Designation: B999 – 15

Standard Specification for
Titanium and Titanium Alloys Plating, Electrodeposited Coatings of Titanium and Titanium Alloys on Conductive and Non-Conductive Substrate[1]

This standard is issued under the fixed designation B999; 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 coatings of titanium and titanium-zirconium alloys on conductive and non-conductive substrates for engineering (functional) uses. The coatings of titanium-zirconium alloys are those that range in zirconium between 10wt% and 14wt% zirconium and are known as "terne" metallic electrodeposits.

1.2 This specification applies for both conductive (metallic) substrates and non-conductive (plastics, fibers, carbon foam, etc.)

1.3 Electrodeposits of titanium and titanium-zirconium alloys on aluminum and conductive substrate and nonconductive substrate are produced where it is desired to obtain atmospheric corrosion resistance. Deposits of titanium and titanium-zirconium alloys particularly on aluminum have shown to have excellent corrosion protective qualities in atmospheric exposure, especially when under-coated by electroless nickel. Titanium and titanium-zirconium alloy deposits provide corrosion protection from dilute sulfuric acid, are used for lining of brine refrigeration tanks, chemical equipment apparatus, storage batteries, and as a wear coating for bearing surfaces.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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:*[2]

B117 Practice for Operating Salt Spray (Fog) Apparatus

B183 Practice for Preparation of Low-Carbon Steel for Electroplating

B242 Guide for Preparation of High-Carbon Steel for Electroplating

B253 Guide for Preparation of Aluminum Alloys for Electroplating

B320 Practice for Preparation of Iron Castings for Electroplating

B322 Guide for Cleaning Metals Prior to Electroplating

B374 Terminology Relating to Electroplating

B487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of Cross Section

B499 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals

B504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method

B507 Practice for Design of Articles to Be Electroplated on Racks

B567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method

B568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry

B571 Practice for Qualitative Adhesion Testing of Metallic Coatings

B602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings

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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 B08.03 on Engineering Coatings.

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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.

B697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings

B727 Practice for Preparation of Plastics Materials for Electroplating

B733 Specification for Autocatalytic (Electroless) Nickel-Phosphorus Coatings on Metal

B849 Specification for Pre-Treatments of Iron or Steel for Reducing Risk of Hydrogen Embrittlement

B850 Guide for Post-Coating Treatments of Steel for Reducing the Risk of Hydrogen Embrittlement

B851 Specification for Automated Controlled Shot Peening of Metallic Articles Prior to Nickel, Autocatalytic Nickel, or Chromium Plating, or as Final Finish

D2982 Test Methods for Detecting Glycol-Base Antifreeze in Used Lubricating Oils

D3359 Test Methods for Measuring Adhesion by Tape Test

D5185 Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

E127 Practice for Fabrication and Control of Aluminum Alloy Ultrasonic Standard Reference Blocks

F519 Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments

3. Terminology

3.1 *Definitions*—Definitions of the terms used in this specification are in accordance with Terminology B374.

4. Classification

4.1 The coating classification system provides for a scheme to select a titanium or titanium alloy coating to meet specific performance requirements based on alloy composition and thickness.

4.1.1 "Type" describes the general composition of the deposit with respect to zirconium alloying content and is divided into two categories which establish deposit properties (see Table 1).

4.2 *Service Condition Based on Thickness:*

4.2.1 Service condition numbers are based on the severity of the exposure in which the coating is intended to perform and minimum coating thickness to provide satisfactory performance (see Table 2).

4.2.2 *SC0 Minimum Service, 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 environment.

TABLE 1 Deposit Compositions by Type

Type	Zirconium % wt
Type I	0 %
Type II	10 to 14%

TABLE 2 Service Conditions and Coating Thickness Requirements

Service Conditions	Minimum Coating Thickness Specification	Coating Thickness, µm	cm
SC0	Minimum Thickness	1.0	0.000102
SC1	Light Service	2.50	0.000254
SC2	Mild/Moderate Service	3.75	0.000381
SC3	Severe Service	5.0	0.000508

4.2.3 *SC1 Light Service, 2.5 µm*—This is defined by a minimum coating thickness of 2.5 µm for extending the life of the parts. Typical environments include light-load lubricated wear and indoor corrosion protection to prevent rusting.

4.2.4 *SC2 Mild/Moderate Service 3.75 µm*—This is defined by mild corrosion and wear environment and moderate environment such a non-marine outdoor exposure, alkali salts at elevated temperature, and moderate wear.

4.2.5 *SC3 Severe Service 5.0 µm*—This is defined by a very aggressive environment. Typical environments would include acid solution, elevated temperature and pressure hydrogen sulfide and carbon dioxide oil service, high temperature chloride systems, very severe wear, and marine immersion.

5. Ordering Information

5.1 The following information shall be supplied by the purchaser in either the purchase order or on the engineering drawing, or 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 and service condition.

5.1.3 Maximum dimension and tolerance requirements, if any.

5.1.4 Peening, if required.

5.1.5 The tensile strength of ferrous base materials in MPa.

5.1.6 Stress relief heat treat requirements before plating, if any.

5.1.7 Hydrogen embrittlement relief baking requirements after plating, if any.

5.1.8 Significant surfaces for plating as well as drawing indications of surfaces not to be plated.

5.1.9 Requirements for sampling.

6. Materials and Manufacture

6.1 *Substrate*—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 before plating. The producer shall not be responsible for coating defects resulting from surface conditions of the metal, if these conditions have been brought to the attention of the purchaser.

6.2 *Pretreatment*—A suitable method shall activate the base material surface and remove oxide and foreign materials, which may cause poor adhesion. See Practices B183, B242, B253, B320, B322, and B727.

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 (UTS) of 1000 MPa or greater, that have been machined, ground, cold formed, or cold straightened subsequent to heat treatment require stress relief heat treatment when specified by the purchaser. The tensile strength of the material shall be supplied by the purchaser. Specification B849 contains a list of pre-treatments, precautions, and caveats that shall be used.

6.3.2 *Peening*—Peening prior to plating may be required on high-strength steel parts to induce residual compressive stresses in the surface, which can reduce loss of fatigue strength and improve stress corrosion resistance after plating. Peening may be performed in accordance with Specification B851.

6.3.2.1 Steel parts which are designed for unlimited life under dynamic loads shall be shot peened.

6.3.2.2 Unless otherwise specified, the shot peening shall be accomplished on all surfaces for which the coating is required and all immediately adjacent surfaces when they contain notches, fillets, or other abrupt changes of section size where stresses will be concentrated.

6.4 *Racking*—Parts should be positioned so as to minimize trapping of hydrogen gas in cavities and holes, allowing the free circulation of solution over all surfaces to obtain uniform coating thickness. Anodes are to be placed in ways that do not result in direct shorts (arcing) or burning on the parts. Contact points used to supply power to the parts may not get fully covered in the plating process and shall be agreed upon between the producer and the purchaser. Further details on preferred racking methods can be found in Practice B507.

6.5 *Plating Process:*

6.5.1 To obtain consistent coating properties, the bath must be monitored periodically for temperature, pH, and metal constituent concentration.

6.5.1.1 Current densities are typically between three and five amps per square decimeter.

6.5.1.2 Anodes shall be made of Platinum, Pure or Titanium/Platinized.

6.5.1.3 The plating solution shall be mechanically agitated to maintain temperature uniformity and filtration is highly recommended to increase coating smoothness and uniformity.

6.5.2 *Electroless Nickle (EN) Strike:*

6.5.2.1 To improve bonding to the substrate, an electroless nickel strike layer may be employed prior to titanium plating. The EN strike layer may be as thick as 25 µm. EN strike layers shall be applied in accordance with B733.

6.5.2.2 EN strike is only needed prior to titanium plating when applied on non-conductive substrates.

6.6 *Hydrogen Embrittlement Relief:*

6.6.1 *Post-Coating Treatments for Iron and Steel for Reducing the Risk of Hydrogen Embrittlement*—Parts that are made of steel with UTS of 1000 MPa or greater as well as surface hardened parts, shall require post-coating hydrogen embrittlement relief baking when specified by the purchaser. The tensile strength shall be supplied by the purchaser. Specification B850 contains a list of post treatments, procedures, limitations, and guidelines that are permitted to be used to reduce the effects of hydrogen embrittlement.

6.6.2 Hydrogen embrittlement relief heat treatments shall be performed within 4 h of plating unless otherwise specified by the purchaser. It is most advantageous to begin embrittlement relief heat treatments as soon as practical.

7. Requirements

7.1 *Process*—Titanium and titanium-zirconium alloy coatings shall be produced by electrodeposition in aqueous solution of salts.

7.1.1 *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 1—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 racking, or both. The thickness requirements of this specification are minimum. The variation in the finish thickness from point to point on a coated article is inherent in electroplating. Therefore, the finish 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. In most cases, the average finish 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 B507) and the characteristics of the plating process. In addition, the average finish 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 finish thickness for the production lot as a whole will be greater than the average necessary to ensure that a single article meets the requirement.

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, dimples, or cracks, or combinations thereof.

7.2.2 *Thickness:* The thickness of the coating shall be equal to or exceed the minimum requirements provided in Table 2. Thickness requirements are based on the Service Condition agreed upon prior to plating or as flowed down to the processer by purchase order or blue print.

7.2.3 *Adhesion:* The coating shall exhibit no signs of adhesion failure from the basis material when subjected to adhesion testing as specified in 9.2.

7.3 *Qualification Requirements and Periodic Testing:* These requirements are placed on the coating and process and are performed on specimens to qualify initial process startup as well as continuous monitoring of the process to ensure the deposits meet the requisite requirements and properties for proper function.

7.3.1 *Composition:* Type II coating shall be tested for alloy composition by testing for zirconium. The wt. % zirconium shall be in the range designated in Table 1.

7.3.2 *Corrosion Resistance:* Titanium and titanium-zirconium coatings shall show neither corrosion products of titanium (or titanium-zirconium) nor basis metal corrosion products at the end of the test period, when tested by continuous exposure to salt spray in accordance with the method described in 9.4. 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. The minimum number of hours of exposure to salt spray testing required for each coating service condition is provided in Table 3.

NOTE 2—Corrosion is defined as the presence of more than 66 rust or white spots per square meter, or more than two rust or white spots in an area less than 3 square decimeters, or rust or white spots larger than 1.6 mm in diameter.

8. Sampling

8.1 *Selection*—Arandom sample of the size required by Test Method B602 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 or nonconforming to each requirement according to the criteria of the sampling plans in Test Method B602.

NOTE 3—Test Method B602 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 B602 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 B602 do not serve the needs, additional ones are given in Guide B697.

NOTE 4—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 re-coating. The purchaser needs to state whether the test is to be considered destructive or nondestructive.

NOTE 5—Definition of the 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.

8.2 *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 incudes chemical composition, grade, condition, and finish of surface prior to electroplating. Due to the impracticality of forging or casting separate test specimens, the separate specimens may also be cut from scrap castings when aluminum 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.

TABLE 3 Minimum Corrosion Resistance Requirements

Service Conditions	Thickness, µm	Min. Hours Exposure
SC0	1	500
SC1	2.5	750
SC2	3.75	980
SC3	5	1000

8.3 *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.

8.4 *Corrosion Resistance Specimens*—If separate specimens for corrosion resistance tests are required, they shall be panels of approximately 150 mm long, 100 mm wide, and approximately 1 mm thick (3 panels for a total surface area = 45,000 mm^2).

8.5 *Hydrogen Embrittlement Relief Specimens*—Specimens used for evaluating hydrogen embrittlement shall be of the notched type a1.a specimens per Test Method F519.

9. Test Methods

9.1 *Composition*—The titanium-zirconium alloy coating deposit may be weighed and then analyzed for one of the metals. Zirconium content may be determined and the titanium calculated by difference, for 1 g sample:

$$\text{Zirconium \%} = 1 - L/A \times 100 \tag{1}$$

$$\text{Mass \% Zirconium} = (W - L/W) \times 100 \tag{2}$$

where:

L = weight of titanium in sample,
A = weight of titanium-zirconium alloy in sample, and
W = 1 g.

9.1.1 The coating composition may be also analyzed by analytical techniques ICP per Test Methods D5185 and D2982, and EDM and AA according to Practice E127.

9.1.2 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.

9.2 *Thickness*—The thickness of the coating may be determined by any of the following Test Methods: B487, B499, B504, B567, and B568. 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.

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

9.3 *Adhesion*—Adhesion testing is performed according to method 3.2 of Practice B571 or Specification D3359.

9.4 *Corrosion Resistance*—When specified, 7.3.2 determines the corrosion resistance requirements in accordance with Test Method B117. Subsection 8.4 provides the number of specimens and the specimen dimensions required to perform the test.

9.5 *Hydrogen Embrittlement Relief*—When specified in the contract or purchase order, the process shall show no sign of hydrogen embrittlement when specimens (8.5) are plated, baked and then tested per Test Method F519.

9.6 *Visual Examination*—The coating shall have a uniform, metallic appearance without visible defects such as blisters, pits, cracks or evidence of improper plating.

10. Rejection and Rehearing

10.1 Articles that fail to conform to the requirements of this specification may be rejected. Material that fails acceptance requirements may be stripped and replated. Material that fails periodic tests may be re-tested once. If upon re-test a second failure occurs, the failure shall be reported to the producer or supplier for disposition of the hardware in question. In case of dissatisfaction with the results of a test, the producer or supplier may make a claim for a hearing.

11. Responsibility for Inspection

11.1 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.

12. Special Government Requirements

12.1 The following special Government Requirements 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.

13. Keywords

13.1 conductive substrate; electrodeposited coatings; nonconductive substrate; titanium alloys; titanium-zirconium alloys

APPENDIX

(Nonmandatory Information)

X1. COATINGS CHARACTERISTICS

X1.1 Coating Characteristics

TABLE X1.1 Coating Characteristics

	Pure Titanium	Titanium-Zirconium Alloy
Grain Structure	5000-50 000 nm	6000-60 000 nm
Uniformity	The surface coating shall have a uniform, metallic appearance without visible defects such as blisters, pits, cracks etc.	The surface coating shall have a uniform, metallic appearance without visible defects such as blisters, pits, cracks etc.
Melting Point	273.15 ± 5 °C	265 ± 5 °C
Physical Properties	1. Brinell hardness: 716 MN m^{-2} 2. Vickers hardness: 970 MN 3. Coefficient of linear thermal expansion: 8.6 x 10^{-6} K^{-1} 4. Conductivity: 21.9 W $^{m-1}$ K^{-1}	1. Brinell hardness: 718 MN m^{-2} 2. Vickers hardness: 980 MN 3. Coefficient of linear thermal expansion: 8.7 x 10^{-6} K^{-1} 4. Conductivity: 22.9 W m^{-1} K^{-1}

X1.2 Performance Characteristics

TABLE X1.2 Performance Characteristics

	Pure Titanium	Titanium-Zirconium Alloy
Thickness	max: 5 microns	max: 5 microns
	min: 500 h	min: 600 h
Corrosion Resistance	max: 2000 h excellent corrosion resistance	max: 2000 h excellent corrosion resistance
Adhesion	excellent	excellent
Flexural Stress	max: 175 Mpa	max: 200 Mpa
Solderability and Weldability	excellent formability	excellent formability
Conductivity	conductivity = 2.0 to 3.0 mOhms/m before SS, and 5.0 mOhms/m after SS test	conductivity = 2.5.0 to 3.5 mOhms/m before SS, and 5.5 mOhms/m after SS test

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