with Method A, Procedure 2 of Practice D 609.

4. Type B—Chemical Treatments

4.1 Method 1, Alkaline Cleaners—Alkaline solutions, such as caustic soda, etch the metal, thus destroying the natural oxide film. They are followed by an acid treatment, preferably nitric acid or phosphoric acid. They shall not be used on assembled structures. Inhibited alkaline cleaners are sometimes employed as a pretreatment to remove grease and oil prior to an acid treatment. Inhibited alkaline cleaners do not etch the surface. They are not generally recommended unless followed by a conversion treatment, such as described in Methods 4, 5, 6, or 7.

4.2 Method 2, Sulfuric Acid, Chromium Trioxide Etch—This treatment provides a clean and uniform surface without undue etching, and is effective for removing oil and water stains and any film formed during heat-treatment. The etching solution is prepared by adding 1 gal (3.78 L) of concentrated sulfuric acid and 45 oz (1.28 kg) of chromium trioxide to 9 gal (34 L) of water. It is used at a temperature of 160 to 180°F (71 to 82°C) (depending on the alloy and the amount of film) for about 5 min and is followed by a water rinse. This treatment produces a passive surface suitable for painting under mild to intermediate exposure conditions and where clear finishes are to be applied.
4.3 **Method 3, Alcoholic Phosphoric Acid Cleaner**—This treatment involves the use of an aqueous solution of phosphoric acid (10 to 15 volume %) with alcohol or other organic solvents, together with wetting agents, emulsifying agents, etc. The solution may be applied by swabbing or dipping at room temperature (70 to 90°F (21 to 32°C)), and should be allowed to remain on the surface for several minutes, followed by thorough rinsing with clean water. A very thin phosphate film is formed which tends to protect the metal and promote paint adhesion under mild to intermediate exposure conditions.

**Note 2**—U.S. Military Specification MIL-M-10578B describes a treatment of this type.

4.4 **Method 4, Crystalline Phosphate Treatment**—This surface-coating method consists in reacting the aluminum surface in a zinc-acid-phosphate solution containing oxidizing agents and other salts for accelerating the coating action. The aluminum surface is converted to a finely crystalline, phosphate coating of the proper texture adapted to inhibit corrosion and increase the adherence and durability of any applied paint film. It is recommended for product finishes. The phosphate coating process may be carried out by immersion or spray application. The aluminum surface is converted to the phosphate coating by immersion in the processing solution for 30 s to 4 min at 125 to 140°F (51 to 60°C), or by spraying the solution for 10 s to 2 min at 125 to 160°F (51 to 71°C).

**Note 3**—Before applying the treatments according to Methods 4, 5, 6, and 7 of Type B, the aluminum surfaces should be freed of grease, oil, or other foreign material by means of the procedure described in Method 3 of Type A, Method 1 of Type B, or any other suitable method.

**Note 4**—This treatment complies with the requirements of U.S. Federal Specification TT-C-490.

4.5 **Method 5, Amorphous Phosphate Treatment**—This process comprises treatment of clean aluminum surfaces in a warm (95 to 130°F (35 to 54°C)) aqueous solution comprising phosphoric, and hydrofluoric acids, chromium trioxide or a solution thereof. The aluminum surface is converted to a thin, adherent, amorphous coating, iridescent green to gray-green in color, depending upon the aluminum alloy treated, which inhibits corrosion and increases the adherence and durability of applied paint films. This method is recommended for use under the more severe conditions of service, and for product finishes. The coating process may be carried out by immersion or spray application. The time of treatment will vary from 15 to 45 s for the spray process, and from 30 s to 3 min for the immersion application (see Note 3 and Note 5).

**Note 5**—Most of the treatments conforming to Methods 5, 6, and 7 of Type B comply with the requirements of U.S. Military Specification MIL-C-5541.

4.6 **Method 6, Carbonate Chromate Treatment**—This process comprises treatment in a hot (180 to 190°F (82 to 88°C)) dilute solution of sodium carbonate and potassium chromate for periods from 2 to 20 min, followed by a water rinse. The surface is then given a final treatment in hot 5% potassium dichromate solution, followed by a final rinse. The treatment produces a thin, adherent, conversion coating on the surface, increasing the corrosion resistance of the metal and promoting paint adhesion. This method is recommended for use under the more severe conditions of service and for product finishes (see Note 3 and Note 5).

4.7 **Method 7, Amorphous Chromate Treatment**—This process comprises treatment of clean aluminum surfaces in aqueous chromium trioxide solutions containing suitable accelerating agents such as fluoride-containing materials. The aluminum surface is converted to an adherent, amorphous, mixed metallic oxide coating, iridescent golden to light-brown in color, which increases the corrosion-resistance and the adherence and durability of any applied paint film. This method is recommended for use under the more severe conditions of service and for product finishes. The coating process may be carried out by immersion, spray, or brush application, at room temperature (70 to 90°F (21 to 32°C)), in from 15 s to 5 min contact time (see Note 3 and Note 5).

4.8 **Method 8, Acid-Bound Resinous Treatment (see Note 6)**—This surface treatment involves the use of a suitably applied acid-bound resinous film of approximately 0.3 to 0.5 mils (7.6 to 12.7 µm) thickness. The treatment is based on three primary components: a hydroxyl-containing resin; a pigment capable of reacting with the resin and an acid; and an acid capable of insolubilizing the resin by reacting with the resin, the pigment, and the metal substrate. The aluminum surface should be prepared by Methods 1, 2, or 3 of Type A (see Section 3) or chemical treatments, Type B, 4.1 or 4.2 prior to the application of this treatment. The film may be applied by brush, spray, or dip. Under normal conditions it should dry sufficiently for recoating within 30 min; and within 8 h it should not be softened by organic solvents commonly used in paint coatings. The film has good adhesion to the metal substrate and promotes good adhesion of most subsequent organic coatings to itself. This method is recommended for severe service conditions, particularly on fabricated structures, either in the shop or in the field.

**Note 6**—Materials meeting the requirements of U.S. Military Specification MIL-P-15328b may be used to apply Method 8 of Type B.

5. **Type C—Anodic Treatments (see Note 7)**

5.1 Anodic treatment in either sulfuric acid or chromium trioxide electrolyte will provide a protective and inert oxide coating which increases the corrosion-resistance of the metal and promotes paint adhesion. It is recommended where maximum corrosion-resistance by the treatment itself is desired. Anodic treatments should not be used on assemblies of dissimilar metals. The two procedures are as follows:

**Note 7**—Treatments conforming to Methods 1 and 2 of Type C comply with the requirements of U.S. Military Specification MIL-A-8625.

5.2 **Method 1, Sulfuric Acid Anodic**—This treatment consists in making the part the anode in a 15% sulfuric acid electrolyte (by weight) and applying current at a potential of about 16 to 18 V and a current density of 12 A/ft² until desired thickness of coating is obtained. After rinsing in cold water, the coating is sealed for 10 min by treatment in a boiling solution of 5% potassium dichromate. This method is not recommended where danger of acid entrapment is encountered.

5.3 **Method 2, Chromium Trioxide Anodic**—This treatment consists in making the part the anode in a 10 weight %
chromium trioxide electrolyte at a temperature of 100°F (38°C) and applying a potential of 40 V for a period of 30 to 60 min. After first rinsing in cold water, the coating is finally rinsed in water at 150°F (66°C) minimum, and dried; an optional final sealing treatment to obtain maximum protection is obtained by immersion in a solution containing 0.01 g chromium trioxide per litre of water (0.0013 or 1 gal) at a temperature of 208 to 212°F (98 to 100°C) for 5 min.

6. Type D—Mechanical Treatments

6.1 Method 1, Hand or Power Wire-Brushing, or other abrasive treatment. Wire-brushing, either by hand or power, roughens the surface of the metal and mechanically improves the anchorage for superimposed paint films. The disadvantages are that the natural oxide film on the metal is disrupted and oil or grease films and other foreign matter are not completely removed.

6.2 Method 2, Sandblasting, where employed on aluminum or its alloys, must be carried out at relatively low pressures and with a fine silica sand. It roughens the surface and mechanically improves the anchorage for paint films, but destroys the natural oxide film on the metal. Where used, it should be followed by an inhibitive chemical treatment.

7. Keywords

7.1 aluminum; pretreatment for painting; surface treatment
Standard Practices for Preparation of Hot-Dip Aluminum Surfaces for Painting

This standard is issued under the fixed designation D 1731; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These practices describe procedures for the preparation of hot-dip aluminum surfaces for painting.

1.2 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

D 609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings and Related Coating Products

2.2 Military Standards:

MIL-C-5541 Chemical Conversion Coatings on Aluminum and Aluminum Alloys

3. Types of Surface Preparations

3.1 Two types of surface preparations are covered as follows:

3.1.1 Solvent Cleaning, and

3.1.2 Chemical Treatments.

3.2 These two types cover several methods as described in Section 5. Variations in surface preparation produce end conditions which differ and which do not necessarily yield identical results when paints are applied. Service conditions will dictate the type of surface preparation that should be selected.

4. Solvent Cleaning

4.1 The surfaces may be prepared for painting by any of the methods for solvent cleaning described in 4.2 and 4.3.

4.2 Method 1—Manual swabbing or dip washing with a solvent such as mineral spirits or high flask solvent naphtha. It is extremely difficult to produce a clean metal surface by this method. Accumulated contaminants in the solvent or on the swab are frequently redeposited on the surface being prepared.

4.3 Method 2—Solvent spray cleaning in accordance with Method A, Procedure 1 of Practice D 609.

4.4 Method 3—Vapor degreasing in special equipment in accordance with the latest revision of Method A, Procedure 2 of Practice D 609, employing trichlorethylene or perchlorethylene vapor. Solvent cleaning does not disturb the natural oxide film on the metal and may prove adequate for some applications.

5. Chemical Treatment

5.1 The surfaces may be prepared for painting by any of the methods for chemical treatment (Note 1) described in 5.2 to 5.9:

NOTE 1—Materials and procedures employed in these methods of treatment are available from a number of sources as proprietary compounds or methods. Selection may be made from available sources. The hexavalent chromium methods given are not recommended as hexavalent chromium is a known carcinogen.

5.2 Method 1, Alkaline Cleaners—Etching alkaline solutions, such as caustic soda, should be avoided because of the possibility of dissolving away the aluminum coating thereby exposing the base metal. Since inhibited alkaline cleaners do not etch the surface, they can be used to remove grease, oil, and other organic solids from hot dip aluminum. After alkaline cleaning, the surface should be thoroughly rinsed with clean water. Most often alkaline-cleaned surfaces are subsequently processed by one of the following methods, described in 5.3-5.9.

5.3 Method 2, Alcoholic Phosphoric Acid Cleaners—This treatment involves the use of an aqueous solution of phosphoric acid with alcohol or other organic solvents, together with wetting agents, emulsifying agents, etc. The solution may be applied by swabbing or dipping (preferably at a temperature of 70 to 90°F (21 to 32°C)). The solution should remain on the surface for several minutes, followed by thorough rinsing with clean water. A very thin phosphate film is formed which tends to protect the metal and promote paint adhesion.

NOTE 2—Materials meeting the requirements of the latest revision of U.S. Military Specification MIL-M-10578B may be used.
5.4 Method 3, Acid Fluoride Deoxidizer—This treatment includes the use of nitric or sulfuric acid with chromium trioxide and a fluoride. It provides a clean and uniform surface without undue roughening and is effective for removing inorganic soils and any film formed during the production of the hot dip aluminum. The deoxidizing solution is generally used at a temperature of 70 to 130°F (21 to 54°C) (depending on the alloy and the amount of film) and is followed by a water rinse.

Note 3—Before applying the treatments according to Chemical Treatment Methods 3, 4, 5, 6 and 7, the aluminum surfaces should be freed of grease, oil or other foreign material by means of the procedure described in Solvent Cleaning Method 3, Chemical Treatment Method 1 or any other suitable method.

5.5 Method 4, Crystalline Phosphate Treatment—This surface-coating method consists in reacting the aluminum surface in a 130 to 145°F (54 to 63°C) (approximately) zinc acid-phosphate solution containing oxidizing agents and other salts for accelerating the coating action. The aluminum surface is converted to a crystalline, phosphate coating of the proper texture adapted to inhibit corrosion and increase the adherence and durability of any applied paint film. The phosphate coating process may be carried out by immersion or spray application. The aluminum surface is converted to the phosphate coating by immersion in the processing solution for 30 s to 4 min, or by spraying the solution for 10 s to 2 min (Note 3).

5.6 Method 5, Amorphous Phosphate Treatment (see Note 4)—This process comprises treatment of clean aluminum surfaces in a 90 to 125°F (32 to 51°C) aqueous solution comprising phosphoric, hydrofluoric acids and chromium trioxide. The aluminum surface is converted to a thin, adherent, amorphous coating, iridescent green to gray green in color, depending upon the aluminum alloy treated, which inhibits corrosion and increases the adherence and durability of applied paint films. The coating process may be carried out by immersion or spray application. The time of treatment will vary from 15 to 45 s for the spray process and 30 s to 3 min for the immersion application (Note 3).

5.7 Method 6, Carbonate Chromate Treatment (see Note 4)—This process comprises treatment in a 160 to 180°F (71 to 82°C) dilute solution of sodium carbonate and potassium chromate for periods from 2 to 20 min, followed by a water rinse. The surface is then given a final treatment in a 150 to 160°F (66 to 71°C), 5% potassium dichromate solution followed by a final rinse. The treatment produces a thin, adherent, conversion coating on the surface, increasing the corrosion resistance of the metal and promoting paint adhesion (Note 3).

5.8 Method 7, Amorphous Chromate Treatment (see Note 4)—This process comprises treatment of clean aluminum surfaces in aqueous chromium trioxide solutions containing suitable accelerating agents such as fluoride-containing materials. The aluminum surface is converted to an adherent, amorphous, mixed metallic oxide coating, iridescent golden to light brown in color, which increases the corrosion resistance and the adherence and durability of any applied paint film. The coating process may be carried out by immersion, spray or brush application, at 70 to 130°F (21 to 54°C), in from 15 s to 5 min contact time. It may be necessary to precede this treatment with a deoxidizer as described in Method 3 (see Note 3).

Note 4—Most of the treatments conforming to Chemical Treatment Methods 5, 6, and 7, comply with the requirements of the latest revision of U.S. Military Specification MIL-C-5541.

5.9 Method 8, Acid-Bound Resinous Treatment (see Note 5)—This surface treatment is based on the deposition of an acid-bound resinous film of approximately 0.3 to 0.5 mils (7.6 to 12.7 µm) in thickness on the aluminum surface in such a manner as to enhance the adhesion of subsequently applied paint coatings. The treatment is based on three primary components: a hydroxyl-containing resin; a pigment capable of reacting with the resin and an acid; and an acid capable of insolubilizing the resin by reacting with the resin, the pigment and the metal substrate. The aluminum surface should be pretreated by Solvent Cleaning Methods 1, 2 or 3 or Chemical Treatment Methods 1 or 2 prior to the application of this treatment. The film may be applied by brush, spray, or dip. Under normal conditions it shall dry sufficiently for recoating within 30 min. The film has good adhesion to the metal substrate and promotes good adhesion of most subsequent organic coatings to itself.

Note 5—Materials meeting the requirements of the latest revision of U.S. Military Specification MIL-C-15328 may be used to apply to Chemical Treatment Method 8.

6. Keywords

6.1 hot-dip aluminum; pretreatment for painting; surface treatments
1. Scope

1.1 These practices cover two classes of treatment for preparation of magnesium alloy surfaces for painting, as follows:

Class I—Chemical Treatments.
Class II—Anodic Treatments.

In general, the latter treatments are the more protective of the two classes. Mechanical (abrasive) treatments, solvent cleaning, alkaline solution treatments, and acid pickles not resulting in protective conversion coatings are suitable preliminary treatments only for metal to be exposed under mildly corrosive (indoor) exposures. When a high degree of corrosion protection and paint adhesion are desired, as in many outdoor environments, surface preparation by one of the above conversion-coat classes is necessary. The hexavalent chromium based methods given are not recommended as hexavalent chromium is a known carcinogen.

Note 1—Testing of Coatings—Quality control tests of coatings are frequently desirable, and these generally consist of exposures, with or without paint, to salt spray, humidity, or natural environments, with suitable procedures for assessing the degree of breakdown suffered after fixed time intervals. It is recommended that quality control tests of coatings shall be made as far as possible with high-purity material (for example AZ31A alloy), the inherent corrosion rate of which is relatively consistent from batch to batch and that precautions shall be taken to remove surface contamination before coatings are applied. Such contamination shall be removed by acid pickling to a depth of at least 0.001 in. (25 µm) per side.

1.2 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. (See Note 11.)

PRELIMINARY TREATMENT OF SURFACES

2. Procedure

2.1 Certain anodic treatments simultaneously produce conversion coatings on, and remove contamination from, magnesium alloy surfaces. In general, however, apply conversion coatings only to surfaces previously freed from all contamination, including oxide, rolling-scale, corrosion product, burned-on drawing and forming lubricant, and the contamination introduced by blast cleaning and fabrication operations. Contamination in or under surface conversion coatings seriously reduces their protective values (Note 2). For the removal of tenacious surface contamination, such as rolling-scale or casting skin, an acid pickle to dissolve some of the actual surface is essential. When organic contamination, such as grease or oil, is also present, an initial degreasing operation in solvent or in an alkaline degreasing solution is usually necessary to allow the subsequent acid to wet the surface. These matters are discussed in more detail under the headings of the specific cleaners or treatments (Note 3), as follows:

2.2 Alkaline Cleaners—Oil, grease, and old (but not baked) chrome-pickle coatings are readily removed by most commercially available heavy-duty alkaline cleaners; but such cleaners are not suitable for removing oxide and the like, for which purpose use acid pickles, preceded by alkaline cleaners. Remove graphite lubricant and also baked chrome-pickle coatings by a solution conforming to the following composition:

Caustic soda (NaOH) 12 oz (90 g)
Wetting agent 0.1 oz (0.74 g)
Water (Note 4) 1 gal (1 litre)

Soak the parts in the above cleaner for 10 to 20 min at boiling-point, and a treatment shall follow either in the chromic acid-nitrate pickle described under 2.3.3 or, for parts machined to fine tolerances, in the chromium trioxide solution described under 2.3.1. After alkaline cleaning, rinse in water very thoroughly. Alkaline cleaners may be held in plain steel tanks.
2.3 Acid Cleaners (Note 1)—Acid picking removes mill-scale, oxide, corrosion product, and the like. Use as a preliminary treatment for surface conversion coatings when the highest degrees of surface uniformity and protective values are required. Acid cleaners are as follows:

2.3.1 For Sand and Permanent Mold Castings:

2.3.1.1 Nitric-Sulfuric Acid Solution—Use a solution of 8 volume % of concentrated nitric acid (HNO₃) plus 2 volume % of concentrated sulfuric acid (H₂SO₄) in water (see Note 1(a)) at 70 to 90°F (21 to 32°C) as a preliminary treatment for new sand castings and to remove the surface-contaminating effects of concentrated sulfuric acid (H₂SO₄) in water (see Note 1). Immerse for about 10 to 15 s, or until 0.002 in. (51 μm) per surface is removed. The solution may be held in ceramic, rubber, synthetic rubber, or vinyl-lined tanks.

2.3.1.2 Chromic Acid Solution—Use a boiling 20 weight % solution of chromic anhydride (CrO₃) in water to remove old scale, oxide, corrosion product, and the like. Use as a preliminary treatment for surface conversion coatings when the above solutions operate at 70 to 90°F (21 to 32°C). The solution shall conform to the following composition:

- Chromium trioxide (CrO₃) 37.5 oz (280 g)
- Hydrofluoric acid (80% HF) 1 fl oz (8 ml)
- Nitric acid (70% HNO₃) 3.25 fl oz (25 ml)
- Water (Note 4) to 1 gal (1 litre)

Immerse the parts in the above solution at 70 to 90°F (21 to 32°C) for 30 s to 2 min, or until a bright, clean surface is obtained. The solution may be held in tanks lined with synthetic rubber or vinyl-base materials.

2.3.2 For Wrought Products:

2.3.2.1 Acetic Acid-Nitrate Solution—This solution rapidly removes surface contamination to 0.001 in. (25.4 μm). Use for wrought parts subsequently to be finished for the maximum protective value. The solution shall conform to the following composition:

- Glacial acetic acid 25.5 fl oz (199 ml)
- Sodium (NaNO₃) 6.6 oz (49.5 g)
- Water (Note 4) to 1 gal (1 litre)

Immerse the parts in the above solution at 70 to 90°F (21 to 32°C) for 30 s upwards, or until a bright, clean surface is obtained. When heavy surface contamination, such as hot-rolled mill-scale is to be removed, immersion times shall be sufficient to remove at least 0.001 in. (25 μm) per surface. The solution may be held in No. 990A aluminum, ceramic, or rubber-lined tanks.

2.3.2.2 Chromium Trioxide-Nitrate Solution—Use this solution following the use of the method described in 2.2 for the removal of burned-on graphite lubricants from hot-formed parts. The solution shall conform to the following composition:

- Chromium trioxide (CrO₃) 1.5 lb (180 g)
- Sodium nitrate (NaNO₃) 2 oz (15 g)
- Water (Note 4) to 1 gal (1 litre)

Immerse the parts in the above solution at 70 to 90°F (21 to 32°C) for approximately 5 min, for the removal of graphite.

2.4 Abnormally slow reaction in the above solution indicates that it is depleted and that the pH has risen to 1.7 or higher. Revivify the solution by the addition of chromium trioxide to bring the pH back to 0.5 to 0.7. Attempt no more than four revivifications. The solution may be held in ceramic, No. 990A aluminum, 18-8 stainless steel, or synthetic rubber-lined tanks.

Note: 2—Removal of Contamination by Welding Fluxes—When a part to be painted has been welded by an operation involving the use of flux, such flux shall be removed before the subjection of the part to any surface preparation process. Such removal shall be made immediately by immersing the part in hot water with scrubbing, and finally by immersing it for 1 h in a boiling 5% solution of sodium dichromate, after which the part shall be well rinsed.

Note: 3—Suspension of Articles for Treatment—The use of magnesium alloy suspension wires is preferred for use in acid pickles in order to avoid objectionable contamination of the solutions through dissolution of the wire materials. Heavy metal contamination, particularly of copper, may deposit on the magnesium surface and lead to seriously reduced corrosion resistance. Copper suspension wires in the hot dichromate solutions are not objectionable.

Note: 4—Quality of Water—In the preparation and makeup of acid pickles, dichromate solutions, and hot-water rinses, precautions shall be taken against the use of water contaminated with heavy-metal impurities, or excessive chlorides or sulfates. No upper limits can be specified at this stage for soluble impurities in the water, but powdering of coatings and poor resistance to corrosion are known to result from the use of contaminated water. Thus, when a choice exists, water from steam condensate or ion-exchange-treated water shall be employed in preference to well water or hard tap water.

SURFACE PREPARATION PROCEDURES—CHEMICAL

3. Class I, Type I (Chrome Pickle)³

3.1 Scope—Class I, Type I treatment is applicable to all forms and alloys of magnesium except certain special alloys containing silver, but since it may remove as much as 0.0006 in. (15 μm) of metal per surface, it shall not be used on parts machined to fine tolerances. When properly applied, the process constitutes a good paint base, but rigid control is required at each step. The treatment is applicable to magnesium alloy containing inserts of, or attached to, other metals.

3.2 Procedure—For wrought parts the bath shall conform to the following composition:

<table>
<thead>
<tr>
<th>Composition</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium dichromate (Na₂Cr₂O₇·2H₂O)</td>
<td>1.5 lb (180 g)</td>
</tr>
<tr>
<td>Nitric acid (HNO₃) (sp gr 1.42)</td>
<td>1.5 pt (187 ml)</td>
</tr>
<tr>
<td>Water (Note 4)</td>
<td>to 1 gal (1 litre)</td>
</tr>
</tbody>
</table>

3.2.1 For die-, sand- and permanent-mold castings the solution shall conform to the following composition:

<table>
<thead>
<tr>
<th>Composition</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium dichromate (Na₂Cr₂O₇·2H₂O)</td>
<td>1.5 lb (180 g)</td>
</tr>
<tr>
<td>Nitric acid (HNO₃) (sp gr 1.42)</td>
<td>1.5 pt (187 ml)</td>
</tr>
<tr>
<td>Sodium potassium, or ammonium acid fluoride (NaH₄F₆, KHF₆, or NH₄HF₂)</td>
<td>2 oz (15 g)</td>
</tr>
<tr>
<td>Water (Note 4)</td>
<td>to 1 gal (1.0 litres)</td>
</tr>
</tbody>
</table>

For wrought products, sand, and permanent-mold castings the above solutions operate at 70 to 90°F (21 to 32°C). The

³ Conforming to Class I, Type I treatments are the Dow No. 1 process, the AMC "A" process, and the Type I process of Military Specification MIL-M-3171A.
immersion times shall be from 1 to 2 min, the necessary time increasing with use of the solution. For die-castings give the parts a 15 to 30 s dip in water at 160 to 180°F (71 to 82°C), followed immediately by a 10-s dip in the second of the above baths, operated at 120 to 140°F (49 to 60°C). Failure to preheat the castings results in no coating in 10 s.

3.2.2 Following immersion remove the parts, allow to drain for not less than 5 s nor more than 30 s, then wash thoroughly in cold running water, followed by a dip in hot water at 160 to 180°F (71 to 82°C) to facilitate drying. Do not allow the parts to drain following the chromate treatment for more than the specified 30 s; excessive drainage times result in powdery coatings of poor value as paint bases. Such coatings also result from the use of hot-water rinses, the temperatures of which are in excess of 180°F (82°C). Paint the parts preferably immediately after they are dry. The solution may be held in Type 316 stainless steel, or ceramic tanks or in steel tanks lined with synthetic rubber or vinyl-base materials. Tanks of ASTM alloy No. 990 A, or its Aluminum Association equivalent, Alloy No. 1100 aluminum are satisfactory for the nonfluoride-containing pickle.

3.3 Revivification of Solutions—Sluggish reaction with the metal, associated with pale yellow, lustrous coatings, indicates that the solution is depleted. Revivification is accomplished by the addition of dichromate and nitric acid, to raise the dichromate again to 1.5 lb (180 g)/gal (1 litre) and the free nitric acid to levels indicated in the table below. Revivify when the free nitric acid content of the solution is depleted to 0.5 pt/gal (62.3 ml/litre) (see Section 11 for analytical procedure) and shall take place once only for M1 and ZK60A alloys, and not more than six times for other alloys if good paint-base properties are desired. Excessive use of the solution or too many revivifications result in smooth, lustrous coatings not possessing the degree of etch necessary for the best paint adhesion with conventional primers. Revivify in accordance with the following table:

<table>
<thead>
<tr>
<th>Revivification Number</th>
<th>Adjust Concentration of HNO₃ to:</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.3 pt/gal (162 ml/litre)</td>
</tr>
<tr>
<td>2</td>
<td>1.1 pt/gal (137 ml/litre)</td>
</tr>
<tr>
<td>3 to 6</td>
<td>0.9 pt/gal (113 ml/litre)</td>
</tr>
</tbody>
</table>

4. Class I, Type II (Sealed Chrome Pickle)⁴

4.1 Scope—Class I, Type II treatment is applicable to all types and forms of magnesium-base alloys, subject only to the limitations of the Class I, Type I treatment, since it is essentially the same process as the latter, followed by sealing. In protective qualities and as a paint base, this treatment is somewhat superior to that of Class I, Type I treatment under severe exposure conditions (Note 5).

4.2 Procedure—Following chrome pickling as specified under Section 3 and rinsing in cold water, transfer the parts immediately to a boiling solution conforming to the following composition:

\[
\text{Sodium dichromate (Na}_2\text{Cr}_2\text{O}_7\cdot2\text{H}_2\text{O)} \quad 1.5 \text{ lb (180 g)}
\]

Boil the parts in the above solution for 30 min, after which rinse them in cold running water, followed by a rinse in hot water at a temperature of not less than 160°F (71°C) nor more than 180°F (82°C) to facilitate drying. Preferably apply the paint coating immediately after the parts are dry. The solution may be held in a steel tank.

Note 5—Causes of Defective Coatings—The following information is intended to provide guidance on the causes of the most usual defects arising in the application of either Class I, Type I or Class I, Type II coatings:

(a) Spotted Coatings are caused by ineffective preliminary degreasing or by the presence of excessive surface contamination not removed prior to or during the chrome pickling treatment, or both.

(b) Nonadherent Powdery Coatings are caused by:
   (1) Too long an interval between removal from the chrome pickle and rinsing,
   (2) Ratio of acid to sodium dichromate too high,
   (3) Temperature of the solution or of the hot-water rinse too high,
   (4) Metal improperly degreased, or
   (5) Solution revivified too many times.

5. Class I, Type III⁵

5.1 Scope—Class I, Type III treatment is applicable to all types and forms of magnesium-base alloys except M1 alloy and certain rare-earth alloys similar to EK30A. It produces in itself no appreciable dimensional change and is, therefore, applicable to parts machined to fine tolerances. The treatment is applicable to magnesium containing inserts of, or attached to, other metals, but in such cases the preliminary treatment (5.2) shall be in the bifluoride bath (Solution No. 2) described in 5.2.2.

Note 6—Causes of Defective Coatings—The following information is intended to provide guidance on the causes of the more usual defects arising in the application of coatings from the Class I, Type III treatment.

(a) Nonadherent Powdery Coatings are caused by:
   (1) Over-dilution of the hydrofluoric acid or acid fluoride solution,
   (2) Low pH (less than 4.0) of the dichromate solution,
   (3) Insufficient precleaning of the metal surface, and
   (4) Direct contact between the steel tank containing the dichromate solution and the article being treated.

(b) Failure to Coat and Nonuniform Coatings are caused by:
   (1) High pH of the dichromate solution,
   (2) Low concentration of the dichromate,
   (3) Insufficient precleaning of the metal surface,
   (4) Omission of fluoride treatment,
   (5) Use of an unsuitable alloy for the treatment, for instance, M1 alloy.

   (6) Excessive immersion time in the hydrofluoric acid solution or use of an H₄F₂ concentration in the dichromate solution in excess of 0.2%.

   (7) Insufficient rinsing after the hydrofluoric acid dip, and

   (8) Insufficient heating of the dichromate solution (minimum temperature should be 200°F (93°C)).

5.2 Procedure—Following cleaning as prescribed in Section 2, treat the parts first by immersion at 70 to 90°F (21 to 32°C) in one or other of the following solutions:

5.2.1 Solution No. 1:

| Calcium or magnesium fluoride (CaF₂ or MgF₂) | 1/6 oz (2.5 g) |
| Water (Note 4) | to 1 gal (1.0 litre) |

⁴ Conforming to Class I, Type II treatments are the AMC “L” process, the Dow No. 10 process, and Type II treatment of Military Specification MIL-M-3171A.

⁵ Conforming to Class I, Type III treatments are the AMC “G” process, the Dow No. 7 process, and the Type III treatment of Military Specification MIL-M-3171A.
5.2.2 Solution No. 2:

Sodium, potassium, or ammonium acid fluoride (NaH₂F₂, KH₂F₂, or NH₄HF₂)
Water (Note 4) to 1 gal (1 litre)

Solutions No. 1 and No. 2 may be held in tanks consisting of steel lined with lead or rubber. In solution No. 1, immerse AZ31A and AZ31B alloy parts from 30 s to 1 min; immerse all other alloys for 5 min. In solution No. 2, immerse all the alloys for 5 min. Solution No. 2 is suitable for use with all forms of magnesium alloys except those castings which have not been acid-pickled after blasting; these castings shall be treated in the hydrofluoric acid solution (solution No. 1). Following one or other of the above treatments, the parts shall be rinsed thoroughly in cold running water and transferred to a solution conforming to the following composition:

Sodium dichromate (Na₂Cr₂O₇·H₂O) 1 to 1.5 lb (120 to 180 g)
Calcium or magnesium fluoride (CaF₂ or MgF₂)
Water (Note 4) to 1 gal (1 litre)

Operate the above solution at boiling point and immerse the parts therein for 30 min, following which thoroughly rinse them in cold running water, followed by a hot-water dip to facilitate drying. Preferably, apply the paint coating as soon as possible thereafter. A mild steel tank is suitable for holding the above solution.

5.3 Maintenance of Solutions:

5.3.1 Fluoride Solutions—Keep the concentration of free hydrofluoric acid in the solutions of 5.2.1 and 5.2.2 constant by the addition, as required, of either hydrofluoric acid or acid fluoride, respectively. See Section 9 for the analytical procedure.

5.3.2 Dichromate Solution—Keep the pH of the dichromate solution within the limits 4.0 to 5.5, or 4.0 to 4.8 in the case of AZ31A or AZ31B alloy, by addition of chromium trioxide as required. Maintain the level by the addition of water. Maintain the solution saturated with respect to either magnesium or calcium fluoride by continuous immersion of a cloth bag containing excess of one or other of these compounds.

SURFACE PREPARATION PROCEDURES—ANODIC

6. Class II, Type I (Galvanic Dichromate Treatment)

6.1 Scope—This treatment produces black coatings of good protective and paint-base qualities, and is applicable to all alloys and forms of magnesium, including M1 alloy, with or without attachments of other metals. No appreciable dimensional change results, and hence the treatment is also applicable to articles machined to fine tolerances.

6.2 Procedure—Following cleaning as prescribed in Section 2, treat the articles in fluoride solution No. 1 or No. 2 as prescribed in 5.2. Use the latter solution when other metals are attached to the magnesium. After rinsing, immerse the articles in a solution conforming to the following composition:

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonium sulfate ([NH₄]₂SO₄)</td>
<td>4 oz (30 g)</td>
</tr>
<tr>
<td>Sodium dichromate ([Na₂Cr₂O₇·2H₂O)</td>
<td>4 oz (30 g)</td>
</tr>
<tr>
<td>Ammonia (NH₄OH) (sp gr 0.880)</td>
<td>½ fl oz (2.6 ml)</td>
</tr>
<tr>
<td>Water (Note 4)</td>
<td>1 gal (1 litre)</td>
</tr>
</tbody>
</table>

Operate the above solution at 120 to 140°F (49 to 60°C). The articles shall be made the anodes in the solution, with the tank, if of mild steel, acting as cathode, or with separate steel cathodes if the tank is lined with nonmetallic materials. No separate generator is necessary, but the magnesium parts must be electrically connected with the tank, or with the separate cathode plates through an external connection, taking care that the parts do not make direct contact with the cathode material.

6.2.1 The time of treatment shall be such that a uniform black coating is obtained on the articles. This takes from 10 to 30 min, and the consumption of from 70 to 150 A·min/ft² (929 cm²). An anodic current density of not more than 10 A/ft² (929 cm²) is desirable.

6.2.2 Following the treatment, rinse the parts thoroughly in cold running water, followed by a hot-water dip to facilitate drying.

6.3 Maintenance of Solutions:

6.3.1 Fluoride Solutions—Maintain the fluoride solutions as under 5.3.1. See Section 9 for the analytical procedure.

6.3.2 Sulfate-Dichromate Solution—Maintain the pH of the solution between 5.6 and 6.2 by periodic additions of a solution containing 5 weight % of both chromic anhydride (CrO₃) and concentrated sulfuric acid (H₂SO₄, sp gr 1.84).

7. Class II, Type II

7.1 Scope—This treatment is applicable to all forms and alloys of magnesium. Many aluminum alloys will anodically polarize in the treatment solution, and hence magnesium parts with aluminum attachments or inserts can be anodized. With aluminum alloys containing copper, however, the coating of the magnesium will proceed only if the area of the aluminum alloy is small compared to that of the magnesium. Metals other than aluminum or its base alloys shall not be in contact with the magnesium.

7.2 Coating Properties—The anodic coating consists of two phases: the first-formed phase is greenish-tan in color and about 0.0002 in. (5 µm) thick; whereas the second phase, formed only at higher voltages following the production of the tan coating, is fused and vitreous in nature, dark-green in color, and of thickness about 0.0012 in. (30 µm), causing a dimensional increase of about 0.001 in. (25 µm) per surface. The corrosion-resistance and paint-base characteristics are similar, and excellent for both the above phases. The thicker coating possesses high abrasion resistance, but spalls under compression deformation. The dark-green coating can be partially impregnated with low-viscosity, penetrating, organic coating materials, a procedure that considerably reduces its spalling tendency.

Note: The dark-green coating of Class II, Type II treatments is recommended in preference to the thin tan coating only when:
(a) Preliminary removal of surface contamination is not convenient.

6 Conforming to Class II, Type I treatment are the AMC “K” process, the Dow No. 9 process, and Type IV treatment of Military Specification MIL-M-3171A.

7 Conforming to Class II, Type II treatment is the Dow No. 17 process.
(b) The highest degree of abrasion-resistance is required from the coating.
(c) A dimensional increase (see above) can be tolerated.
(d) The article will not be subjected in service to impact, deformation, or flexing likely to cause spalling of the coating.

7.3 Procedure—The thin tan coating is formed with a current consumption and in a time approximately one quarter of that required to form the dark-green coating. The latter forms only after the voltage across the solution has reached the spark potential. During the ensuing coating formation, surface contamination, including graphite, is concurrently removed. Thus, articles intended to receive the dark-green coating need not first be cleaned. On the other hand, the formation of the thinner tan coating does not concurrently remove surface contamination. Since such contamination can seriously reduce corrosion resistance, it is mandatory that a preliminary cleaning, including acid pickling (2.2) shall be given to articles intended to receive the lower-voltage tan coating.

7.3.1 Rack and clean parts as appropriate (see above), and anodize either by direct or alternating current in an electrolyte conforming to one of the following compositions, respectively:

<table>
<thead>
<tr>
<th>Direct Current</th>
<th>Alternating Current</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonium acid fluoride (NH₄HF₂)</td>
<td>32 oz (240 g)</td>
</tr>
<tr>
<td>Sodium dichromate (Na₂Cr₂O₇·2H₂O)</td>
<td>13.3 oz (99 g)</td>
</tr>
<tr>
<td>Phosphoric acid (85% H₃PO₄)</td>
<td>11.5 fl oz (90 ml) to 1 gal (1 litre)</td>
</tr>
<tr>
<td>Water (Note 4)</td>
<td>to 1 gal (1 litre)</td>
</tr>
</tbody>
</table>

The temperature of the solution shall preferably be from 160°F to 180°F (71 to 82°C). It will not operate below 140°F (60°C), but temperatures higher than 180°F (82°C) do not deleteriously affect the results. After anodizing rinse the parts in running cold water, followed by a short immersion in hot water, or by treatment with hot air, to facilitate drying. Preferably, apply the paint coating immediately after the parts are dry.

7.4 Power Requirements—A current consumption of from 50 to 500 A·min/ft² (929 cm²) is necessary, according to both the composition of the alloy being treated and the type of coating desired. Thus, in the case of the dark-green coating on AZ31 alloy, for a 10-min treatment with alternating current, a current density of 30 A/ft² (929 cm²) is applied; but greater or lesser currents may be utilized by varying the time of treatment as, for example, application of 50 A/ft² (929 cm²) for 6 min. On the other hand, for the same alloy, the thin tan coating may be applied with alternating current, with a current consumption of 75 A·min/ft² (929 cm²), equivalent to a treatment time of only 1½ min at 50 A/ft² (929 cm²). As the coating forms, the resistance of the circuit increases, and consequently the voltage must be increased to maintain a constant current density. Normally the voltage across the bath will reach from 75 to 95 for the dark-green coating (according to alloy composition) and from 60 to 75 for the thin tan coating. In carrying out the treatment, a constant current control is a considerable advantage, inasmuch as the total treatment time can be preset and manual control of the voltage and current then become unnecessary.

7.4.1 As guides to the current consumptions with various alloys, some data are presented in Table 1. It will be appreciated that rigid current consumptions in the formation of the thin tan coating cannot be specified, since this coating can be varied in thickness considerably between say 0.0001 in. (2.5 µm) and 0.0005 in. (12.7 µm), and still be formed below the critical spark potential at which the dark-green vitreous coating begins to develop.

7.5 Solution Control—With depletion of the bath upon use, the final voltage required to impress the above currents on the article will slowly rise, but even for the full green coating at least 20 ft² (50 dm²) of surface can be treated per gallon (1.0 litre) of solution before any change in the final voltage is experienced; and up to 40 ft² (98 dm²)/gal (1 litre) can be treated before revivification of the solution becomes necessary.

7.5.1 Prior to revivification, analyze for dichromate and phosphoric acid (see Sections 10 and 12), and make up differences from the original composition by additions of sodium dichromate and phosphoric acid.

7.5.2 A simple method for the determination of fluoride in the solution is not yet available. The fluoride concentration, however, is not critical, provided it always exceeds minimum concentration, below which pitting of the articles may ensue during their treatment. When revivifications are made by additions of sodium dichromate and phosphoric acid, make a concurrent addition of ammonium acid fluoride, equal to three times the weight of sodium dichromate added. Secondly, if pitting of the articles should occur under treatment, it is an indication that the fluoride content of the solution is too low, and about 5 oz (38 g)/gal (1 L) of ammonium acid fluoride shall then be added in order to relieve the trouble.

7.6 Tank and Rack Materials—Mild steel is suitable for tanks and heating coils. Other metals, such as copper, aluminum, lead, zinc, Monel, and stainless steels are attacked by the electrolyte. When direct current is used the tank itself can be the cathode, but, when alternating current is used, at least two magnesium parts are necessary, of area ratios not more than 3 to 1, each one serving as electrode. The articles shall not contact the tank, otherwise pitting will ensue.

7.6.1 Racks shall be normally made of magnesium alloy, but aluminum alloys containing magnesium, such as ASTM alloy No. GR20A, or its Aluminum Association equivalent, alloy No. 5052, can be used if desired, provided they are not allowed to be in the solution on open circuit. Use a spring clip with magnesium ends for providing contact with thin sheet material, the liquid level not being allowed to extend beyond the limit of the magnesium ends of the clip. No solution-line attack is

---

**TABLE 1 Current Consumption with Various Alloys**

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Voltage A·min/ft² (929 cm²)</th>
<th>Direct Current</th>
<th>Alternating Current</th>
</tr>
</thead>
<tbody>
<tr>
<td>AZ31A or B</td>
<td>90 70 300 75 95 75 190 50</td>
<td>Dark-Green Thin Tan Dark-Green Thin Tan Dark-Green Thin Tan</td>
<td></td>
</tr>
<tr>
<td>AZ60A</td>
<td>90 68 400 ... 95 72 300 ...</td>
<td>Dark-Green Thin Tan</td>
<td></td>
</tr>
<tr>
<td>AZ63A</td>
<td>80 68 600 100 84 72 400 70</td>
<td>Dark-Green Thin Tan</td>
<td></td>
</tr>
<tr>
<td>AZ91A</td>
<td>75 64 480 ... 80 69 280 ...</td>
<td>Dark-Green Thin Tan</td>
<td></td>
</tr>
<tr>
<td>AZ92A</td>
<td>75 64 600 ... 80 69 400 ...</td>
<td>Dark-Green Thin Tan</td>
<td></td>
</tr>
<tr>
<td>EK30A</td>
<td>90 70 400 ... 95 75 300 ...</td>
<td>Dark-Green Thin Tan</td>
<td></td>
</tr>
<tr>
<td>ZK60A</td>
<td>85 70 300 ... 90 75 190 ...</td>
<td>Dark-Green Thin Tan</td>
<td></td>
</tr>
</tbody>
</table>
experienced on magnesium. Magnesium racks may be stripped by immersion for 2 min in hot 20% chromium trioxide solution or by immersion at room temperature for about 8 min in the chromium trioxide-nitrate solution bath described in Section 2.3.3.2.

8. Class II, Type III

8.1 Scope—This treatment is applicable to all forms and alloys of magnesium, free from attachments or inserts of other metals.

8.2 Coating Properties—The anodic coating consists of two phases: The first-formed subcoating phase is a light tan in color, and causes a dimensional increase of about 0.0002 in. (5 µm) per side; the second and thicker phase, formed at higher voltages, is dark brown and causes a dimensional increase of from 0.001 in. to 0.0012 in. (25 µm to 30 µm) per surface. The paint-base characteristics are similar, and excellent for both the above phases.

8.2.1 The dark-brown coating is hard and highly abrasion-resistant, but it spalls under compression deformation and its formation can cause a loss of fatigue strength of the metal. Such loss is negligible with metal of thickness 0.1 in. (2.5 mm) or over, rising to 3% and 9% for thicknesses of 0.094 in. (2.4 mm) and 0.040 in. (1 mm), respectively, all under reversed bending and corrected for coating thickness. No loss of fatigue strength arises from application of the thin tan coating, and neither is this coating subject to spalling.

8.3 Procedure—Rack and clean with hot alkaline cleaner (2.2) or chromium trioxide (2.3.2), or both, as appropriate, and anodize with alternating current in an electrolyte conforming to the following composition:

<table>
<thead>
<tr>
<th>Composition</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Potassium hydroxide (KOH)</td>
<td>18 to 20 oz (135 to 150 g)</td>
</tr>
<tr>
<td>Aluminum hydroxide (Al(OH)₃) soluble in hot KOH solution (Note 8)</td>
<td>4 oz (30 g)</td>
</tr>
<tr>
<td>Potassium fluoride (K₃F₃)</td>
<td>4.5 oz (34 g)</td>
</tr>
<tr>
<td>Trisodium phosphate (Na₃PO₄)</td>
<td>4.5 oz (34 g)</td>
</tr>
<tr>
<td>Potassium manganate (K₂MnO₄) (Note 9)</td>
<td>2.5 oz (19 g)</td>
</tr>
<tr>
<td>Water (Note 4)</td>
<td>to 1 gal (1 litre)</td>
</tr>
</tbody>
</table>

Dissolve the constituents of the electrolyte in the above order. Maintain the temperature of the electrolyte between 77 and 100°F (25 to 38°C).

8.4 Post-Treatment—After the electrolytic treatment, rinse the parts well in water and immerse for 1 min at room temperature in a solution consisting of 2.7 oz (20 g) of sodium dichromate (Na₂Cr₂O₇·2H₂O) and 13.3 oz (99 g) of ammonium bifluoride (NH₄HF₂) per gal (1 L) of solution. Following the immersion, remove the parts and dry without rinsing. Paint at any convenient time after the parts have dried.

8.5 Power Requirements—The practical range from current density is from 15 to 20 A/ft² (9.3 dm²) with voltages rising to from 70 to 86 (depending on alloy) for the hard brown coating and to 58 V for the thin tan coating. In this current density range, the terminating voltage is reached in from 45 to 60 min for the hard brown coating and in 10 min or less for the thin tan coating. Raise the voltage quickly from zero to 40 or more, and adjust the current and retain at the density desired. Normally the voltage will rise to 55 or more within 1 min, but as the coating forms, the resistance of the circuit increases and the voltage must be raised continuously to maintain the desired current density.

8.6 Solution Control—The operating life of the electrolyte is extremely long, and under normal conditions of use it need never be discarded. Manganese and aluminate deplete slowly with use, whereas the depletion of fluoride and phosphate is extremely slow.

8.6.1 In the case of the dark-brown coating, a lightening of the normal color indicates a depletion of manganate in the solution, and additions shall then be made of 1 oz (7.5 g) of potassium manganate plus 1 ½ oz (11 g) of aluminum hydroxide for each gal (1 litre) of solution. Add the aluminum hydroxide after first dissolving it in a solution containing a weight of potassium hydroxide equal to that of the aluminum hydroxide to be added.

8.6.2 A rough coating indicates either a low, or an excessively-high, alkalinity in the electrolyte. The alkalinity shall be maintained between 10 and 12% KOH, as determined by the method described in Section 13.

8.7 Tank and Rack Materials—Plain black iron is suitable for tanks and cooling coils. Suspend the parts by magnesium-base alloy clips or the like and protect them at the solution level by electroplaters‘ vinyl tape. Magnesium racks may be stripped of coating when necessary by immersion in hot 20% chromium trioxide solution (Note 11).

8.7.1 Divide parts to be treated into two batches each of approximately the same surface area. Rack each of the batches and connect electrically to one or other of the inlet terminals of the power supply so that the magnesium parts constitute both electrodes of the electrolysis bath (Note 12).

8.8 Post-Treatment—After the electrolytic treatment, rinse the parts well in water and immerse for 1 min at room temperature in a solution consisting of 2.7 oz (20 g) of sodium dichromate (Na₂Cr₂O₇·2H₂O) and 13.3 oz (99 g) of ammonium bifluoride (NH₄HF₂) per gal (1 L) of solution. Following the immersion, remove the parts and dry without rinsing. Paint at any convenient time after the parts have dried.

NOTE 11—If, at low initial voltages, the current remains high and coating formation does not proceed, a “surge” current shall be imposed by rapidly raising the voltage to 40, followed by immediately lowering it to zero. The voltage shall then be adjusted in the normal manner to maintain the desired current density.

8.8.1 In the case of the dark-brown coating, a lightening of the normal color indicates a depletion of manganate in the solution, and additions shall then be made of 1 oz (7.5 g) of potassium manganate plus 1 ½ oz (11 g) of aluminum hydroxide for each gal (1 litre) of solution. Add the aluminum hydroxide after first dissolving it in a solution containing a weight of potassium hydroxide equal to that of the aluminum hydroxide to be added.

8.8.2 A rough coating indicates either a low, or an excessively-high, alkalinity in the electrolyte. The alkalinity shall be maintained between 10 and 12% KOH, as determined by the method described in Section 13.

8.8.3 Divide parts to be treated into two batches each of approximately the same surface area. Rack each of the batches and connect electrically to one or other of the inlet terminals of the power supply so that the magnesium parts constitute both electrodes of the electrolysis bath (Note 12).

Note 1—If, at low initial voltages, the current remains high and coating formation does not proceed, a “surge” current shall be imposed by rapidly raising the voltage to 40, followed by immediately lowering it to zero. The voltage shall then be adjusted in the normal manner to maintain the desired current density.

8.8.4 In the case of the dark-brown coating, a lightening of the normal color indicates a depletion of manganate in the solution, and additions shall then be made of 1 oz (7.5 g) of potassium manganate plus 1 ½ oz (11 g) of aluminum hydroxide for each gal (1 litre) of solution. Add the aluminum hydroxide after first dissolving it in a solution containing a weight of potassium hydroxide equal to that of the aluminum hydroxide to be added.

8.8.5 A rough coating indicates either a low, or an excessively-high, alkalinity in the electrolyte. The alkalinity shall be maintained between 10 and 12% KOH, as determined by the method described in Section 13.
(a) **Chrome Pickle Repair Treatment**—The chrome pickle solution for Class I, Type I treatments (for wrought parts) shall be copiously and continuously applied to the damaged area by brush for at least 1 min. The treated surface shall then be thoroughly flushed with water and, when dry, rubbed vigorously with a clean dry rag to remove loose powdery matter which, if allowed to remain, would deleteriously affect the adhesion of superimposed paint.

(b) **Phosphate Repair Treatment**—A solution shall be used conforming to the following composition:
- Monoammonium phosphate (NH₄H₂PO₄) 16 oz (84 g)
- Water (see Note 4) 1 gal (1 litre)
- Ethylene glycol monobutyl ether 13 to 20 fl oz (101 to 156 ml)

The above solution shall be applied copiously to the damaged area by brush in sufficient quantity to keep the surface wet for at least 1 min, or until a medium to dark-gray continuous coating is formed. The treated metal surface shall then be well flushed with water and allowed to dry before the paint is applied.

**ANALYTICAL PROCEDURES**

9. **Fluoride Solutions**

9.1 This method is applicable to the relevant baths of Class I, Type III (Section 5) and Class II, Type I (Section 6) treatments. The concentrations of both hydrofluoric acid (HF) and bifluoride shall be determined by titration with 1 N sodium hydroxide (NaOH) with 1 N sodium hydroxide (NaOH) solution, using phenolphthalein as indicator.

9.2 Maintain the hydrofluoric acid solution described in 5.2 by additions of fresh acid to give a titration of between 10 and 20 ml of 1 N NaOH solution per 2 ml of sample. Maintain the bifluoride solution described in 5.2 so that 10 ml thereof gives a titration of 4.5 to 5.5 ml of 1 N NaOH solution.

9.3 For the hydrofluoric acid the sample shall be drawn into a wax-lined pipet, discharged therefrom into at least 100 ml of distilled water, and titrated immediately. A rubber bulb, or a length of rubber tubing fitted to the suction end of the pipet, shall be used in drawing the sample into the pipet.

10. ** Dichromate Solutions**

10.1 This method is applicable to solutions of the following treatments: Class I, Types I, II, and III; and Class II, Types I and II (Sections 3-7, respectively).

10.2 Add 1 ml of the dichromate solution concerned to 150 ml of distilled water, and mix therewith 5 ml of concentrated hydrochloric acid (HCl, sp gr 1.19) and 5 g of potassium iodide (KI). After at least 2 min, titrate the liberated iodine in the solution with 0.1 N sodium hydroxide (NaOH) solution using starch as an internal indicator.

10.3 **Calculation**:

\[ A \times 0.0415 = B \] \hspace{1cm} (1)

\[ A \times 4.96 = C \] \hspace{1cm} (2)

where:
- \( A \) = millilitres of 0.1 N Na₂S₂O₃ solution,
- \( B \) = pounds per gallon of sodium dichromate (NaCr₂O₇·2H₂O), and
- \( C \) = grams per litre of sodium dichromate.

11. **Nitric Acid Solution**

11.1 This method is applicable to Class I, Types I and II treatment solutions.

11.2 Mix 50 ml of distilled water with 1 ml of the solution in question, and titrate the mixture with 0.1 N sodium hydroxide (NaOH) solution to a pH of 4.0 to 4.05, using a pH meter with a glass electrode.

11.3 **Calculation**:

\[ A \times 0.0505 = B \] \hspace{1cm} (3)

\[ A \times 6.34 = C \] \hspace{1cm} (4)

where:
- \( A \) = millilitres of 0.1 N NaOH solution,
- \( B \) = pints per gallon of nitric acid (HNO₃),
- \( C \) = millilitres of 70% nitric acid per litre.

12. **Phosphoric Acid Solution**

12.1 This method is applicable to the Class II, Type II treatment solution. The phosphoric acid is precipitated as ammonium phosphomolybdate, which is dissolved in an excess of sodium hydroxide, such excess being titrated with standard acid. Interference from fluoride is prevented by converting it to fluoborate, by addition of boric acid.

12.2 **Molybdate Reagent**—Prepare as follows: mix 118 g of 85% molybdic acid (H₂MoO₄) with 400 ml of distilled water, add 80 ml of ammonium hydroxide (NH₄OH) solution (sp gr 0.880) with stirring, and filter when solution is complete. Slowly pour the filtrate, with stirring, into a cold mixture of 400 ml of concentrated nitric acid (HNO₃, sp gr 1.42) and 600 ml of water. Allow the solution to stand over night, and filter.

12.3 **Procedure**—Dilute a 14-ml weighed sample of the solution under examination to 500 ml, and then further dilute 50 ml of this diluted solution to 500 ml. Then again dilute 25 ml of the latter solution to 100 ml, and add 10 g of ammonium nitrate (NH₄NO₃), 1 g of boric acid (H₃BO₃), and 15 ml of concentrated nitric acid (HNO₃, sp gr 1.42). Heat the mixture to 104°F (40°C) and slowly add 40 ml of molybdate reagent. Allow the resulting precipitate of ammonium phosphomolybdate to stand for 30 min, and then filter through a fine-texture, low-ash paper. Wash the precipitate free from acid by means of a 1% solution of potassium nitrate (KNO₃), and mix, together with the filter paper, with an excess of 0.1 N sodium hydroxide (NaOH) solution, using phenolphthalein as indicator. Titrate the excess of NaOH with 0.1 N hydrochloric acid (HCl).

12.4 **Calculation**:

\[ \text{Phosphoric acid (H₃PO₄), } \% = \left( \frac{A - B}{C} \right) \times 0.426 \] \hspace{1cm} (5)

where:
- \( A \) = millilitres of 0.1 N NaOH solution,
- \( B \) = millilitres of 0.1 N HCl, and
- \( C \) = grams of sample in aliquot.

13. **Free Alkali**

13.1 This method is applicable to the Class II, Type III solution. Manganate, fluoride, and phosphate are precipitated by barium nitrate addition, and the filtrate therefrom is titrated with standard acid.

13.2 Pipet a 2-ml sample of the solution under examination into a 25-ml glass-stoppered graduated cylinder, followed by addition of 10 ml of 0.25 N sodium hydroxide (NaOH) solution and 0.5 g of barium nitrate (Ba(NO₃)₂) crystals. Shake this mixture vigorously for 1 min, filter through a medium-texture,
low-ash paper, and wash the precipitate free from alkali, preserving all filtrate. Using a pH meter with a glass electrode, titrate the free alkali in this filtrate to pH 10.5, with 0.25 M hydrochloric acid (HCl).

13.3 Calculation:

\[
\text{Potassium hydroxide, } \% = 2.805 \times \left[ \frac{A}{4} - 2.5 \right]
\]

(6)

where:

\[ A = \text{millilitres of acid used.} \]

14. Keywords

14.1 anodic treatments; magnesium; painting; surface preparation

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Standard Test Method for Hydrogen Loss of Cobalt, Copper, Tungsten, and Iron Powders

1 Scope

1.1 This test method covers the determination of the mass of hydrogen-reducible constituents in the following metal powders: cobalt, copper, iron, and tungsten. This test method consists of subjecting a sample of powder to the action of a hydrogen-containing gas under standard conditions of temperature and time and measuring the resulting loss of mass. This test method is useful for cobalt, copper, and iron powders in the range from 0.05 to 3.0 % oxygen and for tungsten powder in the range from 0.01 to 0.50 % oxygen. This test method does not measure the oxygen contained in oxides such as silicon oxide (SiO₂), aluminum oxide (Al₂O₃), magnesium oxide (MgO), calcium oxide (CaO), titanium dioxide (TiO₂), and so forth that are not reduced by hydrogen at the test temperatures. For total oxygen content, vacuum or inert gas fusion methods are available.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are provided for information only.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Referenced Documents

2.1 ASTM Standards:
- B 215 Practices for Sampling Finished Lots of Metal Powders
- B 243 Terminology of Powder Metallurgy
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3 Terminology

3.1 Definitions—Definitions of powder metallurgy terms can be found in Terminology B 243. Additional descriptive information on powder metallurgy is available in the Related Material section of Vol 02.05 of the Annual Book of ASTM Standards.

4 Significance and Use

4.1 The oxygen content of a powder affects both its green and sintered properties.

4.2 Hydrogen loss is a term widely used in the powder metallurgy industry even though the measurement represents an approximate oxygen content of the powder.

4.3 Oxygen is the most common hydrogen-reducible constituent of the metal powders, and this procedure may be used as a measure of oxygen, reducible under test conditions, if other interfering elements are absent.

5 Interferences

5.1 If carbon or sulfur, or both, is present, they will be largely removed in the test. Their loss in mass is included in the total loss in mass measurement and must be subtracted from the total mass loss.

5.2 If metals or compounds are present that vaporize at the test temperature (such as cadmium, lead, zinc, and so forth), their effect is included in the loss of mass measurement and must be subtracted from the total mass loss.

5.3 If some components are present that are oxidized or hydrided during the test, there is a gain in mass that must be added to the total mass loss.

6 Apparatus

6.1 Furnace, capable of operating at the prescribed temperature.

6.2 Temperature Control, capable of maintaining temperatures to ±15°C (±27°F).

6.3 Gastight Ceramic or Metallic Combustion Tube.

6.4 Flow Meter, to measure flow of hydrogen.

6.5 Supply of Hydrogen and Nitrogen, having dew point lower than −40°C (−40°F).

6.6 Combustion Boat, composed of abundum, quartz, or nickel, depending on test conditions. The boat shall be of such dimensions, for example 75 mm long and 12 mm wide, that the thickness of powder, when uniformly distributed, does not exceed 3 mm (¼ in.).
6.7 Balance, suitable for determining mass to the nearest 0.001 g.

7. Reagents

7.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.\(^4\) Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 Dissociated Ammonia, having a dew point better than −40°C (−40°F). May be used interchangeably with 7.3.

7.3 Hydrogen, having an oxygen content less than 20 ppm and a dew point lower than −40°C (−40°F).

7.4 Nitrogen, having an oxygen content less than 20 ppm and a dew point lower than −40°C (−40°F).

8. Test Specimen

8.1 Obtain the test specimen in accordance with the procedure described in Practices B 215.

8.2 The test specimen shall weigh approximately 5 g.

9. Procedure

9.1 Spread the test specimen to a uniform depth in a combustion boat that has been preconditioned to a constant mass and weighed to the nearest 0.001 g. The depth of powder in the boat shall be approximately 3 mm (⅛ in.). Then weigh the boat and specimen to the nearest 0.001 g.

9.2 Pass the nitrogen through the combustion tube for a period of at least 1 min before insertion of the boat, which shall be placed at the center of the zone of uniform temperature of the furnace.

9.2.1 For cobalt, the temperature is 875°C (1607°F).

9.2.2 For copper, the temperature is 875°C (1607°F).

9.2.3 For iron, the temperature is 1120°C (2050°F).

9.2.4 For tungsten, the temperature is 1120°C (2050°F).

9.3 Start flow of hydrogen, stop the flow of nitrogen and record the time when the hydrogen is introduced.

9.4 Maintain a positive flow of hydrogen through the system for the time of reduction, during which the temperature of the furnace must be held within ±15°C (±27°F).

9.4.1 For cobalt, the time shall be 30 min.

9.4.2 For copper, the time shall be 30 min.

9.4.3 For iron, the time shall be 60 min.

9.4.4 For tungsten, the time shall be 60 min.

9.5 At the end of the prescribed time, discontinue the flow of hydrogen and restart the flow of nitrogen into the tube.

9.6 After nitrogen has been flowing through the combustion tube for at least 1 min, open the tube and withdraw the boat under nitrogen atmosphere to the cooler part of the combustion tube.

9.7 Allow the specimen to cool in the nitrogen atmosphere for 15 to 30 min, then remove the specimen from the tube and permit it to cool to room temperature in a desiccator.

9.8 When the boat has cooled to room temperature, reweigh it to the nearest 0.001 g.

9.9 Alternative Test Method—The following alternative test method may be used satisfactorily if the use of nitrogen is impractical.

9.9.1 Bring the furnace with the combustion tube inserted up to the prescribed temperature.

9.9.2 Spread the test specimen to a uniform depth in the preweighed combustion boat. The depth of powder in the boat should be approximately 3 mm (⅛ in.). Then weigh the boat and specimen to the nearest 0.001 g.

9.9.3 Place the boat containing the specimen in the combustion tube through which dry hydrogen is flowing. The boat is inserted through the hydrogen discharge end of the tube that is open to the atmosphere and at which hydrogen is burning. Place the boat within and at the center of the zone of uniform temperature of the furnace and record the time of insertion.

9.9.4 Maintain a positive flow of dry hydrogen through the system for the time of reduction, during which the temperature of the furnace must be held within ±15°C (±27°F).

9.9.5 At the end of this period, withdraw the entire combustion tube with hydrogen still flowing through it from the furnace and set it on a rack until the specimen has cooled to a point at which it can be removed from the hydrogen atmosphere without danger of reoxidation. This is normally 15 to 30 min. Then remove the specimen from the tube and permit it to cool to room temperature in a desiccator.

9.9.6 When the specimen has cooled to room temperature, reweigh it in the boat to the nearest 0.001 g.

10. Calculation

10.1 Calculate the mass loss as follows:

\[
\text{Loss in mass, } \% = \frac{A - B}{A - C} \times 100
\]  

where:

\[A = \text{ original mass of boat and powder specimen, g;} \]

\[B = \text{ mass of boat and powder specimen after reduction, g;} \]

\[C = \text{ original mass of the boat, g.} \]

10.2 If the hydrogen loss value calculated in 10.1 is to be used as the oxygen content of the powder, correct the value obtained in accordance with 5.1-5.3.

11. Report

11.1 Results shall be reported as hydrogen loss percent to the nearest 0.01 %. Duplicate results within ±0.02 % in absolute terms shall be considered satisfactory.

12. Precision and Bias

12.1 A statement of precision provides guidelines as to the kind of variability that can be expected between test results when the method is used in one or more reasonably competent laboratories.

12.2 The precision of this test method has not been determined by a statistically valid interlaboratory test because of the limited number of participating laboratories (less than six).

12.3 The following precision data were developed using the procedures contained in Test Method E 159 from an interlaboratory test conducted by four laboratories in 1994. The percent loss in mass in hydrogen was determined for four samples, a cobalt powder, a copper powder, an iron powder, and a tungsten powder. Except for the use of only four laboratories, Practice E 691 was followed for the design and analysis of the data. The details are given in an ASTM research report.

12.4 The precision information given below is for the results. The results were obtained from the running of three replicates by each lab on each sample.

<table>
<thead>
<tr>
<th></th>
<th>Cobalt</th>
<th>Copper</th>
<th>Iron</th>
<th>Tungsten</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average, %</td>
<td>0.45</td>
<td>0.15</td>
<td>0.22</td>
<td>0.47</td>
</tr>
<tr>
<td>r, %</td>
<td>0.03</td>
<td>0.03</td>
<td>0.03</td>
<td>0.09</td>
</tr>
<tr>
<td>R, %</td>
<td>0.12</td>
<td>0.03</td>
<td>0.07</td>
<td>0.11</td>
</tr>
</tbody>
</table>

12.5 Repeatability, \( r \), is a measure of the variability between test results obtained within a single laboratory in the shortest practical period of time by a single operator with a specific set of test apparatus using test specimens taken at random from a single quantity of homogeneous material.

12.6 Reproducibility, \( R \), is a measure of the variability between test results obtained in different laboratories using test specimens taken at random from a single quantity of homogeneous material.

12.7 Bias cannot be stated since there is no certified reference material.

13. Keywords

13.1 chemical analysis; cobalt powder; copper powder; hydrogen loss; iron powder; metal powders; oxygen content; tungsten powder
Standard Test Method for Acid-Insoluble Content of Copper and Iron Powders

1 This test method is under the jurisdiction of ASTM Committee B-9 on Metal Powders and Metal Powder Products and is the direct responsibility of Subcommittee B09.02 on Base Metal Powders.


Based on the method developed by the Metal Powder Association (now the Metal Powder Producers Association of the Metal Powder Industries Federation) and described in MPI Standard 6-54, “Determination of Acid Insoluble Matter in Iron and Copper Powders,” which is a standard of the MPIF.

Annual Book of ASTM Standards, Vol 02.05.

Annual Book of ASTM Standards, Vol 03.05.


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1. Scope

1.1 This test method covers the determination of the mineral-acid-insoluble matter content of copper and iron powders in amounts under 1.0 %.

1.2 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:

B 215 Practices for Sampling Finished Lots of Metal Powders

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Summary of Test Method

3.1 The sample is dissolved in the appropriate acid (nitric acid (HNO₃) for copper, hydrochloric acid (HCl) for iron). The insoluble matter is filtered out and ignited in a furnace at 980°C for 1 h.

4. Significance and Use

4.1 The purpose of this test method is to determine the amount of gangue, refractory, inert, etc., materials, that may adversely affect compacting tools and sintered properties of components formed from copper and iron powders.

4.2 The insoluble matter consists of those nonmetallic substances that do not dissolve in the mineral acid used to dissolve the metal. In copper powder, which is treated with nitric acid, the acid-insoluble matter includes silica, silicates, alumina, clays, and other refractory materials that may be introduced either as impurities in the raw material or from the furnace lining, fuel, etc.; lead sulfate may also be present. In iron powder, which is treated with hydrochloric acid, the insoluble matter may include carbides in addition to the substances listed above. The test method excludes insoluble material that is volatile at the ignition temperature specified.

5. Interferences

5.1 Any metallic tin present in the copper powder will be converted into the insoluble tin oxide by the nitric acid treatment; in such cases, provision shall be made for the determination of tin oxide and the appropriate correction applied.

6. Apparatus

6.1 Apparatus and reagents shall conform to the requirements prescribed in Practices E 50.

6.2 Casseroles, 250 mL and 750 mL.

6.3 Filter Paper, Whatman No. 541 or one of equivalent pore size and ash content.

7. Reagents

7.1 Ammonium Iodide (NH₄I).

8. Sampling

8.1 The metal powder shall be sampled in accordance with Practices B 215.

8.2 Store the test sample in a tightly stoppered bottle to protect it from moisture which promotes oxidation of copper and iron.

COPPER POWDER

9. Procedure

9.1 Transfer 5 g of the sample, weighed to the nearest 1 mg, to a 250-mL covered casseroles. Add 100 mL of HNO₃(1 + 1) and let stand at room temperature until the reaction is complete. Place the casseroles on a hot plate and boil until the volume is reduced to 50 mL. Cool, dilute with water to about 100 mL, and bring to a boil. Filter, and wash with hot water until all traces of blue color disappear.

9.2 Transfer paper and precipitate to a porcelain crucible, weighed to the nearest 0.1 mg. Dry, and then ignite in a furnace
at 980°C for 1 h. Cool in a desiccator and reweigh. The difference in weight is the insoluble matter. Reserve the residue.

**Note 1**—If the ignited residue reserved from 9.2 is suspected to contain tin oxide, add 5 g of NH₄I to the crucible. Reheat the crucible and contents in air with a bunsen burner to a dull-red temperature or place in a furnace at 600°C minimum for 15 min or until all fumes have been dispelled, remove the crucible from heat and cool. Add 2 to 3 mL of HNO₃, evaporate to dryness, ignite and weigh. Repeat the treatment with NH₄I and HNO₃ until constant weight is obtained. The loss in weight represents tin oxide.

**10. Calculation**

10.1 Calculate the percentage of insoluble matter as follows:

\[
\text{Insoluble matter, percent } = \left( \frac{A - B}{C} \right) \times 100
\]

where:

- \( A \) = insoluble matter, g,
- \( B \) = correction for grams of tin oxide, if present (Note 1), and
- \( C \) = sample used, g.

**IRON POWDER**

**11. Procedure**

11.1 Transfer 5 g of the sample, weighed to the nearest 1 mg, to a 750-mL covered casserole. With caution, add 100 mL of HCl (1 + 1) (Note 2), and let stand at room temperature until the reaction is complete. Heat the solution to boiling on a hot plate, add 150 mL of water, and reheat to boiling. Filter, and wash the residue alternately with hot HCl (1 + 25) and hot water, 6 times with each, to ensure the removal of all iron salts.

**Note 2**—If it is desired to exclude carbides from the reported insoluble matter, add 20 mL of HNO₃ to the HCl (1 + 1).

11.2 Transfer the paper and residue to a porcelain crucible, weighed to the nearest 0.1 mg, and ignite in a furnace at 980°C for 1 h. Cool in a desiccator and reweigh. The difference in weight is the insoluble matter.

**12. Calculation**

12.1 Calculate the percentage of insoluble matter as follows:

\[
\text{Insoluble matter, percent } = \left( \frac{A}{B} \right) \times 100
\]

where:

- \( A \) = insoluble matter, g, and
- \( B \) = sample used, g.

**13. Report**

13.1 Report the total insoluble matter as a percentage to the nearest 0.01 %.

**14. Precision and Bias**

14.1 Precision—The following precision data were developed using the procedures contained in Test Method E 194 from an interlaboratory study that performed six sets of tests. The percent insoluble was determined for four samples: a –325 mesh iron, a –60 mesh iron, a –325 mesh copper, and a –60 mesh copper. The different particle sizes were used to determine if there were any effects on the precision of testing based on differences in particle size distribution. Practice E 691 was followed for the design and analysis of the data; the details are given in an ASTM Research Report.

14.1.1 The precision information given below is for the comparison of two test results. The results were obtained from the running of three replicates in each test on each sample.

<table>
<thead>
<tr>
<th>Sample Size</th>
<th>Average, %</th>
<th>( S_p ), %</th>
<th>( r ), %</th>
<th>( R ), %</th>
</tr>
</thead>
<tbody>
<tr>
<td>–325 Iron</td>
<td>0.29</td>
<td>0.051</td>
<td>0.06</td>
<td>0.14</td>
</tr>
<tr>
<td>–60 Iron</td>
<td>0.08</td>
<td>0.025</td>
<td>0.03</td>
<td>0.07</td>
</tr>
<tr>
<td>–325 Copper</td>
<td>0.11</td>
<td>0.036</td>
<td>0.04</td>
<td>0.10</td>
</tr>
<tr>
<td>–60 Copper</td>
<td>0.09</td>
<td>0.063</td>
<td>0.05</td>
<td>0.18</td>
</tr>
</tbody>
</table>

14.1.2 Duplicate results from the same laboratory should be considered acceptable at the 95 % confidence level unless they differ by more than \( r \), the repeatability interval.

14.1.3 Duplicate results from two different laboratories should be considered acceptable at the 95 % confidence level unless they differ by more than \( R \), the reproducibility interval.

14.2 Bias—No information can be presented on the bias of the procedure in Test Method E 194 for measuring the acid insoluble content of copper and iron powders because no material having an accepted reference value is available.
Standard Guide for Identification of Metals and Alloys in Computerized Material Property Databases

This standard is issued under the fixed designation E 1338; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide covers the identification of metals and alloys in computerized material property databases. It establishes essential and desirable data elements that serve to uniquely identify and describe a particular metal or alloy sample as well as properties that identify a given metal or alloy in general.

1.1.1 This guide does not necessarily provide sufficient data elements to describe weld metal, metal matrix composites, or joined metals.2

1.1.2 The data element identified herein are not all germane to every metal or alloy group.

1.1.3 Different sets of data elements may also be applied within a given metal or alloy group depending on conditions or applications specific to that metal or alloy group. Further, within a particular metal or alloy group, different sets of data elements may be used to identify specific material conditions.

1.1.4 Table 1 on Recommended Data Elements and Tables 2-17 on values for specific data elements appear at the end of this guide.

1.2 Some of the data elements in this guide may be useful for other purposes. However, this guide does not attempt to document the essential and desirable data element for any purpose except for the identification of metals and alloys in computerized material property databases. Other purposes, such as material production, material procurement, and material processing, each may have different material data reporting requirements distinct from those covered in this guide. A specific example is the contractually required report for a material property testing series. Such a report may not contain all the data elements considered essential for a specific computerized database; conversely, this guide may not contain all the data elements considered essential for a contracted test report.

1.3 Results from material tests conducted as part of the procurement process are often used to determine adherence to a specification. While this guide includes a number of test result data elements, such data elements are included in this guide only for the purposes of material identification.

1.4 Reporting of contracted test results, such as certification test results, shall follow the requirements described in the material specification, or as agreed upon between the purchaser and the manufacturer.

1.5 This guide contains a limited number of data elements related to material test results. These data elements are for material identification purposes and are not intended to replace the more detailed sets of data elements listed in guides such as Guide E 1313 covering data recording formats for mechanical testing of metals. For material identification purposes, the data elements in this guide include typical, nominal, or summary properties normally derived from a population of individual specimen tests. If warranted by the scope of a particular database system, the system might provide links between the material identification data elements given in this guide, and the individual specimen test results recorded in accordance with other guides corresponding to particular test methods.

1.6 Material Classes—See ANSI/AWS A9.1-92 for arc welds, Guide E 1308 for polymers, Guide E 1309 for composite material, and Guide E 1471 for fibers, fillers, and core materials. ASTM Committee E49 is developing guides for other material classes.

2. Referenced Documents

2.1 ASTM Standards:

E 8 Test Methods for Tension Testing of Metallic Materials2
E 8M Test Methods for Tension Testing of Metallic Materials [Metric]2
E 380 Practice for Use of the International System of Units (SI) the Modernized Metric System3
E 527 Practice for Numbering Metals and Alloys (UNS)4
E 616 Terminology Relating to Fracture Testing5
E 1308 Guide for Identification of Polymers (Excludes Thermoset Elastomers) in Computerized Material Property Databases6

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1 This guide is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.01 on Ancillary Activities. This guide was developed in cooperation with Committee B07 on Light Metals and Alloys.


2 Annual Book of ASTM Standards, Vol 03.01.


4 Discontinued; see 1995 Annual Book of ASTM Standards, Vol 01.01.

5 Discontinued; see 1999 Annual Book of ASTM Standards, Vol 14.01.

6 Discontinued; see 1995 Annual Book of ASTM Standards, Vol 03.01.
4. Significance and Use

4.1 This guide describes the types of information that are indispensable for uniquely identifying a metal or alloy in a computerized database. The purpose is to facilitate standardized storage and retrieval of the information with a computer, and allow meaningful comparison of data from different sources.

4.2 Many numbering systems for metals and alloys have been developed which are based on their chemical compositions. Separate systems have also evolved to describe the thermomechanical condition of metals and alloys in order to narrow their description. It is the separation into logical data elements from these complex, historically significant, and overlapping systems of identification that is the challenge in the identification of metals and alloys within computerized databases.

4.3 This guide is intended to provide a common starting point for designers and builders of materials property databases. This guide generally identifies the contents of the database in terms of data elements, but does not recommend any particular logical or physical database design. A database builder has considerable flexibility in designing a database schema, and it is intended that this guide support that flexibility.

4.4 It is recognized that material property databases will be designed for different levels of material information and for different purposes. For example, a database developed by an industry trade group might only identify typical properties generally representative of those for a particular metal or alloy, and not actual values measured on a specific sample. On the other hand, a business might desire to manage data on specific lots it procures, or even properties of a specific piece or sample from a lot. Consequently, some of the data elements identified in this guide might not be applicable in every database instance.

4.5 The extent of material identification implemented in a particular database depends on its specific purpose. A single organization may include substantial detail in its database. Less detail may be included in a common database used by several organizations because of commercial and other considerations. Since metals and alloys are diverse and the technologies are always changing, recommendations should not be regarded as exclusive of additional data elements for material identification. The recommended data elements should be expanded if additional detailed information which serves to identify materials is to be recorded.

4.6 A number of data elements are considered essential to any database and need to exist in the database. Data elements are considered essential if they are required for users to have sufficient information to interpret the data and be confident of their ability to compare sets of data for materials from different sources. Failure to complete an essential data element may render the record unusable in a database or in data exchange. Essential refers to the quality or completeness of recorded data, and does not necessarily have direct meaning relative to database structure. In some cases, the identified data element might be accommodated within a particular database without explicitly including a field just for the essential data element. Additionally, a database schema may require additional data fields to be not null to maintain data record integrity or to implement a mandatory data relationship. These additional fields are beyond the scope of this guide. Finally, it is also noted that a data element identified as essential in this guide might not be relevant for a database created for a specific application of limited scope.

4.7 This guide presents a listing of the data elements and does not intend to define any single organization of the data elements to be used in either a logical or physical model for the database. The data element lists are divided by group headings for discussion purposes only. The group headings are not intended to identify normalization of the database model; this is left to the database designer.

4.8 Numerous data elements listed in this guide may need to be repeated to identify even a single material. Depending on the database purpose or design, it may be appropriate to design the database to enable additional repeatable data elements. How the database should accommodate multiple values for a given data element is another question left to the database designer.

5. Guidelines

5.1 The data elements recommended for material identification are listed in Table 1. Descriptions of each data element are provided in Section 6. Table 1 includes: (1) a data element number, (2) a descriptive name for each data element, (3) data type, and (4) category sets, value sets, or units.

5.1.1 Data Element Number—A reference number for ease of dealing with the individual data elements within this guide. The data element number has no permanent value and does not become part of the database itself.
5.1.2 Descriptive Data Element Name—The complete and unambiguous name, descriptive of the data element being identified.

5.1.3 Data Type—The kind of data to be included in the data element, such as the type of number, character string, and date.

5.1.3.1 String—Textual data element.

5.1.3.2 Real—Any rational, irrational, and scientific real number.

5.1.3.3 Integer—An integral number.

5.1.3.4 Date—The calendar date in the Gregorian calendar in the YYYYY-MM-DD format.

5.1.4 Category Set, Value Set, or Units—A listing of the types of information that would be included in the data element or, in the case of properties or other numeric data, the units in which the numbers are expressed. Candidate values for both category and value sets are frequently given in separate tables in this guide. The database implementation should provide some means of maintaining these lists of allowable values and presenting them to the database user. The distinction between category and value sets identified below primarily relates to the degree of control over additions to these lists of values.

5.1.4.1 A category set is a closed set listing all possible (or acceptable) values the data element may take. Because this guide is intended to apply to databases with different scopes and purposes, values listed in sample tables in this guide are generally not identified as category sets, but it may be appropriate to treat them as such in a particular database application. The database application should control or restrict the addition of a new value to a category set.

5.1.4.2 A value set is a representative set listing sample, but not necessarily all acceptable values the data element may take. In this case, the database application generally should provide some means for a new value to be added to the value set maintained by the database system.

5.1.4.3 The units listed are SI, in accordance with Practice E 380, followed by inch-pound units in parentheses. Although both sets of units are listed in this guide, it is left to the database designer to decide whether a single data unit system will be used to store values for a given data element or whether the database design will support storage of data in mixed units with necessary tracking of units for each data element entry. Data elements for tracking units are generally not identified in this guide.

5.2 This guide does not provide specific recommendations relative to either the logical or the physical design of the database for storing material property data. Accordingly, internal data element associated only with a particular database model, such as index fields or fields containing counts of repeating data, are not provided since their existence will vary depending on the particular database design.

5.3 The value sets and category sets identified in this guide are to be used for the identification of any type of metal or alloy. Aluminum, copper, and steels are three specific types of material which are identified in this guide to serve as examples and because there has been specific interest in providing guidelines for these common metal families. For some of the lists of values, candidate values are identified and associated with one of these types of metal.

5.4 Data elements are provided for characterization of a material’s microstructure in terms of grain size measurements and description of its microstructure, including microstructure classification. Additional data elements should be added for other aspects of metallographic characterization if judged by the database designer to be appropriate for a particular system. Examples of additional items that should be considered for addition are the following: distribution of elements to grain boundaries, presence of voids or inclusions, phase content, and X-ray diffraction measurements. Images are often an important part of the record of materials characterization and should be made available to the user of the database if appropriate for a particular system. Although this guide does not recommend standard means to handle records of images, data elements associated with the storage or indexing of images should be added when appropriate.

6. Description of Data Elements

6.1 The individual data elements recommended for the identification of metals and alloys are described in this section. The data elements are numbered consecutively matching the numbers listed in Table 1. Section headings are used to group data elements both in the following paragraphs and in Table 1. Whether or not these logical groupings have any significance to an actual database will depend on the particular database model used in that instance. Provisions should be designed in the database for repeated values of data elements, or for sets of data elements when it is indicated that they may repeat.

6.1.1 Primary Identifiers—Features which distinguish one material type from another and allow materials data to be grouped by broad material type. The existence of some of the following data elements will likely vary depending on the scope of the database.

6.1.1.1 Material Class (1)—The broad material class, in this case, metal (as distinct from ceramic, polymer, composite, and so forth).

6.1.1.2 Family Name (2)—The broad alloy family defined by the primary alphabetical identifier defined in Practice E 527. Examples are copper and copper alloys and tool steels. See Table 2.

6.1.1.3 Family Subclass (3)—A more specific division of the family name descriptive of a particular alloy system; for example, some subclasses of AISI and SAE Carbon and Alloy Steel may be chromium-molybdenum, rephosphorized, low carbon, and high carbon. The secondary division of some of the alphabetical identifiers in Practice E 527 are additional examples of subclasses. See Table 3.

6.1.1.4 Common Name (4) (essential and may repeat)—Any frequently used domestic or international name for a particular metal or alloy. It may be a commercial or trade name which has broad usage or part of a specification designation, such as 4140. A specific alloy may have more than one common name. Recommended standard common names include those used in Practice E 527.

6.1.1.5 Application Group (5)—A broad end usage for the metal (for example, electrical conductor or pressure vessel).

6.1.1.6 Product Group (6)—A broad end product (for example, water tube, bus bar, wire, or rivet).
6.1.2 Material Specification (may repeat)—The following data elements identify the material specification which is recorded on drawings, requisitions, standards document, and design data in order to procure or fabricate a material. The material being described may be covered by more than one specification. Material standard and specification are synonyms.

6.1.2.1 UNS Number (7) (essential if a UNS number has been defined for the particular alloy)—The assigned number of the metal or alloy within the United Numbering System which generally identifies the chemical composition and corresponds to one or more standard material specifications.

6.1.2.2 Specification Organization (8) (essential)—A company; industry; or national, regional, or international organization issuing the specification (for example, ASTM or SAE).

6.1.2.3 Specification Number (9) (essential)—The specification number within the previously referenced organization.

6.1.2.4 Specification Version (10) (essential)—The year or revision code of the specification.

6.1.2.5 Designation Keyword (11) (essential and may repeat)—The keyword used in the material specification to identify the type of designation or classification for the metal or alloy as identified by the Designation Value data element. Provisions for repeated pairs of Designation Keyword and Designation Value data elements should be provided. See Table 4.

6.1.2.6 Designation Value (12) (essential and may repeat)—The value defined in the material specification for the material type or classification identified in the Designation Keyword data element.

6.1.3 Specified Properties—The following data elements identify chemical composition and mechanical properties identified in the material specification as requirements for certification of the material relative to the material specification.

6.1.4 Composition Requirements (may repeat)—The limits for chemical composition for the material identified in the material specification are recorded by repetition of the following data elements for the chemical element, fraction type, units, and limiting values.

6.1.4.1 Element Symbol (13)—The IUPAC symbol for the chemical element or the identifier for a combination of chemical elements which might be specified to be measured as a unit.

6.1.4.2 Fraction Type (14)—The parameter that labels the fractional composition value. Preferred values are mass, volume, or mole. Due to their extensive usage for metals, weight fraction is an acceptable alternative to mass fraction, and atomic fraction is likewise an alternative to mole fraction. This data element is optional if the definition of the database restricts chemistry values to a single fraction type.

6.1.4.3 Composition Units (15)—The scale of the composition fraction. Allowable values are percent (%) and parts per million (ppm). This data element is optional if the definition of the database restricts chemistry values to a single set of units.

6.1.4.4 Minimum Specified Composition (16)—The minimum value of chemical composition specified in the material specification in terms of the Fraction type and Composition Units.

6.1.4.5 Maximum Specified Composition (17)—The maximum value of chemical composition specified in the material specification in terms of the Fraction type and Composition Units.

6.1.5 Mechanical Properties Requirements (may repeat)—Data element numbers 17 through 38 listed in Table 1 catalog the tensile, hardness, and Charpy impact energy property requirements identified in the material specification. Data elements are included to record the conditions for these tests if specific ones are identified in the specification. All of these data elements are self-descriptive. Tables 5-8 contain value sets or category sets for data elements associated with mechanical properties. The database designer should generalize and add to the data element numbers 35–38 for Charpy impact energy to fracture requirements if needed to encompass other types of impact requirements.

6.1.6 Primary Material Producer:

6.1.6.1 Original Producer (40)—The name of the manufacturer who made the metal or alloy.

6.1.6.2 Country of Origin (41)—The three-character code described in ISO 3166 for the producer’s country.

6.1.6.3 Producer’s Facility (42)—The name of the manufacturing plant.

6.1.6.4 Production Date (43)—The date of material production assigned by the primary material producer.

6.1.6.5 Primary Process Type (44)—Identification of the primary process used to produce the material.

6.1.6.6 Melt Practice (45)—The name of the melting procedure. See Table 9.

6.1.6.7 Cast Practice (46)—The name of the primary casting procedure, for example, ingot or continuous. See Table 10.

6.1.6.8 Heat Number (47) (essential)—The identifying number assigned by the material producer which generally identifies all the material produced in the same primary process event. This number is often associated with the final melt and identifies the common chemical composition of the heat of material.

6.1.7 Material Processing (may repeat)—The following set of data elements should be repeated for each of the significant processes applied to the material following its primary production.

6.1.7.1 Processor’s Name (48)—The name of the organization that processed the material after initial production.

6.1.7.2 Processor’s Country (49)—The three-character code described in ISO 3166 for the producer’s country.

6.1.7.3 Processor’s Facility Name (50)—The name of the processing plant.

6.1.7.4 Processor’s Assigned Production Date (51)—The date of the material’s processing assigned by the processor.

6.1.7.5 Process Type (52) (essential)—Descriptive term or phrase identifying the process performed.

6.1.7.6 Process Lot Number (53)—The number assigned by the processor to identify the material from the same heat and of the same form, condition, and size, and receiving the same heat treatment in a batch process or one continuous process under
the same conditions of temperature, time at heat, and atmosphere. This material processing stage is often associated with the certification of the material in accordance with the material specification.

6.1.8 Heat Treatment (may repeat)—The following set of data elements should be repeated for each heat treatment cycle applied to the material. Provision should be included to know the sequence the cycles are applied.

6.1.8.1 Thermal Step Type (54)—The name or description of one step of the thermal process.

6.1.8.2 Time of Thermal Step (55)—The time in hours of the thermal processing step.

6.1.8.3 Thermal Step Temperature (56)—The temperature used during the thermal processing step.

6.1.8.4 Heating Environment (57)—A brief description of the environment in which the heating treatment was performed.

6.1.8.5 Heating Rate (58)—The rate of temperature increase in degrees per hour.

6.1.8.6 Cooling Environment (59)—A brief description of the environment in which the cooling treatment was performed.

6.1.8.7 Cooling Rate (60)—The rate of temperature decrease in degrees per hour.

6.1.9 Product Detail—Data elements which identify the characteristic size and shape of the product or sample being identified, if applicable.

6.1.9.1 Product Forming Method (61)—The name of the procedure for forming the final shape of the product.

6.1.9.2 Product Identifier (62)—The manufacturer’s code for a product, which may be a key to a record describing the product attributes.

6.1.9.3 Product Shape (63)—The overall geometry of the product, such as cylindrical or rectangular.

6.1.9.4 Product Form (64)—The overall form of the product, such as strip, bar, or wire.

6.1.9.5 Dimension Type (65)—A term describing the basis for the dimensional values, such as nominal or measured.

6.1.9.6 Length (66)—The longest dimension of the product.

6.1.9.7 Width (67)—The second longest dimension of the product for noncircular sections.

6.1.9.8 Thickness (68)—The third longest dimension of the product for noncircular sections.

6.1.9.9 Outside Diameter (69)—The outside diameter dimension of a circular section.

6.1.9.10 Wall Thickness (70)—The wall thickness of hollow sections in circular sections or a characteristic wall thickness for irregular forgings.

6.1.9.11 Weight (71)—The weight of the product or sample being identified.

6.1.9.12 Fabrication History (72)—Identification or description of processes such as machining, joining, forming, or assembling. Examples include: machine and welded per ABC Company Manufacturing Routing 12345; final machined using low-stress grinding.

6.1.9.13 Service History (73)—Indicates briefly any service exposure conditions encountered by the material, such as nuclear radiation or high-temperature exposure.

6.1.10 Material Characterization—The actual chemical composition, mechanical properties, and microstructure descriptors measured on a sample or samples of the material and used to certify or otherwise characterize the material being identified. The specific nature of these properties will vary depending on the purpose of the database, but generally speaking, the data used to characterize a material for identification purposes will be summary or average data from more than one individual test. If specific lots of material are being identified, then these properties will often be recorded as part of the certification data measured for that particular material. If materials are being described in a more general sense, then the purpose of the database might be to record typical properties representing many different heats or lots of material. The data elements listed in this guide are intended to provide for either of these purposes. On the other hand, if individual specimen results from multiple tests are being recorded, then guides such as Guide E 1313, which provide standard data recording formats for specific tests, should be consulted as additional data elements are recommended to record test details.

6.1.11 Measured Chemical Composition (may repeat)—The chemical composition measured on a sample or samples of the material used to certify or otherwise characterize the material. The following group of data elements should be repeated for each of the chemical elements.

6.1.11.1 Source of Chemical Composition Data (74)—Identification of the source for the chemical composition data. Examples are: producer’s ladle analysis and check analysis on a product sample.

6.1.11.2 Element Symbol (75)—The IUPAC symbol for the chemical element or the identifier for a combination of chemical elements which might be measured as a unit to characterize the material.

6.1.11.3 Fraction Type (76)—The parameter that labels the fractional composition value. Preferred values are mass, volume, or mole. Due to their extensive usage for metals, weight fraction is an acceptable alternative to mass fraction, and atomic fraction is likewise an alternative to mole fraction. This data element is optional if the definition of the database restricts chemistry values to a single fraction type.

6.1.11.4 Composition Units (77)—The scale of the composition fraction. Allowable values are percent (%) and parts per million (ppm). This data element is optional if the definition of the database restricts chemistry values to a single set of units.

6.1.11.5 Measured Composition (78)—The actual or typical composition value for the element measured for the particular material being identified in terms of the Fraction Type and Composition Units.

6.1.12 Measured Mechanical Properties and Microstructure (may repeat)—Data elements Numbers 77 through 103, listed in Table 1, record mechanical properties, and Numbers 104 through 107 record microstructure descriptors measured on a sample or samples of the material being identified. These data elements are self-descriptive. Tables 5-8, Table 16, and Table 17 contain value sets or category sets for data elements associated with mechanical properties. Depending on the purpose of the database and the value assigned to the data elements for recording the source of each property, either...
average, individual, or typical values can be used to characterize the material. These fields will need to repeat to accommodate individual results from multiple tests and tests at a variety of conditions. Similar data elements should be added to record other physical or mechanical properties if they are used to characterize the material. Specifically, the database designer should generalize and add to the data element Numbers 98–103 for Charpy impact energy to fracture measurements if needed to encompass other types of impact test results.

7. Keywords

7.1 alloys; computerized databases; computerized material property databases; databases; data elements; metals

<table>
<thead>
<tr>
<th>Number</th>
<th>Data Element Descriptive Name</th>
<th>Data Type</th>
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<td>5</td>
<td>Application group</td>
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<td></td>
</tr>
<tr>
<td>6</td>
<td>Product group</td>
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<td></td>
</tr>
</tbody>
</table>

7. UNS Number

8. Specification organization

9. Specification number

10. Specification version

11. Designation keyword

12. Designation value

13. Element symbol

14. Fraction type

15. Composition units

16. Minimum specified composition

17. Maximum specified composition

18. Orientation of tensile specimen for certification

19. Location of tensile specimen for certification

20. Tensile test temperature for certification

21. Minimum ultimate tensile strength

22. Maximum ultimate tensile strength

23. Minimum yield strength

24. Maximum yield strength

25. Yield strength determination method

26. Yield strength offset or extension

27. Minimum elongation

28. Maximum elongation

29. Original gage length

30. Minimum reduction of area

31. Maximum reduction of area

32. Location of hardness measurement for certification

33. Minimum hardness

34. Maximum hardness

35. Hardness scale

36. Location of Charpy specimen for certification

37. Temperature of Charpy test for certification

38. Minimum Charpy impact energy

39. Maximum Charpy impact energy

40. Original producer

41. Country of origin

42. Producer’s facility

43. Production date

44. Primary process type

45. Melt practice

46. Cast practice

47. Heat number

**TABLE 1 Recommended Data Elements for the Identification of Metals and Alloys**

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---

6
<table>
<thead>
<tr>
<th>Number</th>
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**Heat Treatment**

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<td>58</td>
<td>Heating rate</td>
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<td>°C/h (°F/h)</td>
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<td>59</td>
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<td>Cooling rate</td>
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**Product Detail**

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<td>Product form</td>
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<td>Dimension type</td>
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<td>Thickness</td>
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**Measured Chemical Composition**

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<td>Element symbol</td>
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**Measured Mechanical Properties**

**Measured Tensile Properties**

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<tr>
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<th>Data Type</th>
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<td>80</td>
<td>Orientation of test specimen</td>
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<td>Location of tensile specimen</td>
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<td>83</td>
<td>Ultimate tensile strength</td>
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<td>Number of tensile strength tests, if averaged</td>
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<td>85</td>
<td>Yield strength</td>
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<td>87</td>
<td>Yield strength offset or extension</td>
<td>Real</td>
<td>%</td>
</tr>
<tr>
<td>88</td>
<td>Number of yield strength tests, if averaged</td>
<td>Integer</td>
<td></td>
</tr>
<tr>
<td>89</td>
<td>Total elongation</td>
<td>Real</td>
<td>%</td>
</tr>
<tr>
<td>90</td>
<td>Original gage length</td>
<td>Real</td>
<td>mm (in.)</td>
</tr>
<tr>
<td>91</td>
<td>Number of elongation tests, if averaged</td>
<td>Integer</td>
<td></td>
</tr>
<tr>
<td>92</td>
<td>Type of elongation</td>
<td>String</td>
<td>Value set in Table 16</td>
</tr>
<tr>
<td>93</td>
<td>Reduction of area</td>
<td>Real</td>
<td>%</td>
</tr>
<tr>
<td>94</td>
<td>Number of reduction of area tests, if averaged</td>
<td>Integer</td>
<td></td>
</tr>
</tbody>
</table>

**Measured Hardness**

<table>
<thead>
<tr>
<th>Number</th>
<th>Data Element Descriptive Name</th>
<th>Data Type</th>
<th>Category Set, Value Set, or Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>95</td>
<td>Source or basis for hardness measurement</td>
<td>String</td>
<td></td>
</tr>
<tr>
<td>96</td>
<td>Location of hardness measurement</td>
<td>String</td>
<td>Value set in Table 6</td>
</tr>
<tr>
<td>97</td>
<td>Hardness value</td>
<td>Real</td>
<td></td>
</tr>
<tr>
<td>98</td>
<td>Hardness scale</td>
<td>String</td>
<td>Category set in Table 8</td>
</tr>
<tr>
<td>99</td>
<td>Number of hardness readings, if averaged</td>
<td>Integer</td>
<td></td>
</tr>
</tbody>
</table>

**Measured Charpy Impact Energy to Fracture**

<table>
<thead>
<tr>
<th>Number</th>
<th>Data Element Descriptive Name</th>
<th>Data Type</th>
<th>Category Set, Value Set, or Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>Source or basis for Charpy measurements</td>
<td>String</td>
<td>Value set in Table 6</td>
</tr>
<tr>
<td>101</td>
<td>Location of Charpy specimen</td>
<td>String</td>
<td></td>
</tr>
<tr>
<td>102</td>
<td>Temperature of Charpy test</td>
<td>Real</td>
<td>°C (°F)</td>
</tr>
<tr>
<td>103</td>
<td>Charpy specimen size</td>
<td>String</td>
<td>Category set in Table 17</td>
</tr>
<tr>
<td>104</td>
<td>Charpy impact energy</td>
<td>Real</td>
<td>J (ft-lbf)</td>
</tr>
<tr>
<td>105</td>
<td>Number of Charpy tests, if averaged</td>
<td>Integer</td>
<td></td>
</tr>
</tbody>
</table>
### TABLE 1  Continued

<table>
<thead>
<tr>
<th>Number&lt;sup&gt;A&lt;/sup&gt;</th>
<th>Data Element Descriptive Name</th>
<th>Data Type</th>
<th>Category Set, Value Set, or Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>106</td>
<td>Grain size measurement</td>
<td>Real</td>
<td></td>
</tr>
<tr>
<td>107</td>
<td>Scale for grain size</td>
<td>String</td>
<td></td>
</tr>
<tr>
<td>108</td>
<td>Basis for grain size</td>
<td>String</td>
<td></td>
</tr>
<tr>
<td>109</td>
<td>Description of microstructure</td>
<td>String</td>
<td></td>
</tr>
</tbody>
</table>

<sup>A</sup> Data element numbers are provided for information only.

<sup>B</sup> Essential data element, as described in 4.6.

<sup>C</sup> Provisions should be made in the database for repeated values of this data element, or for the set of data elements in this section.

### TABLE 2  Category Set for Family Name as Listed in Practice

<table>
<thead>
<tr>
<th>E 527</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum and aluminum alloys</td>
</tr>
<tr>
<td>Copper and copper alloys</td>
</tr>
<tr>
<td>Rare earth and rare earth-like metals and alloys</td>
</tr>
<tr>
<td>Low melting point metals and alloys</td>
</tr>
<tr>
<td>Nickel and nickel alloys</td>
</tr>
<tr>
<td>Precious metals and alloys</td>
</tr>
<tr>
<td>Reactive and refractory metals and alloys</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

### TABLE 3 Example Value Sets for Family Subclass Name for Aluminum, Copper, Steel, and Other Metals and Alloys

<table>
<thead>
<tr>
<th>Aluminum:</th>
<th>Copper:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Commercially pure aluminum</td>
<td>Copper</td>
</tr>
<tr>
<td>Aluminum-copper alloy</td>
<td>High copper alloy</td>
</tr>
<tr>
<td>Aluminum-manganese alloy</td>
<td>Beryllium copper</td>
</tr>
<tr>
<td>Aluminum-silicon alloy</td>
<td>Chromium copper</td>
</tr>
<tr>
<td>Aluminum-manganese-silicon alloy</td>
<td>Copper-zinc alloy (brass)</td>
</tr>
<tr>
<td>Aluminum-magnesium alloy</td>
<td>Copper-zinc-lead-alloy (leaded brass)</td>
</tr>
<tr>
<td>Steel:</td>
<td></td>
</tr>
<tr>
<td>Chromium-molybdenum</td>
<td>Copper-tin-lead-phosphorus alloy</td>
</tr>
<tr>
<td>Low carbon</td>
<td></td>
</tr>
<tr>
<td>High carbon</td>
<td></td>
</tr>
<tr>
<td>Austenitic</td>
<td></td>
</tr>
<tr>
<td>Ferritic</td>
<td></td>
</tr>
<tr>
<td>Martensitic</td>
<td></td>
</tr>
<tr>
<td>Precipitation hardening</td>
<td></td>
</tr>
</tbody>
</table>

### TABLE 4 Category Set for Designation Keyword

<table>
<thead>
<tr>
<th>Grade</th>
<th>Type</th>
<th>Composition</th>
<th>Temper</th>
<th>Condition</th>
<th>Class</th>
</tr>
</thead>
</table>

### TABLE 5 Value Set for Specimen Orientation

**Unnotched Specimen:**
- Longitudinal (parallel to working direction)
- Transverse (perpendicular to working direction)
- Long transverse
- Short transverse
- Tangential
- Radial
- Diagonal (to rolling direction)

**Cracked or Notched Specimen:**
See Terminology E 616 for orientation codes

### TABLE 6 Value Set for Location Within Product

<table>
<thead>
<tr>
<th>Outer surface</th>
<th>Internal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inside surface</td>
<td>Surface</td>
</tr>
<tr>
<td>Quarter thickness</td>
<td>Center of thickness</td>
</tr>
<tr>
<td>Leading edge</td>
<td>Trailing edge</td>
</tr>
</tbody>
</table>
### TABLE 7 Category Set for Yield Strength Method (as explained in Test Methods E 8 or E 8M)

<table>
<thead>
<tr>
<th>Offset</th>
<th>Extension under load</th>
<th>Upper</th>
<th>Lower</th>
</tr>
</thead>
</table>

### TABLE 8 Category Set for Hardness Scale

|     | Brinell | Knoop | Rockwell A | Rockwell B | Rockwell C | Rockwell E | Rockwell F | Shore | Vickers | Rockwell 15t | Rockwell 30t | Rockwell 45t | Rockwell 15N | Rockwell 30N | Rockwell 45N |
|-----|---------|-------|-------------|-------------|-------------|-------------|-------------|-------|---------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|

### TABLE 9 Value Set for Melt Practice

<table>
<thead>
<tr>
<th>Argon oxygen decarburization</th>
<th>Basic oxygen furnace</th>
<th>Open hearth</th>
<th>Electric furnace</th>
<th>Remelt</th>
<th>Ladle refining</th>
<th>Vacuum degassing</th>
<th>Vacuum arc remelt</th>
<th>Vacuum oxygen decarburization</th>
<th>Vacuum induction melting</th>
<th>Air induction melting</th>
<th>Electroslag remelt</th>
<th>Electroflux remelt</th>
<th>Electron beam melting</th>
<th>Reverberatory furnace</th>
</tr>
</thead>
</table>

### TABLE 10 Value Set for Cast Practice

<table>
<thead>
<tr>
<th>Continuous</th>
<th>Ingot</th>
<th>Powder metallurgy</th>
<th>Spin</th>
</tr>
</thead>
</table>

### TABLE 11 Value Set for Heating Environment

<table>
<thead>
<tr>
<th>Air</th>
<th>Vacuum</th>
<th>Inert gas</th>
<th>Hydrogen</th>
<th>Other reducing gas</th>
<th>Oxidizing gas atmosphere</th>
</tr>
</thead>
</table>
### TABLE 12 Value Set for Cooling Environment

<table>
<thead>
<tr>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quenched in oil</td>
</tr>
<tr>
<td>Air-cooled</td>
</tr>
<tr>
<td>Inert gas-cooled</td>
</tr>
<tr>
<td>Quenched in water</td>
</tr>
<tr>
<td>Quenched in brine</td>
</tr>
<tr>
<td>Quenched in polymer</td>
</tr>
<tr>
<td>Quenched in air and water</td>
</tr>
</tbody>
</table>

### TABLE 13 Value Set for Forming Method

<table>
<thead>
<tr>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Forging</td>
</tr>
<tr>
<td>Casting</td>
</tr>
<tr>
<td>Extrusion</td>
</tr>
<tr>
<td>Hot rolling</td>
</tr>
<tr>
<td>Cold rolling</td>
</tr>
<tr>
<td>Powder compaction</td>
</tr>
<tr>
<td>Drawing/coining</td>
</tr>
<tr>
<td>Bending</td>
</tr>
</tbody>
</table>

### TABLE 14 Value Set for Product Shape

<table>
<thead>
<tr>
<th>Shape</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flat</td>
</tr>
<tr>
<td>Round</td>
</tr>
<tr>
<td>Hexagonal</td>
</tr>
<tr>
<td>Square</td>
</tr>
<tr>
<td>Structural</td>
</tr>
<tr>
<td>Irregular</td>
</tr>
<tr>
<td>Profile</td>
</tr>
</tbody>
</table>
### TABLE 15 Value Set for Product Form

<table>
<thead>
<tr>
<th>Product Form</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bar</td>
<td>Rod</td>
</tr>
<tr>
<td>Block</td>
<td>Sheet</td>
</tr>
<tr>
<td>Pipe</td>
<td>Shift</td>
</tr>
<tr>
<td>Plate</td>
<td>Strip</td>
</tr>
<tr>
<td>Powder</td>
<td>Tube</td>
</tr>
<tr>
<td>Ring</td>
<td>Wire</td>
</tr>
<tr>
<td></td>
<td>Extruded profile</td>
</tr>
</tbody>
</table>

### TABLE 16 Value Set for Type of Elongation as Explained in Test Methods E 8 and E 8M

<table>
<thead>
<tr>
<th>Elongation Type</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>After fracture</td>
<td></td>
</tr>
<tr>
<td>At fracture</td>
<td></td>
</tr>
</tbody>
</table>

### TABLE 17 Category Set for Charpy Specimen Size

<table>
<thead>
<tr>
<th>Specimen Size</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Full</td>
</tr>
<tr>
<td></td>
<td>One half</td>
</tr>
<tr>
<td></td>
<td>One quarter</td>
</tr>
<tr>
<td></td>
<td>One eighth</td>
</tr>
</tbody>
</table>

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