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# Standard Specification for Autocatalytic (Electroless) Nickel-Phosphorus Coatings on Metal<sup>1</sup>

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

## 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)<sup>2</sup> and Refs (2) (3) (4) and (5).

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, magnetism, electrical conductivity provide diffusion barrier, and solderability. They are also used for the salvage of worn or mismachined parts.

1.6 The low phosphorus (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*:<sup>3</sup>

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

**B374** Terminology Relating to Electroplating

**B380** Test Method for Corrosion Testing of Decorative Electrodeposited Coatings by the Corrodokote Procedure

**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

<sup>1</sup> This specification 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> The boldface numbers given in parentheses refer to a list of references at the end of the text.

<sup>3</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.

**B537** Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure

**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

**B578** Test Method for Microhardness of Electroplated Coatings

**B602** Test Method for Attribute Sampling of Metallic and Inorganic Coatings

**B667** Practice for Construction and Use of a Probe for Measuring Electrical Contact Resistance

**B678** Test Method for Solderability of Metallic-Coated Products

**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

**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

**D1193** Specification for Reagent Water

**D2670** Test Method for Measuring Wear Properties of Fluid Lubricants (Falex Pin and Vee Block Method)

**D2714** Test Method for Calibration and Operation of the Falex Block-on-Ring Friction and Wear Testing Machine

**D3951** Practice for Commercial Packaging

**D4060** Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser

**E60** Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry

**E140** Hardness Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Superficial Hardness, Knoop Hardness, Scleroscope Hardness, and Leeb Hardness

**E156** Test Method for Determination of Phosphorus in High-Phosphorus Brazing Alloys (Photometric Method) (Withdrawn 1993)<sup>4</sup>

**E352** Test Methods for Chemical Analysis of Tool Steels and Other Similar Medium- and High-Alloy Steels

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

**G5** Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements

**G31** Guide for Laboratory Immersion Corrosion Testing of Metals

**G59** Test Method for Conducting Potentiodynamic Polarization Resistance Measurements

**G85** Practice for Modified Salt Spray (Fog) Testing

2.2 *Military Standards:*

**MIL-R-81841** Rotary Flap Peening of Metal Parts<sup>5</sup>

**MIL-S-13165** Shot Peening of Metal Parts<sup>5</sup>

**MIL-STD-105** Sampling Procedures and Tables for Inspection by Attribute<sup>5</sup>

2.3 *ISO Standards:*

**ISO 4527** Autocatalytic Nickel-Phosphorus Coatings—Specification and Test Methods<sup>6</sup>

### 3. Terminology

3.1 *Definitions:*

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.

3.2 *Other Definitions*—Terminology **B374** defines most of the technical terms used in this specification.

### 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 V**, see **Appendix X5**, Alloy **TYPEs**.

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**, 0.1  $\mu\text{m}$ —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.

<sup>5</sup> Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

<sup>6</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

**TABLE 1 Deposit Alloy Types**

Type	Phosphorus % wt
I	No Requirement for Phosphorus
II	1 to 3
III	2 to 4
IV	5 to 9
V	10 and above

<sup>4</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

**TABLE 2 Service Conditions  
Coating Thickness Requirements**

Service Condition	Minimum Coating Thickness Specification	μm	in.
SC0	Minimum Thickness	0.1	0.000004
SC1	Light Service	5	0.0002
SC2	Mild Service	13	0.0005
SC3	Moderate Service	25	0.001
SC4	Severe Service	75	0.003

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 increase 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).

**TABLE 3 Classification of Post Heat Treatment**

CLASS	Description	Temperature (°C)	Time (h)
1	No Heat Treatment, As Plated		
2	Heat Treatment for Maximum Hardness		
	TYPE I	260	20
		285	16
		320	8
		400	1
	TYPE II	350 to 380	1
	TYPE III	360 to 390	1
	TYPE IV	365 to 400	1
	TYPE V	375 to 400	1
3	Adhesion on Steel	180 to 200	2 to 4
4	Adhesion, Carburized Steel and Age Hardened Aluminum	120 to 130	1 to 6
5	Adhesion on Beryllium and Aluminum	140 to 150	1 to 2
6	Adhesion on Titanium	300–320	1–4

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.

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 The tensile strength of the material in MPa (see 6.3.1 and 6.6).

5.1.6 Stress relief heat treatment before plating, (see 6.3).

5.1.7 Hydrogen Embrittlement Relief after plating, (see 6.6).

5.1.8 Significant surfaces and surfaces not to be plated must be indicated on drawings or sample.

5.1.9 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.

5.1.10 Requirement for a vacuum, inert or reducing atmosphere for heat treatment above 260°C to prevent surface oxidation of the coating (see S3).

5.1.11 Test methods for coating adhesion, composition, thickness, porosity, wear and corrosion resistance, if required, selected from those found in Section 9 and Supplemental Requirements.

5.1.12 Requirements for sampling (see Section 8).

NOTE 4—The purchaser should 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. 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 coatings 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 surface and remove oxide and foreign materials, which may cause poor adhesion and coating porosity.

NOTE 5—Heat treatment of the base material may effect its metallurgical properties. An example is leaded steel which may exhibit liquid or solid embrittlement after heat treatment. Careful selection of the pre and post heat treatments are recommended.

### 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), 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, procedures, 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. (See Supplementary Requirements).

**6.3.3 Steel parts which are designed for unlimited life under dynamic loads shall be shot peened or rotary flap peened.**

NOTE 6—Controlled shot peening is the preferred method because there are geometry's where rotary flap peening is not effective. See S11.2.

**6.3.3.1** 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.

**6.4 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.

### 6.5 Plating Process:

**6.5.1** To obtain consistent coating properties, the bath must be monitored periodically for pH, temperature, nickel and hypophosphite. Replenishments to the plating solution should be as frequent as required to maintain the concentration of the nickel and hypophosphite between 90 and 100 % of set point. The use of a statistical regimen to establish the control limits and frequency of analysis may be employed to ensure quality deposits are produced.

**6.5.2** Mechanical movement of parts, agitation of the bath, and filtration is recommended to increase coating smoothness and uniformity and prevent pitting or streaking due to hydrogen bubbles.

**6.6 Post Coating Treatment for Iron and Steel for Reducing the Risk of Hydrogen Embrittlement**—Parts that are made of steel with ultimate tensile strengths of 1000 MPa (corresponding hardness values 300 HV10, 303 HB, or 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 shall be supplied by the

purchaser. Guide B850 contains a list of post treatments, procedures, limitations, and guidelines that are permitted to be used to reduce the effects of hydrogen embrittlement.

**6.6.1** Heat treatment shall be performed preferably within 1 h but not more than 3 h of plating unless the size or weight of the part prevents the initiation of heat treatment within 3 h. In this case, the part shall be heat treated as soon as possible. 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.

**6.7 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 Appendix X3 and Appendix X5.

**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.

NOTE 8—Inert or reducing atmosphere or vacuum sufficient to prevent oxidation is recommended for heat treatment above 260°C. Do not use gas containing hydrogen with high-strength steel parts.

## 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 5.1.10).

**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 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.