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Standard Specification for Electrodeposited Coatings of Palladium-Nickel for Engineering Use¹

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1. Scope

1.1 *Composition*—This specification covers requirements for electrodeposited palladium-nickel coatings containing between 70 and 95 mass % of palladium metal. Composite coatings consisting of palladium-nickel and a thin gold overplate for applications involving electrical contacts are also covered.

1.2 *Properties*—Palladium is the lightest and least noble of the platinum group metals. Palladium-nickel is a solid solution alloy of palladium and nickel. Electroplated palladium-nickel alloys have a density between 10 and 11.5, which is substantially less than electroplated gold (17.0 to 19.3) and comparable to electroplated pure palladium (10.5 to 11.8). This yields a greater volume or thickness of coating per unit mass and, consequently, some saving of metal weight. The hardness range of electrodeposited palladium-nickel compares favorably with electroplated noble metals and their alloys (1, 2).²

NOTE 1—Electroplated deposits generally have a lower density than their wrought metal counterparts.

	Approximate Hardness (HK ₂₅)
Gold	50–250
Palladium	75–600
Platinum	150–550
Palladium-Nickel	300–650
Rhodium	750–1100
Ruthenium	600–1300

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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:

¹ This specification is under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and is under the direct responsibility of Subcommittee B08.08.01 on Engineering Coatings.

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² The boldface numbers in parentheses refer to the list of references at the end of this specification.

- B 183 Practice for Preparation of Low-Carbon Steel for Electroplating³
- B 242 Practice for Preparation of High-Carbon Steel for Electroplating³
- B 254 Practice for Preparation of and Electroplating on Stainless Steel³
- B 281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings³
- B 322 Practice for Cleaning Metals Prior to Electroplating³
- B 343 Practice for Preparation of Nickel for Electroplating with Nickel³
- B 374 Terminology Relating to Electroplating³
- B 481 Practice for Preparation of Titanium and Titanium Alloys for Electroplating³
- B 482 Practice for Preparation of Tungsten and Tungsten Alloys for Electroplating³
- B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section³
- B 488 Standard Specification for Electrodeposited Coating on Gold for Engineering Use³
- B 489 Practice for Bend Test for Ductility of Electrodeposited and Autocatalytically Deposited Metal Coatings on Metals³
- B 507 Practice for Design of Articles to Be Electroplated on Racks³
- B 542 Terminology Relating to Electrical Contacts and Their Use⁴
- B 558 Practice for Preparation of Nickel Alloys for Electroplating³
- B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry³
- B 571 Test Methods for Adhesion of Metallic Coatings³
- B 578 Test Method for Microhardness of Electroplated Coatings³
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings³
- B 689 Specification for Electroplated Engineering Nickel Coatings³
- B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings³

³ Annual Book of ASTM Standards, Vol 02.05.

⁴ Annual Book of ASTM Standards, Vol 03.04.

- B 741 Test Methods for Porosity in Gold Coatings on Metal Substrates by Paper Electrography³
- B 748 Test Method for Measurement of Thickness of Metallic Coatings by Measurement of a Cross Section with a Scanning Electron Microscope³
- B 762 Method of Variables Sampling of Metallic and Inorganic Coatings³
- B 765 Guide to Selection of Porosity Tests for Electrodeposits and Related Metallic Coatings³
- B 798 Test Method for Porosity in Gold or Palladium Coatings on Metal Substrates by Gel-Bulk Electrography⁴
- B 799 Test Method for Porosity in Gold and Palladium Coatings by Sulfurous Acid/Sulfur-Dioxide Vapor⁴
- B 809 Test Method for Porosity in Metallic Coatings by Humid Sulfur Vapor (“Flowers of Sulfur”)³
- B 827 Practice for Mixed Flowing Gas (MFG) Environmental Tests⁴
- B 845 Guide to Mixed Flowing Gas (MFG) Tests for Electrical Contacts⁴
- B 849 Specification for Pre-Treatments of Iron or Steel for Reducing the Risk of Hydrogen Embrittlement³
- B 850 Specification for Post-Coating Treatments of Iron or Steel for Reducing the Risk of Hydrogen Embrittlement³
- D 1125 Test Methods for Electrical Conductivity and Resistivity of Water⁵
- D 3951 Practice for Commercial Packaging⁶

TABLE 1 Composition Type

Type	Nominal Composition (Mass %)	Range (Mass% Pd)
I	75 % Pd/25 % Ni	70–80 % Pd
II	80 % Pd/20 % Ni	75–85 % Pd
III	85 % Pd/15 % Ni	80–90 % Pd
IV	90 % Pd/10 % Ni	85–95 % Pd

TABLE 2 Thickness Class^A

Thickness Class	Minimum Thickness of Pd-Ni (µm)
0.4	0.4
0.5	0.5
0.7	0.7
1.0	1.0
1.3	1.3
1.5	1.5
2.0	2.0
2.5	2.5
3.0	3.0

^A See Appendix X3 on Electrical Contact Performance Versus Thickness Class.

TABLE 3 Gold Overplate^A

Grade	Type	MIL-G-45204	Hardness (Code)	Thickness Range
0	No Overplate
1	1 (99.9 % Au min)	III	90 HK ₂₅ max (A)	0.05–0.12 µm
2	2 (99.7 % Au min)	I	130–200 HK ₂₅ (C)	0.05–0.25 µm

^A See Specification B 488 and Appendix X1 and Appendix X2.

3. Terminology

3.1 *Definitions:* Many terms used in this specification are defined in Terminology B 374 or B 542.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *overplating, n*—a coating applied onto the topmost palladium-nickel coating. The thickness of an overplating or “flash” is usually less than 0.25 µm.

3.2.2 *significant surfaces, n*—those surfaces normally visible (directly or by reflection) or which are essential to the serviceability or function of the article; or which can be the source of corrosion products or tarnish films that interfere with the function or desirable appearance of the article. The significant surfaces shall be indicated on the drawings of the parts, or by the provision of suitably marked samples.

3.2.3 *underplating, n*—a metallic coating layer or layers between the basis metal or substrate and the palladium-nickel coating. The thickness of an underplating is usually greater than 1 µm, in contrast to a strike which is thinner.

4. Classification

4.1 Orders for articles to be plated in accordance with this specification shall specify the coating system, indicating the basis metal, the thicknesses of the underplatings, the type and thickness class of the palladium-nickel coating, and the grade of the gold overplating according to Table 1, Table 2, and Table 3. See Section 7.

5. Ordering Information

5.1 In order to make the application of this specification

complete, the purchaser shall supply the following information to the seller in the purchase order or other governing document:

5.1.1 The name, designation, and date of issue of this specification;

5.1.2 The coating system including basis metal, composition type, thickness class and gold overplate grade (see 4.1 and Table 1, Table 2, and Table 3);

5.1.3 Presence, composition, and thickness of underplating (see 3.2.1). For nickel underplating see 6.5.1;

5.1.4 Significant surfaces shall be defined (see 3.2.3);

5.1.5 Requirements, if any, for porosity testing (see 9.6);

5.1.6 (Steel parts only) Stress relief if required (see Specification B 849);

5.1.7 (Steel parts only) Hydrogen embrittlement relief (see B 850);

5.1.8 Sampling plan employed (see Section 8); and,

5.1.9 Requirement, if any, for surface coating cleanliness (absence of residual salts). See Appendix X6.

6. Manufacture

6.1 Any process that provides an electrodeposit capable of meeting the specified requirements will be acceptable.

6.2 *Substrate:*

6.2.1 The surface condition of the basis metal should be specified and should meet this specification prior to the plating of the parts.

6.2.2 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 should be brought to the attention of the supplier and the purchaser.

⁵ Annual Book of ASTM Standards, Vol 11.01.

⁶ Annual Book of ASTM Standards, Vol 15.09.

6.2.3 Clean the basis metal as necessary to ensure a satisfactory surface for subsequent electroplating in accordance with Practices B 183, B 242, B 254, B 281, B 322, B 343, B 481, B 482 and B 558.

6.2.4 Proper preparatory procedures and thorough cleaning of the basis metal are essential for satisfactory adhesion and performance of these coatings. The surface must be chemically clean and continuously conductive, that is, without inclusions or other contaminants. The coatings must be smooth and as free of scratches, gouges, nicks, and similar imperfections as possible.

NOTE 2—A metal finisher can often remove defects through special treatments such as grinding, polishing, abrasive blasting, chemical treatments, and electropolishing. However, these may not be normal in the treatment steps preceding the plating, and a special agreement is indicated.

6.3 If required (see 5.1.6), steel parts with a hardness greater than 1000 MPa (31 HRC) shall be given a suitable stress relief heat treatment prior to plating in accordance with Specification B 849. Such stress relief shall not reduce the hardness to a value below the specified minimum. Avoid acid pickling of high strength steels.

6.3.1 Apply the coating after all basis metal preparatory heat treatments and mechanical operations on significant surfaces have been completed.

6.4 Racking:

6.4.1 Position parts to allow free circulation of solution over all surfaces. The location of rack or wire marks in the coating should be agreed upon between the producer and supplier.

6.5 Plating Process:

6.5.1 *Nickel Underplating*—Apply a nickel underplating before the palladium-nickel when the product is made from copper or copper alloy. Nickel underplatings are also applied for other reasons. See Appendix X5.

NOTE 3—In certain instances where high frequency analog signals are employed, such as wave guides, the magnetic properties of nickel may attenuate the signal. Palladium-nickel itself is non-ferromagnetic when the nickel content is less than 14 mass %.

NOTE 4—In applications where forming or flaring operations are to be applied to the plated component, a ductile nickel electrodeposit should be specified.

6.5.2 *Strikes*—Good practice suggests the use of a palladium strike to follow any underplate or substrate (other than silver or platinum) immediately prior to applying the palladium-nickel.

6.5.3 *Plating*—Good practice calls for the work to be electrically connected when entering the palladium-nickel solution.

NOTE 5—Some palladium-nickel electroplating solutions attack copper. This can result in codeposition of copper impurity. The situation is further aggravated when low current densities are utilized. Copper can be removed from solutions by low current density electrolysis (0.1 to 0.3 mA/cm²).

6.5.4 *Gold Overplating*—Apply a thin gold overplating after the palladium-nickel in any application in which palladium-nickel plated electrical connectors are mated together in a contact pair. This process is necessary to preserve the performance of the contact surface. See Appendix X1 for other reasons for using a gold overplate.

NOTE 6—When using Type 1 gold, the thickness of the gold overplate shall not exceed 0.12 μm (5 $\mu\text{in.}$) due to increased risk of degrading durability and increasing the coefficient of friction.

6.5.5 *Residual Salts*—For rack and barrel plating applications, residual plating salts can be removed from the articles by a clean, hot (50 to 100°C) water rinse. A minimum rinse time of 2.5 min (racks) or 5 min (barrel) is suggested. Best practice calls for a minimum of three dragout rinses and one running rinse with dwell times of 40 s in each station when rack plating and 80 s when barrel plating. Modern high-velocity impingement type rinses can reduce this time to a few seconds. This is particularly useful in automatic reel-to-reel applications where dwell times are significantly reduced. See Appendix X6.

7. Coating Requirements

7.1 *Nature of Coating*—The palladium-nickel deposit shall have a minimum purity of 70 mass % palladium.

7.2 *Composition*—The composition of the palladium-nickel electrodeposit shall be within ± 5 mass % of the specified type.

7.3 *Appearance*—Palladium-nickel coatings shall be coherent, continuous, and have a uniform appearance to the extent that the nature of the basis metal and good commercial practices permit.

7.4 *Thickness*—Everywhere on the significant surface (see 5.1), the thickness of the palladium-nickel coating shall be equal to or exceed the specified thickness. The maximum thickness, however, shall not exceed the drawing tolerance.

NOTE 7—The coating thickness requirement of this specification is a minimum requirement, that is, the coating thickness is required to equal or exceed the specified thickness everywhere on the significant surfaces while conforming to all maximum thickness tolerances given in the engineering drawing. Variation in the coating thickness from point to point on a coated article is an inherent characteristic of electroplating processes. The coating thickness at any single point on the significant surface, therefore, will sometimes have to exceed the specified value in order to ensure that the thickness equals or exceeds the specified value at all points. Hence, most average coating thicknesses will be greater than the specified value. How much greater is largely determined by the shape of the article (see Practice B 507) and the characteristics of the plating process. In addition, the average coating thickness on products will vary from article to article within a production lot. If all of the articles in a production lot are to meet the thickness requirement, the average coating thickness for the production lot as a whole will be greater than the average necessary to assure that a single article meets the requirement. See 8.1.

7.5 *Adhesion*—The palladium-nickel coatings shall be adherent to the substrate or underplate when tested by one of the procedures summarized in 9.5.

7.6 Integrity of the Coating:

7.6.1 *Gross Defects/Mechanical Damage*—The coatings shall be free of visible mechanical damage and similar gross defects when viewed at magnifications up to 10 \times . For some applications this requirement may be relaxed to allow for a small number of such defects (per unit area), especially if they are outside of or on the periphery of the significant surfaces. See 7.6.2.

7.6.2 *Porosity*—Almost all as-plated electrodeposits contain some porosity, and the amount of porosity to be expected for any one type of coating will increase with decreasing the thickness of that particular coating type. The amount of porosity in the coating that may be tolerable depends on the



severity of the environment that the article is likely to encounter during service or storage. If the pores are few in number, or away from the significant surfaces, their presence can often be tolerated. Acceptance or pass-fail criteria, if required, shall be part of the product specification for the particular article or coating requiring the porosity test. See 9.6.

NOTE 8—Extensive reviews of porosity and porosity testing can be found in the literature (3, 4).

8. Sampling

8.1 The purchaser and producer are urged to employ statistical process control in the coating process. Properly performed, statistical process control will assure coated products of satisfactory quality and will reduce the amount of acceptance inspection. The sampling plan used for the inspection of the quality of the coated articles shall be as agreed upon between the purchaser and the supplier.

8.1.1 When a collection of coated articles (the inspection lot (see 8.2)) is examined for compliance with the requirements placed on the articles, a relatively small number of the articles (the sample) is selected at random and is inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection of the 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. Test Method B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for the sampling inspection of coatings.

8.1.2 Test Method B 602 contains four sampling plans, three for use with tests that are non-destructive and one when they are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 identifies the plan to be used.

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

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

8.2 An inspection lot shall be defined as a collection of coated articles that are of the same kind, that have been produced to the same specifications, 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.

9. Test Methods

9.1 *Appearance*—The coating shall be examined at up to 10× magnification for conformance to the requirements of appearance.

9.2 *Alloy Composition*—Alloy composition of the palladium-nickel can be determined by a wet method, X-ray Fluorescence (XRF), Scanning Electron Microscopy (SEM)/

Energy Dispersive Spectroscopy (EDS), Auger, or by Electron Probe X-ray Microanalysis (EPMA)/Wavelength Dispersive Spectroscopy (WDS).

9.2.1 The method chosen for determination of alloy composition shall not be the same method used for determination of deposit thickness if the deposit is over a nickel underplate or as a referee method. The reason for this is that the determination of alloy composition and the determination of deposit thickness by spectrographic analysis are to some extent interdependent. See 9.2.4.1 and 9.4.1.

9.2.2 *Wet Method*—Use any recognized method to determine quantitatively the relative concentrations of palladium and nickel present. Atomic absorption spectrophotometry (or any other methods with demonstrated uncertainty less than 10 %) may be used to determine the alloy composition.

NOTE 9—Determination of alloy composition by dissolving the coating from a test specimen must be obtained by electroplating the palladium-nickel directly over a non-nickel containing alloy substrate with no intermediate layer. Copper alloy substrates are preferred. Alloy composition is best determined on a special test specimen. One must be careful to arrange the specimen so as to electroplate at a typical current density, similar to what is used in production. Palladium-nickel may be stripped by utilizing a 90 volume % (reagent grade) sulfuric acid, 10 volume % (reagent grade) nitric acid solution.

9.2.3 *XRF*—XRF can be used for composition analysis of palladium-nickel alloy coatings deposited directly onto copper or a copper alloy that does not contain nickel. This method is not suitable for composition analysis of palladium-nickel alloy coatings less than 60 μm in thickness when deposited over nickel or nickel containing substrates.

NOTE 10—If the palladium-nickel coating is less than 60 μm, palladium-nickel alloy composition measurements in the presence of an intermediate nickel layer or nickel containing substrate is degraded by the fact that the nickel X-ray emission of the alloy layer and the intermediate layer (or substrate) cannot be accurately distinguished from one another.

9.2.4 EPMA:

9.2.4.1 EPMA based on electron beam excitation of X-rays characteristic of the elements present can be used to measure composition of palladium-nickel alloy coatings on top of any undercoat or any substrate to an accuracy of 0.1 mass % palladium if the thickness of the coating is ≥1.5 μm. See Appendix X8.

9.2.4.2 EPMA shall be used as the referee method for the determination of alloy composition.

9.2.5 *SEM/EDS*—The SEM/EDS technique is capable of determining composition of palladium-nickel coatings that are ≥1.5 μm thick to an accuracy and precision of ±0.2 mass % palladium. A procedure for calibration of a conventional SEM equipped with an X-ray EDS for routine analysis of palladium-nickel alloy coating composition appears in Appendix X7.

9.2.6 *Auger Electron Spectroscopy (AES) and X-ray Photoelectron Spectroscopy (XPS)*—AES and XPS are capable of analyzing regions that are of the order of 0.002 μm thick. These techniques are potential candidates for analysis of electrodeposited palladium-nickel alloy coatings with a thickness of ≥0.03 μm.

NOTE 11—The use of AES and XPS to determine bulk coating composition requires the sputter removal of 0.01 to 0.02 μm of material from the surface to remove surface contaminants and surface composition



gradients. Some, but not all, commercial AES and XPS instruments are capable of accurate analyses of palladium-nickel alloy composition using an internal procedure for determining correction factors similar to that described in Appendix X7 for an SEM/EDS instrument.

9.3 Deposit Purity—Use any recognized method to determine quantitatively the metallic impurities present. Atomic absorption spectrophotometry (or any other methods with demonstrated uncertainty less than 10 %) may be used to determine the metallic impurities. Initial scanning should be carried out for all elements, in order to detect any unknown or unexpected impurities. Determine deposit purity by subtracting total impurities from 100 %.

NOTE 12—Deposit purity is best determined on a special test specimen. One shall be careful to arrange the specimen so as to electroplate at a typical current density, similar to what is used in production. Palladium-nickel may be stripped by utilizing a 90 volume % (reagent grade) sulfuric acid, 10 volume % (reagent grade) nitric acid solution. The test specimen substrate should be platinum, gold, or an electrodeposit not attacked by the strip solution. For the determination of impurities, the total palladium-nickel deposit should be over 100 mg and the sample weight is determined by a weigh-strip-weigh procedure. The strip solution is then used for quantitative analysis of impurities.

9.4 Thickness:

9.4.1 Measure thickness by methods outlined in Test Methods B 487, B 568, or B 748, or any other test method that has an uncertainty less than 10 %. See Appendix X2 for specific information on thickness measurement of palladium-nickel alloy coatings by XRF.

9.4.2 Use Test Method B 748 as the referee method for the determination of deposit thickness.

9.5 Adhesion—Determine adhesion by one of the following procedures (see Test Methods B 571 for full details):

9.5.1 Bend Test—Bend the electroplated article repeatedly through an angle of 180° on a diameter equal to the thickness of the article until fracture of the basis metal occurs. Examine the fracture at a magnification of 10×. Cracking without separation does not indicate poor adhesion unless the coating can be peeled back with a sharp instrument.

9.5.2 Heat Test—No flaking, blistering, or peeling shall be apparent at a magnification of 10× after the palladium-nickel electroplated parts are heated to 300 to 350°C (570 to 660°F) for 30 min and allowed to air cool.

9.5.3 Cutting Test—Make a cut with a sharp instrument and then probe with a sharp point and examine at a magnification of 10×. No separation of the coating from the substrate or intermediate layers shall occur.

9.6 Plating Integrity—Porosity and microcracks shall be determined by either Test Methods B 741, B 798, B 799, or B 809 unless otherwise specified. Do not use the nitric acid vapor test (palladium-nickel can dissolve in nitric acid).

NOTE 13—The test to be selected will depend on the palladium-nickel thickness, the nature of the basis metal, the nature and thickness of any intermediate layers or underplate, and the shape of the palladium-nickel plated part. Guide B 765 is suitable to assist in the selection of porosity tests for electrodeposits of palladium-nickel alloys.

9.7 Ductility—When required, determine ductility in accordance with Practice B 489.

10. Special Government Requirements

10.1 The following special requirements shall apply when the ultimate purchaser is the federal government or an agent of the federal government.

10.1.1 Sampling—For government acceptance, the sampling plan specified in MIL-STD-105 is to be used instead of the ASTM standards specified in 8.1.

10.1.2 Thickness Testing:

10.1.2.1 In addition to the nondestructive methods outlined in Test Method B 568, a cross-sectioning method, such as that specified by Test Methods B 487 or B 748, shall be used as a referee method to confirm the precision and bias of the particular nondestructive technique that is used.

10.1.2.2 The palladium-nickel thickness on significant surfaces shall be at least 1.3 μm (50 μin.), unless otherwise specified on the drawings or in the contract. The coating on nonsignificant surfaces shall be of sufficient thickness to ensure plating continuity and uniform utility, appearance, and protection. The thickness of plating on nonsignificant surfaces, unless specifically exempted, shall be a minimum of 60 % of that specified for significant surfaces.

10.1.3 Packaging—The packaging and packing requirements shall be in accordance with Practice D 3951 or as specified in the contract or order.

APPENDIXES

(Nonmandatory Information)

X1. SOME REASONS FOR USING A GOLD OVERPLATE

X1.1 A gold overplate is employed to enhance the performance of the palladium-nickel surface. Two types of gold are used:

X1.1.1 Type 1 gold is used in the critical areas in thickness ranges of 0.05 to 0.12 μm.

X1.1.2 Type 2 gold is used in the critical areas in thickness ranges of 0.05 to 0.25 μm or higher.

X1.2 The gold overplate offers the following performance

enhancements to palladium-nickel:

X1.2.1 Durability—Palladium-nickel has a higher coefficient of friction than gold. A gold overplate of proper thickness, therefore, reduces friction and enhances durability. The gold overplate actually provides a low shear strength solid lubricant that reduces friction wear (**5, 6**). Either Type 1 or Type 2 gold works in this application (**6-8**). Type 1 gold should be used at a thickness no greater than 0.12 μm to maintain a low



coefficient of friction. Palladium-nickel should not be mated against itself in a sliding contact pair when durability and resistance to fretting is desired.

X1.2.2 *Mating Force*— Application of Type 1 or Type 2 gold reduces friction and mating force. Type 1 should be no more than 0.12 μm thick.

X1.2.3 *Fretting*—Fretting occurs when two surfaces undergo low amplitude, repetitive motions. Depending on conditions and contact surface materials, fretting wear or fretting corrosion can occur. Fretting wear is loss of material along the wear track. Fretting corrosion is the formation of surface oxides at the contact surface. The addition of a Type 1 or Type 2 gold can often reduce fretting corrosion that is due to fretting motions (9). The occurrence of fretting is influenced greatly by contact design. See Terminology B 542.

X1.2.4 *Frictional Polymerization*—Frictional polymerization is the formation of insulating polymeric films at the contact spot. Such occurrences have been documented for palladium-nickel, pure palladium, and other metals (5). The addition of a Type 1 or Type 2 gold overplate can often reduce frictional polymer formation (9).

X1.2.5 *Solderability*— The addition of a Type 1 or Type 2 gold overplate enhances the solderability and shelf life of palladium-nickel. Type 1 gold is more solderable than Type 2.

X1.2.6 *Thermal Stability*—Gold overplating of palladium-nickel plated surfaces greatly improves the stability of contact resistance during prolonged exposure to temperatures ranging from 105° to 120°C (10). A Type 1 gold overplate imparts better thermal stability than a Type 2 gold overplate.

X2. THICKNESS MEASUREMENTS OF PALLADIUM-NICKEL ALLOY COATINGS BY XRF

X2.1 These guidelines are intended to aid purchasers of palladium-nickel alloy electroplating systems in properly setting up XRF instruments to measure the deposit thickness. This calibration method and measurement mode is for the determination of coating thickness only. The procedure is not intended for alloy composition measurement.

X2.2 Base metal type (substrate composition) must be considered for each product to be measured. Due to X-ray interference from the tin in phosphor-bronze substrates, some X-ray units must be calibrated using standards having the same substrate material as the product to be measured. Some commercially available X-ray units have substrate correction capability and do not require different standards for different copper alloy substrates. Follow the instrument manufacturer's instructions when calibrating the instrument for measurement on copper alloy substrates.

X2.3 A bare substrate, a palladium-nickel alloy saturation thickness standard, and at least two palladium-nickel alloy thickness standards having the same alloy composition as the product to be measured are required for calibration of the X-ray unit. The thickness of the alloy standards must bracket the production plating thickness range to be measured. The thickness saturation standard should have a thickness greater than 60 μm (2400 μin.).

X2.3.1 Alternatively, pure palladium standards could be used in place of the palladium-nickel standards of X2.3. By this method, the XRF will measure only the palladium counts emitted from the palladium-nickel sample. *Providing the alloy composition is known*, the palladium-nickel thickness can be determined by multiplying the XRF value by the correction factor given in Table 4.

X2.4 In calibrating the X-ray unit, the following guidelines are recommended:

X2.4.1 Select single coating excitation measurement and calibration mode;

X2.4.2 Do not use a filter (absorber);

X2.4.3 Calibrate with the same collimator size as will be

TABLE 4 Correction Factors

Mass % Pd	Pd-Ni Density	Factor
60.0	10.58	1.84
62.0	10.64	1.77
64.0	10.69	1.71
66.0	10.75	1.65
68.0	10.80	1.59
70.0	10.86	1.54
72.0	10.92	1.49
74.0	10.97	1.44
76.0	11.03	1.40
78.0	11.08	1.35
80.0	11.14	1.31
82.0	11.20	1.27
84.0	11.25	1.24
86.0	11.31	1.20
88.0	11.36	1.17
90.0	11.42	1.14
92.0	11.48	1.11
94.0	11.53	1.08
96.0	11.59	1.05
98.0	11.64	1.03
100.0	11.70	1.00

needed to measure product;

X2.4.4 Total calibration measurement time per standard (number of measurements per standard multiplied by the calibration measurement time) should be at least 120 s. Multiple readings should be taken from each standard, for example, 4 measurements per standard multiplied by a 30 s measurement time each equals 120 s.

X2.4.5 Verify that a proper region of interest window (R.O.I.) has been set. Some instruments set the R.O.I. automatically from a list of applications (older instruments require manual R.O.I. setting).

X2.4.6 Ensure that the instrument has been adjusted for intensity variations (intensity correction or reference measurement, depending on instrument manufacturer).

X2.4.7 Following the calibration, measure the calibration standards to verify that the instrument is properly calibrated. Measure each standard ten times. The mean value of the ten measurements should fall within the predicted theoretical instrument precision for a mean of ten readings.

X2.4.8 Ensure the optical alignment (collimator to optics)



of the XRF instrument is correct.

X2.4.9 Carefully focus on the measurement area of the test specimen.

X2.4.10 The test specimen should be properly oriented with respect to the X-ray detector.

X2.4.11 The surface to be measured should be flat and level with respect to the measurement stage. Curved surfaces may be measured subject to proper collimator size selection and proper orientation of the axis of curvature. Follow instrument manufacturer's instructions.

X2.4.12 Handle the calibration standards and product carefully since surface damage will affect accuracy.

X2.4.13 Make adjustments periodically to the instrument to correct for drift effects as instructed by the manufacturer.

Following this adjustment, verify the instrument calibration by measuring a known thickness standard or reference specimen.

X2.5 Prior to measuring the product containing a gold flash overplate, remove the gold with a suitable gold stripping solution. Potassium or sodium cyanide based gold stripping solutions are commercially available.

X2.6 Recommended measurement times to ensure accurate and reproducible results are as follows:

Plating Thickness (µm)	Measurement Time (s)
0-0.25	25
0.25-1.0	20
>1.0	15

X3. ELECTRICAL CONTACT PERFORMANCE VERSUS THICKNESS CLASS

X3.1 *General*—It is difficult to specify electrical contact performance as a function of plating thickness because performance is also strongly affected by such things as metallurgy, contact design, quality, and fabrication techniques. It is possible, therefore, to have a wide range of thickness classes for a particular application.

X3.2 Electrical contact performance is generally defined in terms of the ability of a contact system to maintain a low and stable interfacial resistance when exposed to mating cycles in the product's service or storage environment. This is usually determined by some type of environmental performance test

such as those involving mixed flowing gas, for example Test Methods B 827 and B 845.

X3.3 Palladium-nickel thickness ranges that are often used in the electronics industry for specific applications are as follows:

Thickness (µm)	Application
0.8-3.0	Printed circuit edge card connectors
0.8-1.5	Low energy electrical contacts
0.4-0.5	Electrical contacts where little adverse environmental, electrical, or mechanical action is expected.

X4. INTERMATEABILITY GUIDELINES

X4.1 Most mating combinations are acceptable when both mating halves have either precious metal or non-precious metal. The mixture of precious metal with non-precious metal is generally not recommended.

X4.2 The keys to good contact functioning are contact force, stability through good design, and the proper selection of coatings. Lubrication, with its advantages and disadvantages, also can play a beneficial role.

X4.3 The use of special lubricants can serve to improve environmental resistance, wear resistance, and reduce mating force. Lubrication can also reduce the severity of fretting wear and fretting corrosion (11).

X4.4 In unlubricated situations, the following combinations are recommended:

Contact 1 Coating Metallurgy	With:	Contact 2 Coating Metallurgy
PdNi		Gold (Type 2)
PdNi		PdNi with gold overplate (Type 1 or 2 gold)
PdNi with gold overplate (Type 1 or 2 gold)		PdNi with gold overplate (Type 1 or 2 gold)

PdNi with gold overplate (Type 1 or 2 gold)	Gold (Type 2)
PdNi with gold overplate (Type 1 or 2 gold)	Pd with gold overplate (Type 1 or 2 gold)
PdNi	Pd with gold overplate (Type 1 or 2 gold)
PdNi with gold overplate (Type 1 or 2 gold)	Pd

X4.5 Specific applications with wide differences in thickness class, high normal forces, or unusual performance requirements should be investigated to ensure functionality.

X4.6 In unlubricated situations, the following combinations are not recommended:

Contact 1	With:	Contact 2
PdNi		PdNi
PdNi		Pd
PdNi		Solder or tin
PdNi with gold overplate (Type 1 or 2 gold)		Solder or tin

X4.7 When suitable lubricants are applied to one or both mating halves, the guidelines in X4.4 still apply. Lubricants, however, can change the performance and alter the guidelines expressed in X4.6, with the exception of those for solder or tin.

X5. SOME REASONS FOR USING A NICKEL UNDERPLATE FOR PALLADIUM-NICKEL ELECTROPLATING

X5.1 Diffusion Barrier—To inhibit diffusion of copper from the basis metal into the palladium-nickel.

X5.2 Levelling Layer—To produce a smoother surface than the basis metal in order to ensure a lower porosity palladium-nickel top coat, for example, levelling nickel over a rough substrate.

X5.3 Pore Corrosion Inhibitor—A nickel underplate under the palladium-nickel top coat will form passive oxides at the base of pores in humid air, provided the environment does not contain significant amounts of acidic pollutants, such as SO₂ or HCl.

X5.4 Load Bearing Underlayer for Contacting Surfaces—A hard nickel underplate can serve as a load bearing

foundation for the palladium-nickel top coat and reduce the wear of the precious metal during sliding of the contacting surfaces.

X5.5 For all of these purposes, the nickel underplating must be intact, that is, not cracked, and must have sufficient thickness to achieve the particular function for which it was intended. As a general rule, the minimum thickness should be 1.3 μm (50 μin.), preferably greater. For some levelling purposes, a greater thickness may be required.

X5.6 The use of nickel also reduces the potential for copper dissolution by protecting copper based substrates against chemical attack. Some palladium-nickel electroplating solutions are prone to attacking copper and subsequent codeposition of copper impurities.

X6. RESIDUAL SALTS

X6.1 Electroplated parts are placed in water of known conductivity and agitated for a specific time. The conductivity of the water extract is measured and the increase in conductivity due to residual salts and other conducting impurities is calculated. A suggested water extract conductivity test method uses a procedure in accordance with Test Methods D 1125, Method A.

X6.2 Conductivity of water for extract test shall be 1 μS/cm or less (resistivity 1 MΩ·cm or more).

X6.2.1 A sample of the coated parts having a total surface area of 30 cm² shall ordinarily be used and extracted in 100 cm³ of equilibrated water. To prepare equilibrated water, fill a clean polyethylene bottle half-way with high-purity water (X6.1), replace the bottle cap and shake the bottle vigorously for 2 min to equilibrate the water with the CO₂ in the air. CO₂ is a component of air, is soluble in water, and forms carbonic acid, which ionizes and is at equilibrium at 0.8 μS/cm. Slowly

agitate the solution for 10 min before determining the conductivity of the extract. In a closed polyethylene bottle, the equilibrated water will remain in the range from 0.8 to 1 μS/cm for at least 1 week.

X6.3 Inspection under a source of ultraviolet light is often employed to determine whether electroplating salts have been removed by the rinsing following gold electroplating. The presence of salts is evidenced by a characteristic fluorescence and should not be confused with fluorescing dirt or dirt particles.

X6.4 Water or purging stains, resulting from blind holes or from parts that were assembled before electroplating, as normally obtained in good commercial practice, are permissible except where they occur on surfaces to which electrical contact is to be made or on which subsequent soldering operations are performed.

X7. PROCEDURE FOR ALLOY COMPOSITION ANALYSIS BY SCANNING ELECTRON MICROSCOPY/X-RAY ENERGY DISPERSIVE SPECTROSCOPY

X7.1 The composition of electrodeposited palladium-nickel alloy coatings containing 70 to 95 mass % Pd that are ≥1.5 μm can be analyzed to a precision of ±0.2 mass % by the procedure outlined below:

X7.1.1 Obtain two EPMA certified composition standards that bracket the desired range of palladium-nickel alloy compositions (see 9.2.4).

X7.1.2 Make three EDS analyses of mass percent palladium in the certified region of each standard under the conditions cited below:

Area Analyzed: 200 μm × 200 μm (1000× magnification)
Accelerating Voltage: 20 kV

Working Distance: Optimum for the microscope
Tilt Angle: 0°
Count Time: 100 s
Beryllium Window: In

X7.1.3 The sample should be moved slightly between readings and obvious coating defects, such as pores or mechanical damage, should be avoided.

X7.1.4 Average the three EDS composition analyses on each standard and calculate a correction factor (*CF*) for each composition standard from the equation below:

$$(CF) = \frac{(\text{Certified Mass \% Pd for Standard})}{(\text{Average Measured Mass \% Pd on Day } X)} \quad (X7.1)$$



X7.1.5 Calculate the average correction factor, $(CF)_{ave}$ for the two standards.

X7.1.6 Make three EDS mass percent palladium composition analyses on the unknown coating samples according to the procedure outlined in X7.1.2.

X7.1.7 Multiply the average EDS mass percent palladium for the unknown sample times $(CF)_{ave}$ for day X to obtain the correct alloy composition.

X7.1.8 The standard deviation for the $(CF)_{ave}$ should be ≤ 0.005 based on measurements made on a large number of different commercial SEM/EDS instruments. If the standard deviation for the $(CF)_{ave}$ is ≥ 0.010 , the procedure described

above is not satisfactory and Reference 13 should be consulted concerning possible alternatives.

X7.2 For palladium-nickel alloy coatings that are 1.0 μm thick and deposited directly onto a 1.5 μm thick coating of pure nickel, the palladium content measured by this technique will be about 0.3 to 1.0 mass % less than the actual coating composition. For palladium-nickel alloy coatings that are 0.75 μm thick and deposited directly onto a 1.5 μm thick coating of pure nickel, the palladium content measured by this technique will be about 1 to 2 mass % less than the actual coating composition.

X8. ELECTRON PROBE X-RAY MICROANALYSIS (EPMA)

X8.1 The EPMA can be used to measure composition of palladium-nickel alloy coatings on top of any undercoat or any substrate if the thickness of the coating is $\geq 1.5 \mu\text{m}$ and the accelerating voltage for the electrons is in the range of 16 to 22 kV (an accelerating voltage of 20 kV is preferred). Under these conditions, there will be no significant excitation of characteristic X-rays from elements in substrates or undercoatings (12).

X8.2 The composition of palladium-nickel alloy coatings can be accurately analyzed by EPMA to a precision of ± 0.1

mass % using an X-ray WDS and a computer program developed by NIST that corrects for background, absorption, secondary fluorescence, and atomic number effects (12, 13). This technique is suitable for the certification of alloy standards for composition analyses by other techniques based on excitation of X-ray and electron radiations such as those cited in 9.2.5 and 9.2.6. The recommended palladium-nickel alloy coating thickness for an EPMA certified standard is $\geq 2.3 \mu\text{m}$.

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