



Standard Specification for Autocatalytic (Electroless) Nickel-Phosphorus Coatings on Metal¹

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This standard has been approved for use by agencies of the 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)² 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:³

- B 368 Test Method for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Testing)
- B 374 Terminology Relating to Electroplating
- B 380 Test Method of Corrosion Testing of Decorative Electrodeposited Coatings by the Corrodokote Procedure
- B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section
- B 499 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals
- B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method
- B 537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure

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² The boldface numbers given in parentheses refer to a list of references at the end of the text.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

- B 567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method
- B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
- B 571 Practice for Qualitative Adhesion Testing of Metallic Coatings
- B 578 Test Method for Microhardness of Electroplated Coatings
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coating
- B 667 Practice for Construction and Use of a Probe for Measuring Electrical Contact Resistance
- B 678 Test Method for Solderability of Metallic-Coated Products
- B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
- B 762 Test Method of Variables Sampling of Metallic and Inorganic Coatings
- B 849 Specification for Pre-Treatments of Iron or Steel for Reducing Risk of Hydrogen Embrittlement
- B 850 Guide for Post-Coating Treatments of Steel for Reducing the Risk of Hydrogen Embrittlement
- B 851 Specification for Automated Controlled Shot Peening of Metallic Articles Prior to Nickel, Autocatalytic Nickel, Chromium, or As A Final Finish
- D 1193 Specification for Reagent Water
- D 2670 Test Method for Measuring Wear Properties of Fluid Lubricants (Falex Pin and Vee Block Method)
- D 2714 Test Method for Calibration and Operation of the Falex Block-on-Ring Friction and Wear Testing Machine
- D 3951 Practice for Commercial Packaging
- D 4060 Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser
- E 60 Practice for Analysis of Metals, Ores, and Related Materials by Molecular Absorption Spectrometry
- E 140 Hardness Conversion Tables for Metals Relationship Among Brinell Hardness, Vickers Hardness, Rockwell Hardness, Superficial Hardness, Knoop Hardness, and Scleroscope Hardness
- E 156 Test Method for Determination of Phosphorus in High-Phosphorus Brazing Alloys (Photometric Method)⁴
- E 352 Test Methods for Chemical Analysis of Tool Steels and Other Similar Medium-and High-Alloy Steel
- F 519 Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments
- G 5 Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements
- G 31 Practice for Laboratory Immersion Corrosion Testing of Metals
- G 59 Practice for Conducting Potentiodynamic Polarization Resistance Measurements
- G 85 Practice for Modified Salt Spray (Fog) Testing
- 2.2 *Military Standards:*

⁴ Withdrawn.

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

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

MIL-R-81841 Rotary Flap Peening of Metal Parts⁵

MIL-S-13165 Shot Peening of Metal Parts⁵

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attribute⁵

2.3 *ISO Standards:*

ISO 4527 Autocatalytic Nickel-Phosphorus Coatings—Specification and Test Methods⁶

3. Terminology

3.1 *Definition:*

3.1.1 *significant surfaces*—those substrate surfaces which the coating must protect from corrosion or wear, or both, and that are essential to the performance.

3.2 *Other Definitions*—Terminology B 374 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).

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

⁶ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

4.2.2 *SC0 Minimum Service*, 0.1 μm —This is defined by a minimum coating thickness to provide specific material properties and extend the life of a part or its function. Applications include requirements for diffusion barrier, undercoat, electrical conductivity and wear and corrosion protection in specialized environments.

4.2.3 *SC1 Light Service*, 5 μm —This is defined by a minimum coating thickness of 5 μm for extending the life of the part. Typical environments include light-load lubricated wear, indoor corrosion protection to prevent rusting, and for soldering and mild abrasive wear.

4.2.4 *SC2 Mild Service*, 13 μm —This is defined by mild corrosion and wear environments. It is characterized by industrial atmosphere exposure on steel substrates in dry or oiled environments.

4.2.5 *SC3 Moderate Service*, 25 μm —This is defined by moderate environments such as non marine outdoor exposure, alkali salts at elevated temperature, and moderate wear.

4.2.6 *SC4 Severe Service*, 75 μm —This is defined by a very aggressive environment. Typical environments would include acid solutions, elevated temperature and pressure, hydrogen sulfide and carbon dioxide oil service, high-temperature chloride systems, very severe wear, and marine immersion.

NOTE 2—The performance of the autocatalytic nickel coating depends to a large extent on the surface finish of the article to be plated and how it was pretreated. Rough, non uniform surfaces require thicker coatings than smooth surfaces to achieve maximum corrosion resistance and minimum porosity.

4.3 *Post Heat Treatment Class*—The nickel-phosphorus coatings shall be classified by heat treatment after plating to 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).

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.

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
		350 to 380	1
	TYPE II	360 to 390	1
	TYPE III	365 to 400	1
	TYPE IV	375 to 400	1
	TYPE V	180 to 200	2 to 4
	Adhesion on Steel	120 to 130	1 to 6
3	Adhesion, Carburized Steel and Age Hardened Aluminum	140 to 150	1 to 2
4	Adhesion on Beryllium and Aluminum	300–320	1–4
5	Adhesion on Titanium		

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 B 849 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 B 850 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.

7.2.3 *Adhesion*—The coating shall have sufficient adhesion to the basis metal to pass the specified adhesion test (see 9.4 and Practice B 571).

7.2.4 *Porosity*—The coatings shall be essentially pore free when tested according to one of the methods of 9.6. The test

method, the duration of the test, and number of allowable spots per unit area shall be specified (see 5.1.11 and 9.6).

7.3 Qualification Requirements—These requirements are placed on the deposit and process and are performed on specimens to qualify the deposit and plating process. The tests for these qualification requirements shall be performed monthly or more frequently.

7.3.1 Composition—Type II, III, IV, V deposits shall be analyzed for alloy composition by testing for phosphorus (see 9.1). The weight percent of phosphorus shall be in the range designated by type classification (see 4.1).

7.3.2 Microhardness—The microhardness of Class 2 deposits shall be determined by Test Method B 578 (Knoop). For Class 2 coatings, the microhardness shall equal or exceed a minimum of 850 (HK100 (or equivalent Vickers) (see 4.3 and 9.5). The conversion of Vickers to Knoop using Tables E 140 is not recommended.

7.3.3 Hydrogen Embrittlement—The process used to deposit a coating onto high strength steels shall be evaluated for hydrogen embrittlement by Test Method F 519.

8. Sampling

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

8.1.1 Sampling plans can only screen out unsatisfactory products without assurance that none of them will be accepted. (7)

8.2 The sampling plan used for the inspection of a quantity of coated parts (lot) shall be Test Method B 602 unless otherwise specified by purchaser in the purchase order or contract (see 5.1.12 and S.11.1).

NOTE 10—Usually, when a collection of coated parts (the inspection lot 8.2) is examined for compliance with the requirements placed on the parts a relatively small number of parts, the sample, is selected at random and inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection sample. The size of the sample and the criteria of compliance are determined by the application of statistics. The procedure is known as sampling inspection. Three standards Test Method B 602, Guide B 697, and Test Method B 762 contain sampling plans that are designed for the sampling inspection of coatings.

Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one for use with tests that are destructive. The purchaser and producer may agree on the plan(s) to be used. If they do not, Test Method B 602 identifies the plan to be used.

Guide B 697 provides a large number of plans and also gives guidance on the selection of a plan. When Guide B 697 is specified, the purchaser and producer need to agree on the plan to be used.

Test Method B 762 can be used only for coating requirements that have a numerical limit, such as coating thickness. The last must yield a numerical value and certain statistical requirements must be met. Test Method B 762 contains several plans and also gives instructions for calculating plans to meet special needs. The purchaser and producer may agree on the plan(s) to be used. If they do not, Test Method B 762 identifies the plan to be used.

An inspection lot shall be defined as a collection of coated parts which are of the same kind, that have been produced to the same specification, that have been coated by a single producer at one time or approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

8.3 All specimens used in the sampling plan for acceptance tests shall be made of the same basis material and in the same metallurgical condition as articles being plated to this specification.

8.4 All specimens shall be provided by the purchaser unless otherwise agreed to by the producer.

NOTE 11—The autocatalytic nickel process is dynamic and a daily sampling is recommended. For coatings requiring alloy analysis and corrosion testing weekly sampling should be considered as an option.

9. Test Methods

9.1 Deposit Analysis for Phosphorus:

9.1.1 Phosphorus Determination—Determine mass % phosphorus content according to Practice E 60, Test Methods E 352, or Test Method E 156 on known weight of deposit dissolved in warm concentrated nitric acid.

9.1.2 Composition can be determined by atomic absorption, emission or X-ray fluorescence spectrometry.

NOTE 12—Inductively coupled plasma techniques can determine the alloy to within 0.1 %. The following analysis wavelength lines have been used with minimum interference to determine the alloy.

Ni 216.10 nm	Cd 214.44 nm	Fe 238.20 nm
P 215.40 nm	Co 238.34 nm	Pb 283.30 nm
P 213.62 nm	Cr 284.32 nm	Sn 198.94 nm
Al 202.55 nm	Cu 324.75 nm	Zn 206.20 nm

9.2 Appearance—Examine the coating visually for compliance with the requirements of 7.2.1.

9.3 Thickness:

NOTE 13—Eddy-current type instruments give erratic measurements due to variations in conductivity of the coatings with changes in phosphorus content.

9.3.1 Microscopical Method—Measure the coating thickness of a cross section according to Test Method B 487.

NOTE 14—To protect the edges, electroplate the specimens with a minimum of 5 µm of nickel or copper prior to cross sectioning.

9.3.2 Magnetic Induction Instrument Method—Test Method B 499 is applicable to magnetic substrates plated with autocatalytic nickel deposits, that contain more than 11 mass % phosphorus (not ferromagnetic) and that have not been heat-treated. The instrument shall be calibrated with deposits plated in the same solution under the same conditions on magnetic steel.

9.3.3 Beta Backscatter Method—Test Method B 567 is only applicable to coatings on aluminum, beryllium, magnesium, and titanium. The instrument must be calibrated with standards having the same composition as the coating.

NOTE 15—The density of the coating varies with its mass % phosphorus content (See Appendix X2).

9.3.4 Micrometer Method—Measure the part, test coupon, or pin in a specific spot before and after plating using a suitable micrometer. Make sure that the surfaces measured are smooth, clean, and dry.

9.3.5 Weigh, Plate, Weigh Method—Using a similar substrate material of known surface area, weigh to the nearest milligram before and after plating making sure that the part or coupon is dry and at room temperature for each measurement.

Calculate the thickness from the increase in weight, specific gravity, and area as follows:

$$\text{coating thickness, } \mu\text{m} = 10 W / (A \times D) \quad (1)$$

where:

W = weight gain in milligrams,

A = total surface area in square centimetres, and

D = grams per cubic centimetres (see Appendix X2).

9.3.6 Coulometric Method—Measure the coating thickness in accordance with Test Method B 504. The solution to be used shall be in accordance with manufacturer's recommendations. The surface of the coating shall be cleaned prior to testing (see Note 14).

9.3.6.1 Calibrate standard thickness specimens with deposits plated in the same solution under the same conditions.

9.3.7 X-Ray Spectrometry—Measure the coating thickness in accordance with Test Method B 568. The instrument must be calibrated with standards having the same composition as the coating.

NOTE 16—This method is only recommended for deposits in the as-plated condition. The phosphorus content of the coating must be known to calculate the thickness of the deposit. Matrix effect due to the distribution of phosphorus in layers of the coating also effect the measurement accuracy and require that calibration standards be made under the same conditions as the production process.

9.4 Adhesion:

9.4.1 Bend Test (Practice B 571)—A sample specimen is bent 180° over a mandrel diameter 4× the thickness (10 mm minimum) of the specimen and examined at 4× power magnification for flaking or separation at the interface. Fine cracks in the coating on the tension side of the bend are not an indication of poor adhesion. Insertion of a sharp probe at the interface of the coating and basis metal to determine the adhesion is suggested.

NOTE 17—Appropriate test specimens are strips approximately 25 to 50 mm wide, 200 to 300 mm long and 3 to 6 mm thick.

9.4.2 Impact Test—A spring-loaded center punch with a point having 2 to 3 mm radius is used to test adhesion of the coating on nonsignificant surfaces of the plated part. Make three closely spaced indentations and examine under 10× magnification for flaking or blistering of the coating, which is cause for rejection.

9.4.3 Thermal Shock—The coated part is heated to 200°C in an oven and then quenched in room temperature water. The coating is examined for blistering or other evidence of poor adhesion at 4× magnification.

9.5 Microhardness—The microhardness of the coating can be measured by Test Method B 578 using Knoop indenter and is reported in Knoop Hardness Number (HK). It will vary depending on loads, type of indenter, and operator. A100 g load is recommended. The rhombic Knoop indenter gives higher hardness readings than the square-base pyramidal Vickers diamond indenter for 100 to 300 g loads, see Ref (6). For maximum accuracy, a minimum coating thickness of 75 μm is recommended. Conversions of Vickers or Knoop hardness number to Rockwell C is not recommended.

NOTE 18—On thick (75 μm +) coatings on steel a surface microhardness

determination is permissible.

9.6 Porosity—There is no universally accepted test for porosity. When required, one of the following tests can be used on the plated part or specimen.

9.6.1 Ferroxyl Test for Iron Base Substrates—Prepare the test solution by dissolving 25 g of potassium ferricyanide and 15 g of sodium chloride in 1 L of distilled water. After cleaning, immerse the part for 30 s in the test solution at 25°C. After rinsing and air drying, examine the part for blue spots, which form at pore sites.

9.6.2 Boiling Water Test for Iron-Base Substrates—Completely immerse the part to be treated in a vessel filled with aerated water at room temperature. Apply heat to the beaker at such a rate that the water begins to boil in not less than 15 min, nor more than 20 min after the initial application of heat. Continue to boil the water for 30 min. Then remove the part, air dry, and examine for rust spots, which indicate pores.

NOTE 19—Aerated water is prepared by bubbling clean compressed air through distilled water by means of a glass diffusion disk at room temperature for 12 h. The pH of the aerated water should be 6.7 + 0.5.

9.6.3 Aerated Water Test for Iron-Base Substrates—Immerse the part for 4 h in vigorously aerated Type IV or better water (see Specification D 1193) at 25 ± 2°C temperature and then examine the part for rust spots.

9.6.4 Alizarin Test for Aluminum Alloys—Wipe the plated part or specimen with 10 mass % sodium hydroxide solution. After 3 min contact, rinse, and apply a solution of alizarin sulfonate prepared by dissolving 1.5 g of methyl cellulose in 90 mL of boiling water to which, after cooling, 0.1 g sodium alizarin sulfonate, dissolved in 5 mL of ethanol is added. After 4 min contact, apply glacial acetic acid until the violet color disappears. Any red spots remaining indicate pores.

9.6.5 Porosity Test for Copper Substrates—Wipe the plated part or specimen with glacial acetic acid. After 3 min, apply a solution of potassium ferrocyanide prepared by dissolving 1 g of potassium ferrocyanide and 1.5 g methyl cellulose in 90 mL of boiling distilled water. The appearance of brown spots after 2 min indicate pores.

9.7 Other Test Methods—Test methods which have been developed that are equal to or better than these may be substituted. The precision and bias requirements will vary for each type of test. If an alternate test is specified it shall be agreed upon between the producer and the purchaser.

10. Rejection and Rehearing

10.1 Part(s) that fail to conform to the requirements of this standard may be rejected. Rejection shall be reported to the producer promptly in writing. In the case of dissatisfaction occurs with the results of a test, the producer may make a claim for a hearing. Coatings that show imperfections may be rejected.

11. Certification

11.1 When specified in the purchase order or contract, the purchaser shall be furnished certification that the samples representing each lot have been processed, tested and inspected as directed in this specification and the requirements have been

met. When specified in the purchase order or contract, a report of the test results shall be furnished.

12. Keywords

12.1 autocatalytic; chemical nickel; coatings; conductive; corrosion resistance; electroless; functional; nickel; nickel phosphorus; wear resistance

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirements shall apply only when specified by the purchaser in the contract or order.

S1.1 Shot Peening—When specified by the purchaser in the ordering information, the part(s) shall be shot peened prior to plating in accordance with Specification B 851 or MIL-S-13165.

S1.2 Composition—When specified by the purchaser in the ordering information the phosphorus content shall be maintained in the deposit to within 1 %. Use the test methods described in 9.1.

S1.3 Inert Atmosphere—When specified by the purchaser in the ordering information, the coating shall be heat treated in a vacuum, inert, or reducing atmosphere to prevent surface oxidation of the coating.

S1.4 Hydrogen Embrittlement—When specified by the purchaser in the ordering information the plating process shall be evaluated at the time of processing parts for hydrogen embrittlement using Test Method F 519.

S1.5 Abrasive Wear—When specified by the purchaser in the ordering information, the coating shall be tested for abrasion wear resistance using the method in Appendix X1 of this specification. The coating shall meet a maximum wear rate which is specified by the purchaser and agreed to by the producer.

S1.6 Adhesive Wear—When specified by the purchaser in the ordering information, the coating shall be tested for adhesive wear resistance using Test Method D 2714 or Test Method D 2670. The wear rate shall be specified by the purchaser and agreed to by the producer.

S1.7 Contact Resistance—When specified by the purchaser in the ordering information, the coating shall be tested for contact resistance using Practice B 667.

S1.8 Solderability—When specified by the purchaser in the ordering information, the unaged coating shall pass Test Method B 678 on solderability.

S1.9 Corrosion Resistance—When specified by the purchaser in the ordering information the coating shall pass any special corrosion tests agreed to by the producer. The corrosion resistance of the coating to a specific liquid medium can be determined by means of immersion tests (see Practice G 31) or electrochemical test (see Test Method G 5 and Practice G 59).

S1.10 Pitting Corrosion Resistance—Use Practice G 85 (acetic acid-salt spray test), Test Method B 368 (copper-accelerated acetic acid-salt spray, CASS), or Test Method B 380 (Corrodokote) to evaluate the corrosion resistance of the coating to pitting.

S1.11 Special Government Requirements:

S1.11.1 Sampling—Part(s) plated for the US Government and Military use shall use MIL-STD-105 as the sampling plan.

S1.11.2 Shot Peening—High strength steel part(s) processed for US Government and Military use shall be shot peened in accordance with MIL-S-13165 or rotary flap peened in accordance with MIL-R-81841. (see Note 6).

S1.11.3 Packaging—Parts shall be packaged in accordance with Practice D 3951.

APPENDIXES

(Nonmandatory Information)

X1. TABER ABRASER WEAR TEST METHOD

X1.1 Scope

X1.1.1 This test method will evaluate the resistance of the coating to abrasive wear. The test is performed by rotating a plated panel under rotating rubber wheels and weighing the panel after each 1000 cycles for weight loss. Duration of the test is 6000 cycles and it can be extended to 25 000 cycles for more complete results.

NOTE X1.1—Variation in results have been attributed to the humidity in the laboratory and the storage conditions of the CS-10 wheels. Care should be taken to control the humidity between tests.

X1.1.2 The results are variable between tests and therefore three plated test specimens should be tested to 6000 cycles each. The results should be averaged without the first 1000 cycles and the abrasion wear resistance is reported as the weight loss in mg/1000 cycles (Taber Wear Index).

X1.2 Apparatus

X1.2.1 *Taber Abraser Wear Testing Unit*—The unit must be capable of loading with 1000 g load and operating with a vacuum.

X1.2.2 *Abrasion Wheels*— Use CS-10 (resilient rubber) Taber wheels. To reface the wheels use CS-11 discs from Taber. The hardness of CS-10 wheels can change with time and can effect the reproducibility of results (see Test Method D 4060).

X1.2.3 *Test Specimens*— Test specimens shall be made from 20 gage CR steel 4 by 4 in. (100 by 100 by 1.3 mm) with a 0.250 (6.35 mm) hole in the center. Test specimens are available from Taber.

X1.2.4 *Analytical Balance*—Scale which is capable of measuring to 150 g $\pm$ 0.1 mg.

X1.3 Procedure

X1.3.1 Plate three specimens with 0.001 in. (25 μ m) of nickel phosphorus coating.

X1.3.2 Wear test the specimens. For each of the three specimens complete the following steps:

X1.3.2.1 Run the CS-10 wheels on the coating for 1000 cycles to remove any surface roughness. The wheels shall be loaded with 1000 g with a vacuum on high for the entire test.

X1.3.2.2 Cool and weigh the specimen to the nearest 0.1 mg.

X1.3.2.3 Dress the CS-10 wheels with a CS-11 disc for 50 cycles.

X1.3.2.4 Abrasion test the coating with 1000 g load for 1000 cycles.

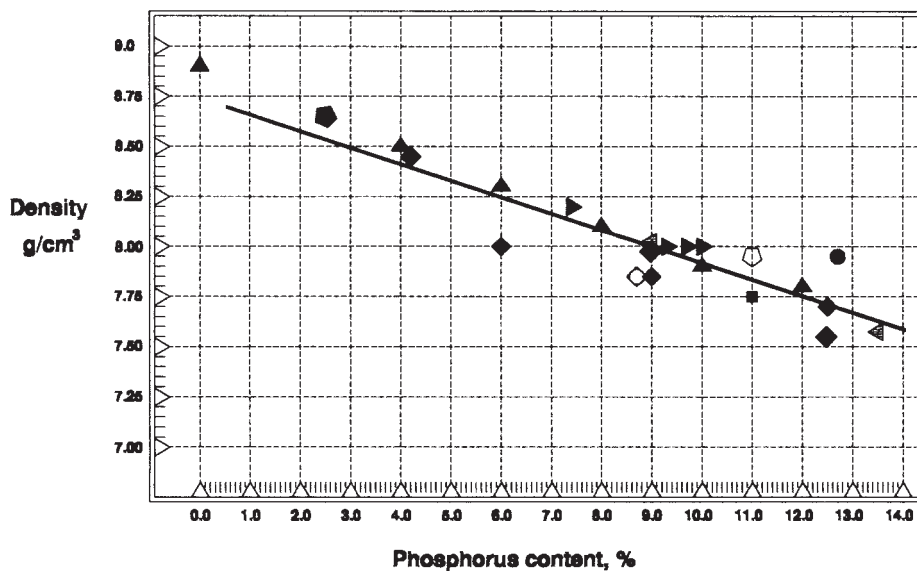
X1.3.2.5 Repeat X1.3.2.2, X1.3.2.3 and X1.3.2.4 until a total of 6000 cycles have been accomplished for each specimen.

X1.4 Reporting

X1.4.1 Determine the average weight loss in milligrams for each specimen per 1000 cycles Taber Wear Index and the mean weight loss per 1000 cycles for all specimens. Report the mean and standard deviation for the coating.

X2. DENSITY OF AUTOCATALYTIC NICKEL DEPOSITS

Density of Electroless Nickel Deposits



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FIG. X2.1 Density of Autocatalytic Nickel Phosphorus Alloy Summary of Reported Values

X3. HARDNESS VERSUS 1 H HEAT TREATMENT

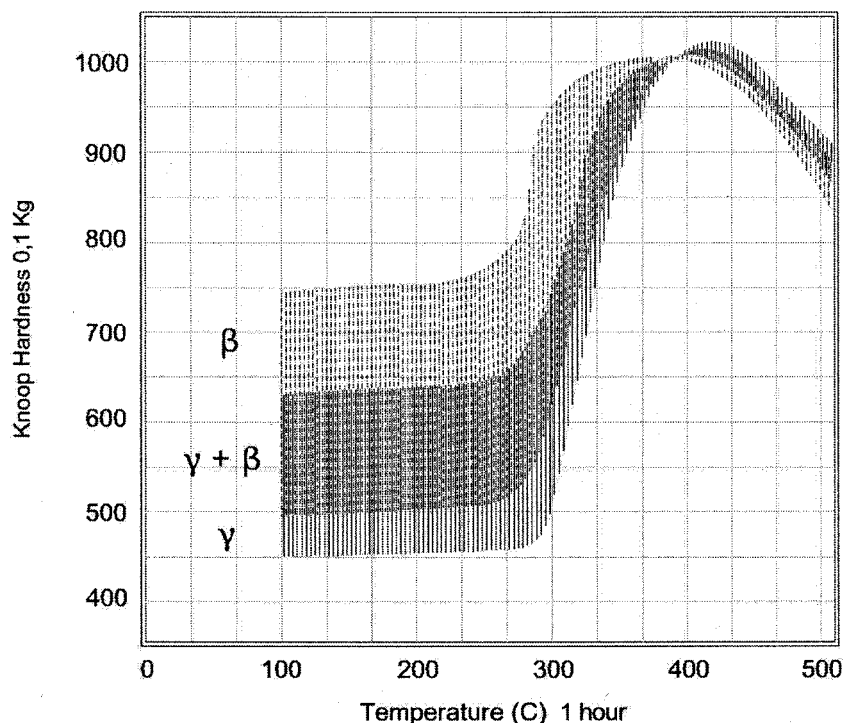


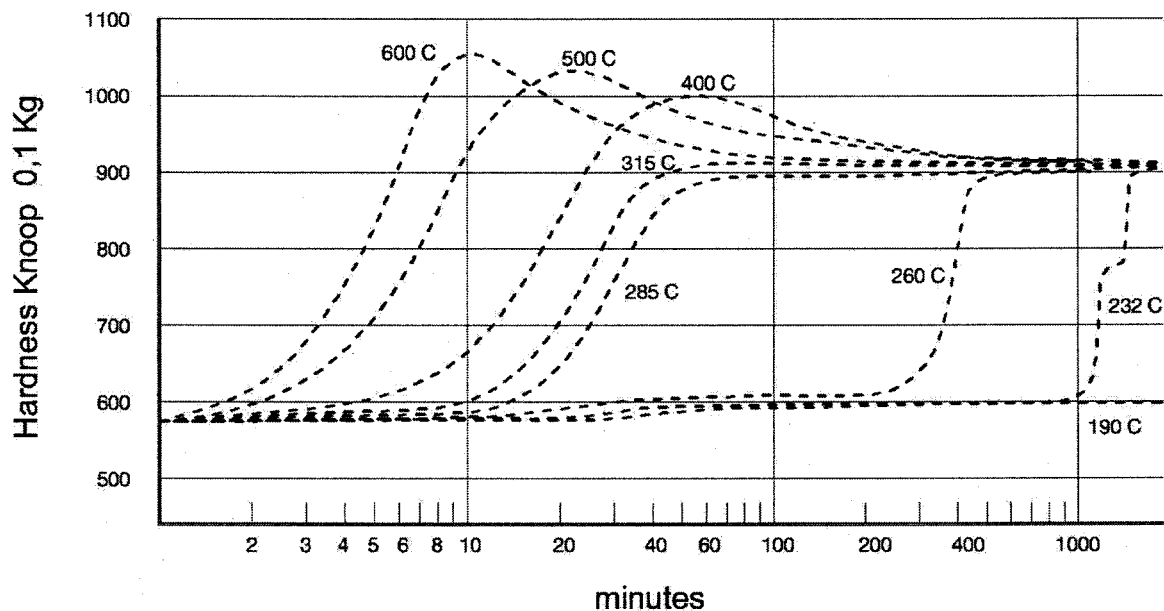
FIG. X3.1 Hardness of Autocatalytic Nickel Phosphorus Versus 1 h Heat Treatment Versus Phosphorus

X3.1 This graph information on the relationship of hardness and heat treatment for 1 h for different phases which approximate the TYPEs. Deposits that are low in phosphorus (β) are harder as plated and will transition between 200 and 400°C, producing a mixture of fcc Ni and Ni_3P . Deposits that are high in phosphorus (γ) are softer in the as plated condition and will also transition between 275 and 350°C, producing a mixture of fcc Ni and Ni_3P . Deposits in the range of 4 to 8 % are a mixture of both β and γ and have properties that are less predictable after heat treatment.

X3.2 The ultimate hardness of the deposit is dependent on the time and temperature of heat treatment as well as the phosphorus content, age of the solution, concentration of hypophosphite, and type of additive system used. Type I and IV deposits are subject to greater structural variation and their properties are less predictable.

X3.3 Fig. X3.1 and Appendix X4 can be used to approximate the hardness after heat treatment.

X4. HARDNESS VERSUS TIME AND TEMPERATURE



NOTE—These graphs are average and may vary with deviations from normal practice.

**FIG. X4.1 Hardness and Annealing Time Versus Temperature
9 % Phosphorus Deposit**

X4.1 Fig. X4.1 provides information on the effect of annealing time on the precipitation of nickel and 9 % phosphorus to Ni_3P and fcc Ni. Alloys of other phosphorus contents have differing hardness versus time and temperature. Deposits that are less than 7.5 % will have lower strength and start

precipitation hardening at 200°C. Deposits higher than 7.5 % will be stronger and when the deposit reaches around 11.2 % will be saturated with phosphorus. The precipitation of these high phosphorus deposits may be delayed for several minutes in the mid-range temperatures.

X5. ALLOY TYPES

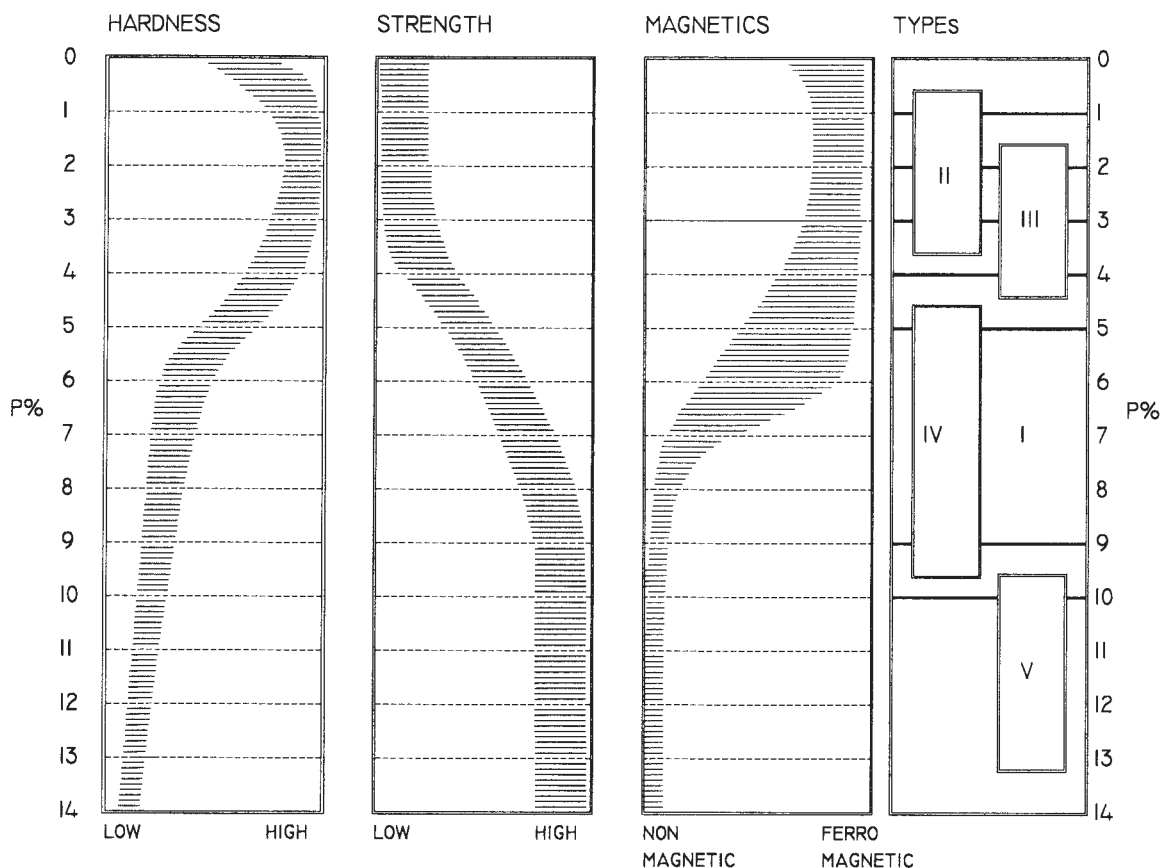


FIG. X5.1 Selected Properties of As Plated Autocatalytic Nickel Phosphorus Coatings by Phosphorus Content

NOTE X5.1—These different alloy TYPES are produced from processes which are specifically formulated and controlled. Additional requirements for internal stress, purity, and elongation may be necessary for some applications.

X5.1 The physical differences of these TYPES provide for a wide variation in performance in wire bonding, contact resistance, machineability, lubricity, reactivation and melting

point. These differences are produced by micro structural differences between Ni + Ni₃P and Ni₃P. These differences also effect non-destructive thickness testing by X-Ray and magnetic means. Careful attention to the selection of TYPE will insure optimum performance of the deposit in the intended application.

X6. SUMMARY PROGRAM 14

TABLE X6.1 Results of 110 Month Exposure of Autocatalytic (Electroless) Nickel Deposits at Kure Beach, North Carolina^A

Deposit Type	Source	Lot	Surface	Heat Treatment	Thickness, μm Target	Thickness, μm Actual	Phosphorus Alloy % wt		Practice B 537 Ratings 110 Months
							ICP	EDAX	
V	G	1	Smooth	No	12.5				3.6 ± 3.0
		2	Smooth	No	25				9.2 ± 0.3
		3	Smooth	No	75	80.5	10.5	10.9	10 ± 0
		4	Smooth	Yes	25				9.2 ± 0.3
		5	Ground	No	25				7.8 ± 0.4
V	E	1	Smooth	No	12.5				0.0 ± 0
		2	Smooth	No	25				8.0 ± 0
		3	Smooth	No	75	77	10.0	10.2	9.8 ± 0.4
		4	Smooth	Yes	25				8.2 ± 0.8
		5	Ground	No	25				4.2 ± 0.4
V	C	1	Smooth	No	12.5				6.8 ± 1.3
		2	Smooth	No	25				6.2 ± 0.4
		3	Smooth	No	75	46	9.9	10.4	8.8 ± 1.1
		4	Smooth	Yes	25				6.8 ± 1.3
		5	Ground	No	25				0.0 ± 0
IV	J	1	Smooth	No	12.5				0.0 ± 0
		2	Smooth	No	25				5.8 ± 0.4
		3	Smooth	No	75		9.0		9.6 ± 1.1
		4	Smooth	Yes	25				7.0 ± 0
		5	Ground	No	25				1.0 ± 2
IV	H	1	Smooth	No	12.5				0.6 ± 0.5
		2	Smooth	No	25				7.4 ± 0.9
		3	Smooth	No	75		8.9		10.0 ± 0
		4	Smooth	Yes	25				4.8 ± 1.1
		5	Ground	No	25				0.0 ± 0
IV	B	1	Smooth	No	12.5				0.0 ± 0
		2	Smooth	No	25				1.8 ± 1.6
		3	Smooth	No	75	79	8.3	7.3	6.0 ± 1.0
		4	Smooth	Yes	25				5.0 ± 0
		5	Ground	No	25				0.0 ± 0
IV	D	1	Smooth	No	12.5				0.0 ± 0
		2	Smooth	No	25				0.0 ± 0
		3	Smooth	No	75	77	8.2	9.5	5.0 ± 1.4
		4	Smooth	Yes	25				5.4 ± 0.5
		5	Ground	No	25				0.0 ± 0
IV	K	1	Smooth	No	12.5				0.0 ± 0
		2	Smooth	No	25				0.0 ± 0
		3	Smooth	No	75		7.5		0.6 ± 0.8
		4	Smooth	Yes	25				5.6 ± 2.8
		5	Ground	No	25				0.0 ± 0
IV	I	1	Smooth	No	12.5				0.0 ± 0
		2	Smooth	No	25				0.2 ± 0.4
		3	Smooth	No	75	80	7.1		0.4 ± 0.5
		4	Smooth	Yes	25				4.6 ± 0.8
		5	Ground	No	25				0.0 ± 0

^A The conclusions from this marine exposure program have been summarized by Dr. George DiBari of INCO in his paper Marine Corrosion Performance of EN Coating on Steel, Final Report on ASTM Program 14 which was presented at EN91 Conference, Product Finishing.

X6.1 Results of 110 Month Exposure of Autocatalytic Nickel Deposits at Kure Beach, North Carolina:

X6.1.1 Program 14 is part of an ongoing marine exposure testing process at the 75 m site at LaQue Center for Corrosion Technology, Wrightsville, NC (Kure Beach). The program called for the plating of both Type IV and Type V deposits on standard smooth and ground steel Q Panels.

X6.1.2 There were nine different sources of deposits, each providing five lots of five panels. The program involved plating 12.5, 25, and 75 μm thicknesses on smooth and ground surface and one smooth lot heat treated for hardness at 550°C for 2 h. The heat treatment temperature was considerably higher than

typical processing and was chosen to evaluate the formation of diffusion products of iron, nickel and phosphorus.

X6.1.3 The following matrix of test panels were prepared and exposed with subsequent analysis for alloy and thickness. Panels were rated at Kure Beach each year by a team from ASTM Committee B08 using Practice B 537.

X6.1.4 The interpretation of the results of these exposure test should be made on the basis of general performance of the coating on panels. Base metal condition, undercoats, surface preparation, and post processing all have a significant effect on the performance and should be given careful consideration when designing the part and pretreatment processing sequence.



X7. STRIPPING AND REWORK

X7.1 The stripping of electroless nickel deposits requires the use of aggressive chemical or mechanical systems that may damage the base material. Consideration of the risk of stripping the deposit should always be considered when selecting this coating. Undercoats may be required to facilitate rework. The commercially available strippers that have minimal attack of the substrate are available to minimize the risk of etching or pitting of the substrate.

X7.2 Nitric acid is recommended to strip electroless nickel deposits from aluminum. A solution of between 20 to 30 % by weight nitric acid should be prepared. After mixing, let stand until the temperature reaches ambient. If significant area is to be stripped, the solution should be cooled. Articles to be stripped should be clean and free of oils and surface debris that may interfere with the stripping process. Racks and fixtures made with nylon and drylen are not recommended and are not compatible with nitric acid and will decompose to a sticky tar in the stripper. This tar will be difficult to remove from the article after stripping. After all the electroless nickel is removed, the part should be rinsed in clean rinse water.

X7.3 Highly alkaline solutions using nitro benzoic acids are used to remove electroless nickel from steels and coppers.

These solutions are heated and generally remove the deposit at a slower rate than nitric acid. As the stripping process proceeds, a corrosion product may form on the surface which will slow the stripping. When this occurs, the steel or copper plated article is removed, rinsed, electrocleaned, rinsed, and returned to the stripper. As the deposit nears complete removal, it is common for a slight etch of the substrate to occur. The amount of material removed varies from nothing to 0.0003 in.

X7.4 Mechanical peening with a grit is effective in removing electroless nickel from hard steel parts with non-critical tolerances. The article is placed in a chamber and blasted with a heavy grit until all the deposit is removed. This method is effective when parts are large and stripping solutions would be difficult to manage.

X7.5 Generally the rework process is limited to less than three times. This is because there is some substrate removal each time the article is stripped. When processing some copper or steel alloys, it is advisable to apply a copper undercoat that will prevent the attack of the substrate as the stripping nears completion. This is always a good practice when dealing with high value copper alloy articles.

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