

Designation: B 699 - 86 (Reapproved 2003)

# Standard Specification for Coatings of Cadmium Vacuum-Deposited on Iron and Steel<sup>1</sup>

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  $\mu$ m (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<sup>2</sup>
- B 183 Practice for Preparation of Low-Carbon Steel for Electroplating<sup>3</sup>
- B 201 Practice for Testing Chromate Coatings on Zinc and Cadmium Surfaces<sup>3</sup>
- B 242 Practice for Preparation of High-Carbon Steel for Electroplating<sup>3</sup>
- B 254 Practice for Preparation of and Electroplating on Stainless Steel<sup>3</sup>
- B 320 Practice for Preparation of Iron Castings for Electroplating<sup>3</sup>
- $^{\rm I}$  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.
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  - <sup>2</sup> Annual Book of ASTM Standards, Vol 03.02.
  - <sup>3</sup> Annual Book of ASTM Standards, Vol 02.05.

- B 322 Practice for Cleaning Metals Prior to Electroplating<sup>3</sup>
- B 440 Specification for Cadmium<sup>4</sup>
- B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section<sup>3</sup>
- B 499 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals<sup>3</sup>
- B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method<sup>3</sup>
- B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method<sup>3</sup>
- B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry<sup>3</sup>
- B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings<sup>3</sup>
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings<sup>3</sup>
- B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings<sup>3</sup>
- E 396 Test Methods for Chemical Analysis of Cadmium<sup>5</sup>
- F 519 Method for Mechanical Hydrogen Embrittlement of Plating Processes and Service Environments<sup>6</sup>

## 3. Classification

3.1 *Classes*—Vacuum-deposited cadmium coatings shall be classified on the basis of thickness, as follows:

| Class | Minimum Thickness, µm |
|-------|-----------------------|
| 12    | 12                    |
| 8     | 8                     |
| 5     | 5                     |

- 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

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vol 02.04.

<sup>&</sup>lt;sup>5</sup> Annual Book of ASTM Standards, Vol 03.05.

<sup>&</sup>lt;sup>6</sup> 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  $1.33 \times 10^{-4}$  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  $\mu$ m), for Class 8 it shall be Class 5 (5  $\mu$ m), and for Class 5 it shall be 4  $\mu$ m.
- 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 on 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 blisters, 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  $4 \times$  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.

#### **APPENDIXES**

(Nonmandatory Information)

#### 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.

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