



Designation: B994/B994M – 15

## Standard Specification for Nickel-Cobalt Alloy Coating<sup>1</sup>

This standard is issued under the fixed designation B994/B994M; 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 describes the requirements for corrosion-resistant coatings of electrodeposited nickel-cobalt on metallic substrates and electrodeposited nickel-cobalt used for electroforming.

NOTE 1—The nickel-cobalt alloy is principally deposited as a coating on steel products. It can also be electrodeposited on iron, stainless steel, aluminum, titanium and any other metal substrate.

NOTE 2—The nickel-cobalt alloy coating has a low coefficient of friction of 0.08 that provides a dry lubricant on part surfaces that are in contact with each other and are subject to galling.

1.2 This specification incorporates an accelerated exposure test method to evaluate the effects of corrosion and galling on the coating, and incorporates a means of reporting the results to the purchaser.

1.3 The specification incorporates a classification scheme that establishes service conditions for thickness, classes of deposits based on the level of monitoring, and type based on supplemental coatings used after deposition.

1.4 The coating thickness ranges from 5 to >30  $\mu\text{m}$ , and it can be applied to machined parts, springs, latches, threaded parts, fasteners, etc. The deposit can also be used to electroform parts requiring high strength with the alloy being maintained at 50% nickel-cobalt.

1.5 The nickel-cobalt alloy is used to protect ferrous metals in contact with corrosive environments such as: oil and gas production facilities, costal marine, and ACQ (Alkaline Copper Quaternary) treatments for wood treatments.

1.6 The values stated 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.

1.7 *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*

*appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.8 *This standard has been revised to comply with the Restriction of Hazardous Substances **RoHS Requirements** that seek to limit the exposure of workers and the public to toxic metals. The nickel-cobalt alloy does not contain any of the six Restricted Hazardous Substances.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

A193/A193M Specification for Alloy-Steel and Stainless Steel Bolting for High Temperature or High Pressure Service and Other Special Purpose Applications

A194/A194M Specification for Carbon and Alloy Steel Nuts for Bolts for High Pressure or High Temperature Service, or Both

A681 Specification for Tool Steels Alloy

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

B254 Practice for Preparation of and Electroplating on Stainless Steel

B320 Practice for Preparation of Iron Castings for Electroplating

B322 Guide for Cleaning Metals Prior to Electroplating

B368 Test Method for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test)

B374 Terminology Relating to Electroplating

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

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

B762 Test Method of Variables Sampling of Metallic and Inorganic Coatings

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

- 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
- D1193** Specification for Reagent Water
- E2465** Test Method for Analysis of Ni-Base Alloys by Wavelength Dispersive X-Ray Fluorescence Spectrometry
- F519** Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments
- F1470** Practice for Fastener Sampling for Specified Mechanical Properties and Performance Inspection
- G59** Test Method for Conducting Potentiodynamic Polarization Resistance Measurements
- G85** Practice for Modified Salt Spray (Fog) Testing
- G102** Practice for Calculation of Corrosion Rates and Related Information from Electrochemical Measurements
- G193** Terminology and Acronyms Relating to Corrosion
- 2.2 *ISO Standard*.<sup>3</sup>
- ISO/IEC 17025:2005** General Requirements for the Competence of Testing and Calibration Laboratories

### 3. Terminology

3.1 *Definitions*—Definitions of terms used in this specification are in accordance with Terminologies **B374** and **G193**.

### 4. Classification

4.1 The coatings of nickel-cobalt have been classified as follows: service condition by thickness, class by test methods required, and type by secondary treatments.

4.2 *Service Condition (SC)*—This number signifies the thickness requirement based on the type of environment and desired mechanical properties.

NOTE 3—The 50% NiCo alloy produces a high strength alloy that is used in electroforming to manufacture parts. These deposits can be used at various thicknesses ranging from 1  $\mu\text{m}$  to hundreds of microns.

4.2.1 SC5 signifies that the application is slightly corrosive and that a minimum 5  $\mu\text{m}$  of coating is sufficient for protection and to provide the mechanical properties.

4.2.2 SC12 signifies that the application is mildly corrosive and that a minimum 12  $\mu\text{m}$  of coating is sufficient to protect against corrosion and provide the mechanical properties.

4.2.3 SC18 signifies that the application is corrosive and that a minimum of 18  $\mu\text{m}$  of coating is sufficient to protect against corrosion and provide the mechanical properties.

4.2.4 SC25 signifies that the application is corrosive and that a minimum 25  $\mu\text{m}$  of coating is sufficient to protect against corrosion and provide the mechanical properties.

4.3 *Class*—This number signifies the type and frequency of testing and monitoring programs that are used to control the process.

4.3.1 *Class 1*—Incorporates these standard tests: Galling (see **Appendix X1**), Composition (see **9.3**), Test Methods **F519** and **B368**, and Practices **B117** and **G85**.

4.3.2 *Class 2*—Incorporates these standard tests: Composition (see **9.3**), Test Methods **F519** and **B368**, and Practices **B117** and **G85**.

4.3.3 *Class 3*—Incorporates these standard tests: Composition (see **9.3**) and Practice **B117**.

4.3.4 *Class 4*—Incorporates the standard test of composition (see **9.3**).

4.4 *Type*—This number signifies the type of secondary treatment applied after the coating to modify the surface properties.

NOTE 4—The type classification is used to identify supplemental coating that enhances the performance in various applications. For qualification test purposes, if a supplemental coating is not used, that is Type 0. Type 1 supplemental coatings are used to lock the threads or metal-to-metal contact points to increase the amount of energy needed to disassemble the junction. Type 2 supplemental coatings are used to reduce the friction and aid in disassembly. Materials like gold, thallium, and PTFE can be used to provide lubricity and reduce the break torque.

4.4.1 Type 0 coatings are not treated with any supplemental coatings.

4.4.2 Type 1 coatings are treated with a material to secure the coating to itself.

4.4.3 Type 2 coatings are treated with a material to reduce the friction between coated surfaces.

### 5. Ordering Information

5.1 The purchaser shall specify the following when ordering this coating:

5.1.1 The specification number and revision year,

5.1.2 The service condition, class, and type,

5.1.3 The significant surface on the part as identified on a drawing,

5.1.4 The base metal alloy designation under ASTM standard or equivalent,

5.1.5 The tensile strength of the base metal in MPa,

5.1.6 Any supplemental requirements including Type 1 and Type 2 secondary treatments,

5.1.7 Any exception to stress relief prior to coating (see section **7.3**),

5.1.8 Any exceptions to post anneal heat treatments (see section **7.4**),

5.1.9 Any exceptions to tests specified in the acceptance tests (see section **8**), and

5.1.10 Any exceptions to tests specified in the qualifications tests (see section **9**).

5.1.11 Any special requirements of shot peening or location of rack or wire in the coating process, or both, should be specified by the purchaser. Otherwise refer to **7.2.3** and **7.3**.

### 6. Coating Requirements

6.1 The coatings shall be nickel-cobalt alloy produced by electrodeposition.

6.2 The nickel shall be in the range of 43 to 61% and the cobalt shall be in the range of 39 to 57%.

6.3 This coating shall be applied to the significant surface to the desired minimum thickness of the service condition.

6.4 The coating shall meet all applicable requirements as established in the purchase order in accordance with Section **5**.

<sup>3</sup> Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, <http://www.iso.org>.

## 7. Materials and Manufacture

7.1 *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 coatings defects resulting from surface conditions of the metal, if these conditions have been brought to the attention of the purchaser.

7.2 The substrate may be subjected to such polishing or buffing operations as are necessary to yield deposits with the desired final performance and appearance.

7.2.1 *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 strength and improve stress corrosion resistance after plating.

7.2.2 Steel parts that are designed for unlimited life under dynamic loads shall be shot peened or rotary flap peened.

NOTE 5—Controlled shot peening is the preferred method because there are geometries where rotary flap peening is not effective.

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

7.3 *Racking*—Parts should be positioned so as to minimize trapping of hydrogen gas in cavities and holes, allowing free circulation of solution over all surfaces to obtain uniform coating thickness. The location of rack or wire marks in the coating shall be agreed upon between the producer and purchaser.

7.4 *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 standards be used: Practices B183, B254, and B320, and Guides B242 and B322.

7.5 *Pretreatment*—A suitable method shall activate the surface and remove oxide and foreign materials, which may cause poor adhesion and coating porosity.

7.6 *Pretreatment of Iron or Steel for the Purpose of Reducing the Risk of Hydrogen Embrittlement*—All steel parts having an ultimate tensile strength greater than 1000 MPa (31 HRC) and that have been machined, ground, cold formed, or cold straightened, shall be heat treated for stress relief to reduce the risk of hydrogen embrittlement in the part before clean and electroplate processes. If these heat treatments are not required, the purchaser shall specify in the ordering information SR-0. If the purchaser does not specify an SR-0 exception to heat treatment, then the plater shall use Table 1 in Specification B849 to determine the appropriate heat treatment for the steel based on its tensile strength.

7.7 *Post-Coating Treatments of Iron and Steel for the Purpose of Reducing the Risk of Hydrogen Embrittlement*—All

electroplated steel parts having a tensile strength greater than 1000 MPa (31 HRC) as well as surface-hardened parts, shall be baked to reduce the risk of hydrogen embrittlement. If these heat treatments are not required, the purchaser shall specify in the ordering information ER-0. If the purchaser does not specify an ER-0 exception to heat treatment, then the producer shall use Guide B850 to determine the appropriate heat treatment for the steel based on its tensile strength.

## 8. Acceptance Tests

8.1 These tests are performed on the article that has been coated and can be performed by the producer prior to delivery and by the purchaser upon delivery.

8.2 *Appearance*—The surface of the article shall be uniform in appearance, 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.

8.3 *Adhesion*—The adhesion of the coating shall be such that when examined by one of the test methods described in Practice B571 it shall not show separation from the basis metal.

8.4 *Coating Thickness*—Coating thickness shall meet the minimum as specified in the purchase order by service condition. Use an appropriate method for the part. Test Method B568 provides a rapid measurement of the mass per unit area of the deposit.

## 9. Qualification Tests

9.1 These tests are performed on the process to ensure the system is capable of meeting the coating requirements by class. These tests shall be performed on SC25, Type 0 coupons at a frequency established in subsection 9.8 based on the class.

9.2 *Galling Test*—The producer shall perform the galling test in accordance with the procedures established in Appendix X1. The producer shall prepare the finishing lot galling report and shall demonstrate that loaded specimens will pass 608 days of evaluation with the torque ration of less than 2.5.

9.3 *Chemical Composition*—The nickel-cobalt alloy composition shall be measured quarterly or more frequently as specified using Test Method E2465 or other elemental analysis methods that can be demonstrated to meet precision and bias requirements of better than 1% mass weight.

9.4 *Hydrogen Embrittlement Test*—The producer shall perform quarterly, or more frequently, the procedure outlined in Test Method F519 by processing a set of four standard 1a specimens. They shall pass Test Method F519 criteria of no failure after 200 hours at 75% UTS.

9.5 Corrosion Testing:

9.5.1 *Electrochemical Corrosion Rate*—The nickel-cobalt alloy coated article shall have a corrosion rate of less than 0.8636  $\mu\text{m}/\text{y}$  when tested under Test Method G59. Corrosion rate shall be calculated in accordance with Practice G102. The test solution shall be prepared by dissolving  $5 \pm 1$  parts by mass of sodium chloride in 95 parts of water conforming to Type IV water in Specification D1193 (except that for this practice, limits for chlorides and sodium may be ignored). Careful attention should be given to the chemical content of the

salt. The salt used shall be sodium chloride with not more than 0.3 % by mass of total impurities. Halides (bromide, fluoride, and iodide) other than chloride shall constitute less than 0.1 % by mass of the salt content. Copper content shall be less than 0.3 ppm by mass. Sodium chloride that has had anti-caking agents added shall not be used because such agents may act as corrosion inhibitors. The level of pH shall be equal to 6.5 to 7.2, temperature  $25 \pm 3^\circ \text{C}$ , with atmospheric pressure and static conditions. The frequency of this shall be quarterly unless the purchaser specifies an increased frequency.

#### 9.6 Environmental Testing

9.6.1 *CASS Test*—The producer shall evaluate the process corrosion protection by performing Test Method **B368** on test coupons quarterly or more frequently as required. Panels processed to SC25 shall not show any red rust before 120 hours of exposure

9.6.2 *Salt Spray (Fog) Test*—The producer shall evaluate the process corrosion protection of the nickel-cobalt alloy by Practice **B117** quarterly or more frequently as specified. Panels processed to SC25 shall pass with no red rust before 500 hours of exposure to salt spray (fog) as outlined in Practice **B117**.

9.6.3 *Modified Salt Spray (Fog) Test*—The producer shall evaluate the process corrosion protection of the nickel-cobalt alloy by Test Method **G85** (A2 cyclic acidified salt fog testing) quarterly or more frequently as specified. Coupons processed to SC25 shall pass with no red rust before 200 hours of exposure to the Test Method **G85** testing.

9.7 *Third Party Reports*—If a third party is used to perform these qualification tests, the test report shall include: Testing laboratory name, name and signature of person responsible for the test, location, list of equipment, current calibration certificates, and required permits for operating equipment (x-ray).

9.8 *Frequency of Qualification Tests*—The maintenance of a process requires frequent testing to ensure the quality is maintained and to instruct the operators in good practice to ensure good workmanship at all times. **Table 1** shall be used to establish the maximum frequency for qualification testing of the process by class.

### 10. Sampling

10.1 The purchaser and producer are urged to employ statistical process control in the coating process. Properly performed, statistical process control will ensure coated products of satisfactory quality and will ensure the amount of acceptance inspection.

10.2 When a collection of coated articles are 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 **B602**, Guide **B697**, and Test Method **B762** contain sampling plans that are designed for sampling inspection of coatings.

10.3 Test Method **B602** contains four sampling plans, three for use with tests that are nondestructive and one when they are destructive. Test Method **B602** provides a default plan if one is not specified.

10.4 Guide **B697** provides a large number of plans and also gives guidance in the selection of a plan. Guide **B697** provides a default plan if one is not specified.

10.5 Test Method **B762** 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 **B762** contains several plans and also gives instructions for calculating plans to meet special needs. Test Method **B762** provides a default plan if one is not specified.

10.6 Practice **F1470** may be used for threaded articles such as internally threaded, externally threaded, and non-threaded fasteners and washers. This guide provides for two plans: one designated the “detection process” and one designated the “prevention process.”

10.7 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.

### 11. Rejection

11.1 The purchaser can randomly test any article in accordance with the material testing report in **12.3**. Failure to pass any test is cause of rejection of the purchase order.

### 12. Certification

12.1 *Acceptance Test*—Upon passing of acceptance tests, the producer shall specify that the plated articles are in compliance with the acceptance requirements in the test report.

12.2 *Qualification Tests*—Upon passing of the qualification tests, the producer shall specify that the plated articles were processed in a qualified process incorporating the most recent results in the test report.

**TABLE 1**

	Galling	Composition	Hydrogen Embrittlement	Corrosion	Test Method <b>B368</b>	Practice <b>B117</b>	Test Method <b>G85</b>
Class 1	120 days	30 days	90 days	90 days	90 days	30 days	90 days
Class 2	None	90 days	90 days	90 days	90 days	90 days	90 days
Class 3	None	90 days	None	None	None	30 days	None
Class 4	None	30 days	None	None	None	None	None