



Designation: B456 – 17

Standard Specification for Electrodeposited Coatings of Copper Plus Nickel Plus Chromium and Nickel Plus Chromium¹

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

1. Scope

1.1 This specification covers requirements for several types and grades of electrodeposited copper plus nickel plus chromium or nickel plus chromium coatings on steel, nickel plus chromium coatings on copper and copper alloys, nickel plus chromium coatings on Type 300 and 400 series stainless steel and copper plus nickel plus chromium coatings on aluminum and its alloys and zinc alloys for applications where both appearance and protection of the basis metal against corrosion are important. Five grades of coatings are provided to correspond with the service conditions under which each is expected to provide satisfactory performance: namely, extended very severe, very severe, severe, moderate, and mild. Definitions and typical examples of these service conditions are provided in [Appendix X1](#).

1.2 This specification does not cover the requirements for the plating on plastics, see Specification [B604](#).

1.3 The following hazard caveat pertains only to the test methods portions, [Appendix X2](#), [Appendix X3](#), [Appendix X4](#), and [Appendix X5](#) of this specification: *This standard does not purport to address all of 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.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

¹ This specification is under the jurisdiction of ASTM Committee [B08](#) on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee [B08.05](#) on Decorative Coatings.

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2. Referenced Documents

2.1 *ASTM Standards*:²

- [B183 Practice for Preparation of Low-Carbon Steel for Electroplating](#)
- [B242 Guide for Preparation of High-Carbon Steel for Electroplating](#)
- [B252 Guide for Preparation of Zinc Alloy Die Castings for Electroplating and Conversion Coatings](#)
- [B253 Guide for Preparation of Aluminum Alloys for Electroplating](#)
- [B254 Practice for Preparation of and Electroplating on Stainless Steel](#)
- [B281 Practice for Preparation of Copper and Copper-Base Alloys for Electroplating and Conversion Coatings](#)
- [B320 Practice for Preparation of Iron Castings for Electroplating](#)
- [B368 Test Method for Copper-Accelerated Acetic Acid-Salt Spray \(Fog\) Testing \(CASS Test\)](#)
- [B380 Test Method for Corrosion Testing of Decorative Electrodeposited Coatings by the Corrodokote Procedure](#)
- [B487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of Cross Section](#)
- [B489 Practice for Bend Test for Ductility of Electrodeposited and Autocatalytically Deposited Metal Coatings on Metals](#)
- [B490 Practice for Micrometer Bend Test for Ductility of Electrodeposits](#)
- [B499 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals](#)
- [B504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method](#)
- [B530 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Electrodeposited Nickel Coatings on Magnetic and Nonmagnetic Substrates](#)

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

- B537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure
 - B568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry
 - B571 Practice for Qualitative Adhesion Testing of Metallic Coatings
 - B602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings
 - B604 Specification for Decorative Electroplated Coatings of Copper Plus Nickel Plus Chromium on Plastics
 - B659 Guide for Measuring Thickness of Metallic and Inorganic Coatings
 - B697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings
 - B762 Test Method of Variables Sampling of Metallic and Inorganic Coatings
 - B764 Test Method for Simultaneous Thickness and Electrode Potential Determination of Individual Layers in Multilayer Nickel Deposit (STEP Test)
 - B995 Test Method for Chloride Resistance Test for Chromium Electroplated Parts (Russian Mud Test)
 - D1193 Specification for Reagent Water
 - D3951 Practice for Commercial Packaging
 - E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
 - G85 Practice for Modified Salt Spray (Fog) Testing
- 2.2 *ISO Standards:*
- ISO 1456 Metallic coatings—Electrodeposited coatings of nickel plus chromium and of copper plus nickel plus chromium³

3. Terminology

3.1 Definitions:

3.1.1 *significant surfaces*—those surfaces normally visible (directly or by reflection) that are essential to the appearance or serviceability of the article, or both, when assembled in normal position; or that can be the source of corrosion products that deface visible surfaces on the assembled article. When necessary, the significant surfaces shall be specified by the purchaser and shall be indicated on the drawings of the parts, or by the provision of suitably marked samples.

3.1.2 *p-points*—specific points of measurement that are encouraged to be determined and agreed upon with the customer early in the contract review process. These are used for measurement of critical characteristics that vary with current density such as thickness, STEP, active sites, etc. and may be designated at multiple locations per part.

4. Classification

4.1 Five grades of coatings designated by service condition numbers and several types of coatings defined by classification numbers are covered by this specification.

4.2 Service Condition Number:

4.2.1 The service condition number indicates the severity of exposure for which the grade of coating is intended:

- SC 5 extended severe service
- SC 4 very severe service,
- SC 3 severe service,
- SC 2 moderate service, and
- SC 1 mild service.

4.2.2 Typical service conditions for which the various service condition numbers are appropriate are given in **Appendix X1**.

4.3 *Coating Classification Number*—The coating classification number comprises:

4.3.1 The chemical symbol for the basis metal (or for the principal metal if an alloy) followed by a slash mark, except in the case of stainless steel. In this case, the designation shall be SS followed by the designated AISI number followed by a slash, that is, SS463/.

4.3.2 The chemical symbol for copper (Cu) (if copper is used),

4.3.3 A number indicating the minimum thickness of the copper coating in micrometers (if copper is used),

4.3.4 A lower-case letter designating the type of copper deposit (if copper is used) (see 4.4 and 6.2.3),

4.3.5 The chemical symbol for nickel (Ni),

4.3.6 A number indicating the minimum thickness of the nickel coating, in micrometers,

4.3.7 A lower-case letter designating the type of nickel deposit (see 4.4 and 6.2.4),

4.3.8 The chemical symbol for chromium (Cr), and

4.3.9 A letter (or letters) designating the type of chromium deposit and its minimum thickness in micrometers (see 4.4 and 6.2.5).

4.4 *Symbols for Expressing Classification*—The following lower-case letters shall be used in coating classification numbers to describe the types of coatings:

- a —ductile copper deposited from acid-type baths
- b —single-layer nickel deposited in the fully-bright condition
- d —double- or triple-layer nickel coatings
- r —regular (that is, conventional) chromium
- mc —microcracked chromium
- mp —microporous chromium

4.5 *Example of Complete Classification Numbers*—A coating on steel comprising 15 μm minimum (ductile acid) copper plus 25 μm minimum (duplex) nickel plus 0.25 μm minimum (micro-cracked) chromium has the classification number: Fe/Cu15aNi25d Cr mc (see 4.3 and 6.2 for explanation of symbols).

5. Ordering Information

5.1 When ordering articles to be electroplated in conformance with this standard, the purchaser shall state the following:

5.1.1 The ASTM designation number of this standard.

5.1.2 Either the classification number of the specific coating required (see 4.3) or the substrate material and the service condition number denoting the severity of the conditions it is required to withstand (see 4.2). If the service condition number is quoted and not the classification number, the manufacturer is free to supply any of the types of coatings designated by the

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

classification numbers corresponding to the specified service condition number, as given in [Table 1](#), [Table 2](#), [Table 3](#), [Table 4](#), or [Table 5](#). On request, the manufacturer shall inform the purchaser of the classification number of the coating applied.

5.1.3 The appearance required, for example, bright, dull, or satin. Alternatively, samples showing the required finish or range of finish shall be supplied or approved by the purchaser.

5.1.4 The significant surfaces, to be indicated on drawings of the parts, or by the provision of suitably marked specimens (see [3.1](#)).

5.1.5 The positions on significant surfaces for rack or contact marks, where such marks are unavoidable (see [6.1.1](#)).

5.1.6 The extent to which defects shall be tolerated on nonsignificant surfaces.

5.1.7 The elongation of copper if other than the standard value (see [6.4](#)).

5.1.8 The ductility of the nickel if other than the standard value (see [6.5](#)).

5.1.9 The extent of tolerable surface deterioration after corrosion testing (see [6.8.3](#)).

5.1.10 Sampling methods and acceptance levels (see [Section 7](#)).

5.1.11 The minimum and maximum values of the electrode potential differences between individual nickel layers as measured in accordance with Test Method [B764](#) within the limits given in [6.9](#).

5.1.12 *Adhesion Test*—The adhesion test to be used (see [6.3](#)).

6. Product Requirements

6.1 Visual Defects:

6.1.1 The significant surfaces of the electroplated article shall be free of clearly visible plating defects, such as blisters, pits, roughness, cracks, and uncoated areas and shall not be stained or discolored. On articles where a visible contact mark is unavoidable, its position shall be agreed upon by the purchaser and the plater. The electroplated article shall be clean and free of damage.

TABLE 1 Nickel Plus Chromium Coatings on Steel

NOTE 1—When permitted by the purchaser, copper may be used as an undercoat for nickel but is not substitutable for any part of the nickel thickness specified. If the use of copper is permitted, [Table 2](#) may be used to obtain the same service conditions.

NOTE 2—Satin nickel may replace or be deposited over the bright nickel layer per supplier recommendations.

NOTE 3—Substrate condition can have a significant impact on corrosion performance.

Service Condition No.	Classification No.	Nickel Thickness, μm
SC 5	Fe/Ni35d Cr mc	35
	Fe/Ni35d Cr mp	35
SC 4	Fe/Ni30d Cr mc	30
	Fe/Ni30d Cr mp	30
SC 3	Fe/Ni25d Cr mc	25
	Fe/Ni25d Cr mp	25
SC 2	Fe/Ni20b Cr r	20
	Fe/Ni15b Cr mc	15
SC 1	Fe/Ni15b Cr mp	15
	Fe/Ni10b Cr r	10

TABLE 2 Copper Plus Nickel Plus Chromium Coatings on Steel

Service Condition No.	Classification No.	Nickel Thickness, μm
SC 5	Fe/Cu15a Ni30d Cr mc	30
	Fe/Cu15a Ni30d Cr mp	30
SC 4	Fe/Cu15a Ni25d Cr mc	25
	Fe/Cu15a Ni25d Cr mp	25
SC 3	Fe/Cu12a Ni20d Cr mc	20
	Fe/Cu12a Ni20d Cr mp	20

TABLE 3 Copper Plus Nickel Plus Chromium Coatings on Zinc Alloy

Service Condition No.	Classification No.	Nickel Thickness, μm
SC 5	Zn/Cu5 Ni35d Cr mc	35
	Zn/Cu5 Ni35d Cr mp	35
SC 4	Zn/Cu5 Ni30d Cr mc	30
	Zn/Cu5 Ni30d Cr mp	30
SC 3	Zn/Cu5 Ni20d Cr mc	20
	Zn/Cu5 Ni20d Cr mp	20
SC 2	Zn/Cu5 Ni20b Cr r	20
	Zn/Cu5 Ni15b Cr mc	15
SC 1	Zn/Cu5 Ni15b Cr mp	15
	Zn/Cu5 Ni10b Cr r	10

TABLE 4 Nickel Plus Chromium Coatings on Copper or Copper Alloy

Service Condition No.	Classification No.	Nickel Thickness, μm
SC 4	Cu/Ni25d Cr mc	25
	Cu/Ni25d Cr mp	25
SC 3	Cu/Ni20d Cr mc	20
	Cu/Ni20d Cr mp	20
	Cu/Ni30b Cr r	30
	Cu/Ni25b Cr mc	25
SC 2	Cu/Ni25b Cr mp	25
	Cu/Ni15b Cr r	15
	Cu/Ni10b Cr mc	10
SC 1	Cu/Ni10b Cr mp	10
	Cu/Ni5b Cr r	5

6.1.2 Defects in the surface of the basis metal, such as scratches, porosity, nonconducting inclusions, roll and die marks, cold shuts, weld imperfections, and cracks, may adversely affect the appearance and the performance of coatings applied thereto despite the observance of the best electroplating practices. Accordingly, the plater's responsibility for defects in the coating resulting from such conditions shall be waived.

NOTE 1—To minimize problems of this type, the specifications covering the basis material or the item to be electroplated should contain appropriate limitations on such basis metal conditions. Furthermore, areas such as welds may be excluded from certain performance criteria based upon mutual agreement of purchaser and supplier.

6.2 Process and Coating Requirements:

6.2.1 Proper preparatory procedures and thorough cleaning of the basis metal surface are essential for satisfactory adhesion and corrosion performance of the coating. Accordingly, the applicable practices for the preparation of various basis metals for electroplating shall be followed. Practices [B183](#), [B242](#), [B252](#), [B281](#), and [B320](#) are examples of practices that may be used for the preparation of basis metals.

6.2.2 Following the preparatory operations, the parts (articles) to be electroplated are introduced in such plating baths

TABLE 5 Nickel Plus Chromium^A on Stainless Steels, AISI Designated Type 300 and 400 Series,^B and Copper Plus Nickel Plus Chromium on Aluminum and Its Alloys

NOTE 1—Before nickel-chromium plating, the stainless steel surface and the aluminum substrate shall be prepared by a pretreatment from Practice B254,^C Guide B253,^D or equivalent, which is agreed upon between the supplier and the user.

Service Condition No.	Classification No.	Nickel Thickness, μm
SC 4	SS-3XX ^E /Ni20b/Cr mp	20
SC 4	SS-4xx ^E /Ni25b/Cr mp	25
SC 5	Al/Cu15a/Ni40d/Cr mp	40

^A Data in Table 5 were obtained using only microporous chromium systems. No data were available for the use of standard or microcracked systems.

^B The stainless steel alloy numbers used in this specification are based on the AISI system. They may not be interchangeable with other numbering systems such as the United Numbering System (UNS) or foreign designations.

^C Preplate for stainless steel substrates.

^D Preplate for aluminum substrates.

^E Insert AISI number for specific 300 or 400 alloy.

as required to produce the types of deposits described by the specific coating classification numbers or one of the coating classification numbers listed in Table 1, Table 2, Table 3, Table 4, or Table 5 appropriate for the specified service condition number.

6.2.3 Type of Copper and Deposit Thickness:

6.2.3.1 *Type of Copper*—The type of copper is designated by the following symbols that are placed after the thickness value:

a for ductile copper deposited from acid-type baths containing additives that promote leveling by the copper deposit and that have an elongation not less than 8 %.

No symbol is placed after the thickness value if a minimum elongation is not required or if a deposit from a non-leveling bath is permitted.

6.2.3.2 *Thickness of Copper Deposits*—The number following the chemical symbol for copper (Cu) indicates in micrometers the minimum thickness of the copper deposit at points on significant surfaces (see 3.1).

6.2.4 Type of Nickel and Deposit Thickness:

6.2.4.1 *Type of Nickel*—The type of nickel is designated by the following symbols, which are placed after the thickness value:

b for nickel deposited in the fully bright condition.

d for a double-layer or triple-layer nickel coating.

The bottom layer of this coating system shall contain less than 0.005 mass % sulfur (Note 3), and a minimum ductility of 67 % (see Practice B490). The top layer of this system shall contain more than 0.04 mass % sulfur (Note 3 and Note 4), and

TABLE 6 Summary of the Requirements for Double- and Triple-Layer Nickel Coatings

Type of Nickel Layer	Ductility	Sulfur Content	Double Layer	Triple Layer
Bottom	67 %	<0.005 mass %	60 to 80 %	50 to 70 %
Middle (high-sulfur)	...	>0.15 mass %	...	≤10 %
Top (bright)	11 %	>0.04 mass %	20 to 40 % (see Note 2 ¹)	≥20 %
Test Method	See B490	See Note 3 ¹	See Note 4 ¹	See Note 4 ¹

¹For Note 3 and Note 4, see Section 6.

have a minimum ductility of 11 %. If there are three layers, the intermediate layer shall contain not less than 0.15 mass % sulfur. These requirements for multilayer nickel coatings are summarized in Table 6.

NOTE 2—The percentages listed in Table 6 are intended to be a percent of the minimum thickness required for a particular service condition. Therefore, the overall ratio of the nickel layers may not be consistent with these values, but the minimum amount of nickel for each layer will be present. As an example, a double layer application requiring 35 μm of total nickel should have a minimum of 21 μm (60 %) of semi-bright and 14 μm (40 %) of bright. Additional bright nickel may be added beyond the minimum amount for cosmetic purposes, which will alter the final ratio of the two nickel layers but will still meet the minimum thickness requirements.

NOTE 3—The sulfur contents are specified in order to indicate which type of nickel electroplating solution must be used. Although at present, no simple method is available for determining the sulfur content of a nickel deposit on a coated article, chemical determinations are possible using specially prepared test specimens (see Appendix X3). The correct sulfur content has a significant effect on the corrosion performance of the nickel deposits.

NOTE 4—It will usually be possible to identify the type of nickel by microscopical examination of the polished and etched section of an article prepared in accordance with Test Method B487. The thickness of the individual nickel layers in double-layer and triple-layer coatings, as well as the electrochemical relationships between the individual layers, can also be measured by the STEP Test,⁴ in accordance with Test Method B764.

6.2.4.2 *Thickness of Nickel Deposit*—The number following the chemical symbol Ni indicates, in micrometers, the minimum thickness of the nickel electrodeposit at points on the significant surface (see 3.1 and Note 5).

6.2.5 Type of Chromium and Deposit Thickness:

6.2.5.1 *Type of Chromium*—The type of chromium deposit is designated by the following symbols placed after the chemical symbol Cr:

r for “regular” (that is, conventional) chromium.

mc for microcracked chromium, having more than 30 cracks/mm in any direction (Appendix X4) over the whole of the significant surface. The cracks shall be invisible to the unaided eye (see 6.11).

mp for microporous chromium containing a minimum of 10 000 pores/10 by 10 mm² (10 000/cm²) using the Dubpernell method (Appendix X4), or a minimum of 2000 pores/10 by 10 mm² (2000 pores/cm²) using the active site method (Appendix X5). The pores shall be invisible to the unaided eye (see 6.11).

6.2.5.2 A specially formulated noble nickel in between the bright nickel and the chromium deposits (see 6.9.4) may be used to induce micropores or microcracks in the chromium deposits. The thickness of this layer is recommended to be 2 to 4 μm minimum thickness. Controlled particle impingement of the plated standard chromium deposit may also be used to induce microporous chromium. Trivalent chromium deposits, as plated, may be microporous, microcracked, or both.

6.2.5.3 *Thickness of Chromium Deposit*—The minimum thickness of the chromium deposit shall be 0.2 μm on significant surfaces (see 3.1), except that for service condition SC 1

⁴ Harbulak, E. P., “Simultaneous Thickness and Electrochemical Potential Determination of Individual Layers in Multilayer Nickel Deposits,” *Plating and Surface Finishing*, Vol 67, No. 49, February 1980.

(see 4.2.1) the minimum thickness may be reduced to 0.13 μm . The thickness of chromium is designated by the same symbol as the type instead of by numerals as in the case of copper and nickel.

NOTE 5—Electroplated chromium deposits consist mainly of chromium metal with chromium oxides and other compounds. Hexavalent chromium ions would only be present if the surface of the part is not thoroughly rinsed. Rinsing is essential to meet regulations banning the presence of hexavalent chromium ions on the part.

total thickness of the nickel, and the thickness of the copper. The STEP test, Test Method B764, which is similar to the coulometric method, may be used to closely estimate the thicknesses of individual layers of nickel in a multilayer coating.

6.7.3.2 The microscopical method described in Test Method B487 may be used to measure the thickness of each nickel layer and of the copper layer. In cases where thickness

TABLE 7 Corrosion Tests Appropriate for Each Service Condition Number

Basis Metals	Service Condition No.	Corrosion Test and Duration h		
		CASS Method B368	Corrodokote Method B380	Acetic-salt Method G85
Steel, zinc alloy, or copper and copper alloy, stainless steel and aluminum alloys	SC 5	66
	SC 4	22	Two 16-h cycles	144
	SC 3	16	16	96
	SC 2	8	4	24
	SC 1	8

6.2.5.4 When plating chromium over a nickel strike containing micro-particles used to induce microporosity in the subsequent chromium deposit, excess chromium thickness will bridge the nonconductive particles within the nickel layer. A maximum of 0.5 μm is recommended.

6.3 *Adhesion*—The coating shall be sufficiently adherent to the basis metal, and the separate layers of multilayer coatings shall be sufficiently adherent to each other, to pass the appropriate tests detailed in Test Methods B571. The particular test or tests to be used shall be specified by the purchaser.

6.4 *Elongation*—The elongation of copper shall be such that it will not be less than stated in 6.2.3.1 when tested by the method given in Appendix X2. Greater elongation may be requested but shall be subject to agreement between the purchaser and the manufacturer.

6.5 *Ductility*—The ductility of the composite nickel deposit on a finished part is considered acceptable when foils plated out of the individual nickel processes meet or exceed the values listed in Table 6. See test details in Test Method B490.

6.6 *p-points*—See 3.1.2.

6.7 *Coating Thickness:*

6.7.1 The minimum coating thickness shall be as designated by the coating classification number.

6.7.2 It is recognized that requirements may exist for thicker coatings than are covered by this specification (see Note 2).

6.7.3 The thickness of a coating and its various layers shall be measured at points on the significant surfaces (See Section 3 and Note 6).

NOTE 6—When significant surfaces are involved on which the specified thickness of deposit cannot readily be controlled, such as threads, holes, deep recesses, bases of angles, and similar areas, the purchaser and the manufacturer should recognize the necessity for either thicker deposits on the more accessible surfaces or for special racking. Special racks may involve the use of conforming, auxiliary, or bipolar electrodes or nonconducting shields.

6.7.3.1 The coulometric method described in Test Method B504 may be used to measure thickness of the chromium, the

measurements conflict, microscopical will be the prevailing method.

6.7.3.3 The X-ray method described in Test Method B568 may be used to measure thickness of the chromium, thickness of a single layer nickel as well as the thickness of copper. In the case of duplex/triple nickel coatings, the X-ray method will give a total nickel thickness reading based on the average density of the individual nickel coatings.

6.7.3.4 Other methods may be used if it can be demonstrated that the uncertainty of the measurement is less than 10 %, or less than that of any applicable method mentioned in 6.7.3. Other methods such as B499 and B530, as outlined in Guide B659, may be used if agreed upon between the purchaser and manufacturer.

6.8 *Corrosion Testing:*

6.8.1 Coated articles shall be subjected to the corrosion test for a period of time that is appropriate for the particular service condition number (or for the service condition number corresponding to a specified classification number) as shown in Table 7. The test is described in detail in the referenced ASTM designation.

NOTE 7—There is no direct relation between the results of an accelerated corrosion test and the resistance to corrosion in other media, because several factors, such as the formation of protective films, influence the progress of corrosion and vary greatly with the conditions encountered. The results obtained in the test should, therefore, not be regarded as a direct guide to the corrosion resistance of the tested materials in all environments where these materials may be used. Also, performance of different materials in the test cannot always be taken as a direct guide to the relative resistance of these materials in service.

6.8.2 After the article has been subjected to the treatment described in the relevant corrosion test method, it shall be examined for corrosion of the basis metal or blistering of the coating. Any basis metal corrosion or blistering of the coating shall be cause for rejection. It is to be understood that occasional widely scattered, small corrosion defects such as surface pits may be observed after the testing period. In general, “acceptable resistance” shall mean that such defects are not, when viewed critically, significantly defacing or

otherwise deleterious to the function of the electroplated part. A method of rating corrosion is given in Practice **B537**.

NOTE 8—In environments where road salts such as calcium chloride are used, a specific type of corrosion has been observed. **B995** (Russian Mud Test) simulates this type of surface corrosion between the top nickel and chromium deposits. No correlation has yet been established between the test results and actual service performance. The number of hours the test is conducted and the results shall be agreed upon between the purchaser and supplier.

6.8.3 Surface deterioration of the coating itself is expected to occur during the testing of some types of coatings. The extent to which such surface deterioration will be tolerated shall be specified by the purchaser.

6.9 STEP Test Requirements:

6.9.1 The electrode potential differences between individual nickel layers shall be measured for multilayer coatings corresponding to SC5, SC4, and SC3 in accordance with Test Method **B764** (STEP Test).

6.9.2 The STEP potential difference between the semi-bright nickel layer and the bright nickel layer has an accepted range of 100 to 200 mV. For all combinations of nickel layers, the semi-bright nickel layer is more noble (cathodic) than the bright nickel layer. See **Note 9**.

NOTE 9—For optimum balance between cosmetics and corrosion protection, the STEP is recommended to be between 120 and 160 mV.

6.9.3 The STEP potential difference between the high-activity nickel layer and the bright nickel layer in triple-layer coatings has an accepted potential range of 15 to 45 mV. The high-activity nickel layer is more active (anodic) than the bright nickel layer.

6.9.4 The STEP potential difference between the bright nickel layer and a nickel (noble nickel) layer between the bright nickel layer and the chromium layer has an accepted potential range of 10 to 90 mV. The bright nickel layer is more active (anodic) than the particle nickel layer prior to chromium. See **Note 10**.

NOTE 10—For optimum balance between cosmetics and corrosion protection, the STEP is recommended to be at the high end of this range.

6.10 Sulfur Content:

6.10.1 The sulfur content of the nickel deposit shall meet the maximum or minimum values as stated in **6.2.4.1** and **Table 6**.

6.10.2 A method to determine sulfur is presented in **Appendix X3**. Any reliable method may be used.

6.11 Density and Measurement of the Discontinuities in Chromium:

6.11.1 The density of cracks or pores in microcracked or microporous chromium deposits shall meet minimum values. Microcracked chromium shall have more than 30 cracks/mm (300 cracks/cm) in any direction over the whole of the significant surface. Microporous chromium shall contain a minimum of 10 000 pores/10 by 10 mm² (10 000 pores/cm²) using the Dubpernell method, or a minimum of 2000 pores/10 by 10 mm² (2000 pores/cm²) using the active site method. Cracks and pores can be measured in several locations over the whole of the significant surface and shall be invisible to the unaided eye.

6.11.2 A method for measuring the discontinuities, referred to as Dubpernell sites, is given in **Appendix X4**. A method for measuring the number of corrosion sites formed during corrosion, referred to as active sites, is given in **Appendix X5**.

7. Sampling Requirements

7.1 The sampling plan used for the inspection of a quantity of coated articles shall be as agreed upon by the purchaser and supplier.

NOTE 11—Usually, when a collection of coated articles, the inspection lot, is examined for compliance with the requirements placed on the coating, 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. Three standards, Test Method **B602**, Guide **B697**, and Method **B762** contain sampling plans that are designed for the sampling inspection of coatings.

Test Method **B602** 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 **B602** identifies the plan to use.

Guide **B697** provides a large number of plans and also gives guidance on the selection of a plan. When Guide **B697** is specified, the buyer and seller need to agree on the plan to be used.

Method **B762** 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 **B762** 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, Test Method **B762** identifies the plan to be used.

NOTE 12—When both destructive and nondestructive tests exist for the measurement of a characteristic, the purchaser needs to state which is to be used so the proper sampling plan is selected. A test may destroy the coating but in a noncritical area; or, although it may destroy the coating, a tested part may be reclaimed by stripping and recoating. The purchaser needs to state whether the test is to be considered destructive or nondestructive.

7.2 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, that have been coated by a single supplier at one time or approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

7.3 If separate test specimens are used to represent the coated articles in a test, the specimens shall be of the nature, size, and number and be processed as required in **Appendix X2**, **Appendix X3**, **Appendix X4**, and **Appendix X5**. Unless a need can be demonstrated, separately prepared specimens shall not be used in place of production items for nondestructive tests and visual examination. For destructive tests including determination of adhesion, ductility, sulfur contents, the number of discontinuities, and corrosion testing, separately prepared specimens may be used.

8. Packaging

8.1 Parts plated for the U.S. Government and military, including subcontracts, shall be packaged in accordance with Practice **D3951**.

9. Keywords

9.1 corrosion; decorative; electrodeposited chromium; electrodeposited copper; electrodeposited nickel

APPENDIXES

(Nonmandatory Information)

X1. DEFINITIONS AND EXAMPLES OF SERVICE CONDITIONS FOR WHICH THE VARIOUS SERVICE CONDITION NUMBERS ARE APPROPRIATE

X1.1 *Service Condition No. SC 5 (Extended Very Severe)*—Service conditions that include likely damage from denting, scratching, and abrasive wear in addition to exposure to corrosive environments where *long-time protection* of the substrate is required; for example, conditions encountered by some exterior components of automobiles.

X1.2 *Condition No. SC 4 (Very Severe)*—Service conditions that include likely damage from denting, scratching, and abrasive wear in addition to exposure to corrosive environments; for example, conditions encountered by exterior components of automobiles and by boat fittings in salt water service.

X1.3 *Service Condition No. SC 3 (Severe)*—Exposure that is likely to include occasional or frequent wetting by rain or dew or possibly strong cleaners and saline solutions; for example, conditions encountered by porch and lawn furniture; bicycle and perambulator parts; hospital furniture and fixtures.

X1.4 *Service Condition No. SC 2 (Moderate)*—Exposure indoors in places where condensation of moisture may occur; for example, in kitchens and bathrooms.

X1.5 *Service Condition No. SC 1 (Mild)*—Exposure indoors in normally warm, dry atmospheres with coating subject to minimum wear or abrasion.

X2. ELONGATION TEST

NOTE X2.1—Practice B489 is used to ensure compliance of the type of copper deposit with the appropriate definition given in 6.4. Practice B489 should be followed with these conditions.

X2.1 *Preparation of Test Piece:*

X2.1.1 Prepare an electroplated test strip, 150 mm long, 10 mm wide, and 1 mm thick by the following method:

X2.1.1.1 Polish a sheet of the appropriate basis metal, similar to that of the articles being electroplated, except that if the basis metal is zinc alloy the sheet may be of soft brass. (Use a sheet sufficiently large to allow the test strip to be cut from its center after trimming off a border 25 mm wide all around.) Electroplate the polished side of the sheet with copper to a thickness of 25 μm under the same conditions and in the same bath as the corresponding articles.

X2.1.1.2 Cut the test strip from the electroplated sheet with a flat shear. Round or chamfer the longer edges of the strip, at least on the electroplated side, by careful filing or grinding.

X2.2 *Procedure*—Bend the test strip with the electroplated side in tension (on the outside), by steadily applying pressure, through 180° over a mandrel of 12 mm diameter until the two ends of the test strip are parallel. Ensure that contact between the test strip and the mandrel is maintained during bending.

X2.3 *Assessment*—The electroplating is deemed to comply with the minimum requirement of an elongation of 8 % if after testing there are no cracks passing completely across the convex surface. Small cracks at the edges do not signify failure.

X3. DETERMINATION OF SULFUR IN ELECTRODEPOSITED NICKEL (NOTE X3.1)

The following two methods for the determination of sulfur in electroplated nickel are given as guidelines for use to test compliance of the type of nickel deposit with the appropriate definition given in 6.2.4.1. They represent methods that have been used with success commercially; they are not ASTM standards, nor is it the intent in publishing these methods to preclude the use of other methods or variations in these methods.

X3.1 Total Sulfur in Electroplated Nickel by Combustion-Iodate Titration

X3.1.1 *Scope*—This method covers the determination of sulfur in concentrations from 0.005 to 0.5 mass %.

X3.1.2 *Summary of Method*—A major part of the sulfur in the sample is converted to sulfur dioxide (SO_2) by combustion in a stream of oxygen using an induction furnace. During the combustion, the SO_2 is absorbed in an acidified starch-iodide solution and titrated with potassium iodate solution. The latter is standardized against steels of known sulfur content to compensate for characteristics of a given apparatus and for day-to-day variation in the percentage of sulfur recovered as SO_2 . Compensation is made for the blank because of accelerators and crucibles.

NOTE X3.1—Instruments are available for measuring the sulfur dioxide from combustion by infrared detection methods and using built-in computers to integrate and display the sulfur content as a percentage. Some units also simultaneously measure the percentage of carbon.

X3.1.3 *Interferences*—The elements ordinarily present in electroplated nickel do not interfere.

X3.1.4 *Apparatus*—Induction heating apparatus for determination of sulfur by direct combustion as described in Practices E50 (Apparatus No. 13).

X3.1.5 Reagents:

X3.1.5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ Other grades may be used, provided it is first determined that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

X3.1.5.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D1193.

X3.1.5.3 *Hydrochloric Acid (3 + 97)*—Mix 3 volumes of concentrated hydrochloric acid (HCl) (sp gr 1.19) with 97 volumes of water.

X3.1.5.4 *Iron (Low-Sulfur) Accelerator*—Chips.

X3.1.5.5 *Iron (Low-Sulfur) Accelerator*—Powder.

X3.1.5.6 *Potassium Iodate, Standard Solution A* (1 mL = 0.1 mg S)—Dissolve 0.2225 g of potassium iodate (KIO_3) in 900 mL of water and dilute to 1 L.

X3.1.5.7 *Potassium Iodate, Standard Solution B* (1 mL = 0.02 mg S)—Transfer 200 mL of potassium iodate Solution A (1 mL = 0.1 mg S) to a 1-L volumetric flask, dilute to volume, and mix.

NOTE X3.2—The sulfur equivalent is based on the complete conversion of sulfur to sulfur dioxide. The recovery of sulfur as the dioxide may be less than 100 %, but it is consistent when the temperature and the rate of oxygen flow are maintained constant. An empirical factor must be determined by an analysis of a standard sample.

X3.1.5.8 *Starch-Iodide Solution*—Transfer 1 g of soluble or arrowroot starch to a small beaker, add 2 mL of water, and stir until a smooth paste is obtained. Pour the mixture into 50 mL of boiling water. Cool, add 1.5 g of potassium iodide (KI), stir until dissolved, and dilute to 100 mL.

X3.1.5.9 *Tin (low sulfur) Accelerator*, granular.

X3.1.6 *Standards*—Standards for calibration are National Institute of Standards and Technology steels of the proper sulfur content.

X3.1.7 Sample Preparation:

X3.1.7.1 Prepare a test panel of cold-rolled steel 150 mm long by 100 mm wide by 1 mm thick or any other convenient size. Clean, acid dip, and electroplate with approximately 7.5 μm of an adherent nickel deposit and thoroughly rinse. Buffed nickel or buffed stainless steel may also be used as alternatives to steel electroplated with nickel.

X3.1.7.2 Passivate the test panel anodically at 3 V for 5 to 10 s in a hot alkaline cleaner (temperature 70 to 80°C) containing 30 g/L of sodium hydroxide (NaOH) and 30 g/L of trisodium phosphate (Na_3PO_4) or 60 g/L of any other suitable anodic alkaline cleaner.

X3.1.7.3 Coat the passivated test panel with 25 to 37 μm of nickel deposited from the same solution using the same parameters as for the coated articles represented by the test specimen. Use a sufficient amount of solution so that additives do not have to be added during the test in order to maintain the nickel's properties.

NOTE X3.3—Plating the test panel along with production parts is a viable method. However, only the nickel being tested can be plated on the panel.

X3.1.7.4 Remove the edges of the electroplated panel with a hand or power shear or any other convenient method that permits ready separation of the test foil.

X3.1.7.5 Separation from the panel, wash the nickel foil electroplate with water to remove salts and blot dry. Cut into pieces 2 to 3 mm per side with a scissors. Transfer to a 100-mL beaker, cover with water, and heat to boiling. Pour off the water and wash with methanol. Air dry the nickel on filter paper.

X3.1.8 *Weight for Standards and Samples*—Select and weigh to the nearest 0.1 mg an amount of sample as follows:

⁵ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

Expected Sulfur Content, mass %	Weight of Sample, g
0.005 to 0.10	1.0 ± 0.02
0.10 to 0.50	0.2 ± 0.02

X3.1.9 *Calibration*—Select a minimum of two standards with sulfur contents near the high- and low-limits of the range for a given sample weight and also one near the mean. The mean standard may be simulated, if necessary, by taking one-half the sample weight of each of the other two. Follow the steps of the procedure.

X3.1.10 *Procedure:*

X3.1.10.1 To the crucible add 1 g of iron chips, 0.8 g of iron powder, and 0.9 g of tin. Transfer the proper weight of sample and cover.

X3.1.10.2 Turn on the power of the induction furnace and allow the unit to heat to operating temperature. With oxygen flowing through the absorption vessel, fill it to a predetermined point with HCl (3 + 97) (X3.1.5.3) (Note X3.4). Add 2 mL of starch solution to the vessel. With the oxygen flow adjusted to 1.0 to 1.5 L/min (Note X3.5), add KIO₃ solution specified until the intensity of the blue color is that which is considered as the end point. Refill the buret.

NOTE X3.4—Always fill the titration vessel to the same point.

NOTE X3.5—The oxygen flow rate may be adjusted to meet the requirements of individual operators or equipment; however, the flow rate must be the same for the test samples and the standard samples.

X3.1.10.3 After the unit has been at operating temperature for at least 45 s, place the covered crucible containing the sample and accelerators on the pedestal. With the oxygen flow adjusted, raise the crucible, close the furnace, and turn on the power. Burn the sample for 8 to 10 min. Titrate continuously with the KIO₃ solution at such a rate as to maintain as nearly as possible the original intensity of the blue color. The end point is reached when the original blue color is stable for 1 min. Record the final buret reading and drain the titration vessel through the exhaust stopcock.

X3.1.10.4 *Blank*—Determine the blank by placing the same amount of accelerators used in the test sample in a pre-ignited crucible. Cover and proceed as in X3.1.10.3.

X3.1.11 *Calculation*—Calculate the sulfur factor of the potassium iodate as follows:

$$\text{Sulfur factor, g/unit volume} = \frac{A \times B}{(C - D) \times 100} \quad (\text{X3.1})$$

where:

- A = grams of standard sample used,
- B = percent sulfur in the standard sample
- C = millilitres of KIO₃ solution required for titration of the standard sample (Note X3.6), and
- D = millilitres of KIO₃ solution required for titration of the blank (Note X3.6).

NOTE X3.6—Or apparent percentage of sulfur for “direct-reading” burets.

X3.1.11.1 Calculate the percentage of sulfur in the test sample as follows:

$$\text{Sulfur, mass \%} = \frac{(E - D)F}{G} \times 100 \quad (\text{X3.2})$$

where:

- E = KIO₃ solution required for titration of the test sample (Note X3.6), mL,
- D = KIO₃ solution required for titration of the blank, mL,
- F = average sulfur factor of the KIO₃ for the standards used (see X3.1.11), g/unit volume, and
- G = sample used, g.

X3.2 **Determination of Sulfur in Electroplated Nickel by the Evolution Method**

X3.2.1 *Scope*—This method covers the determination of sulfide sulfur in electroplated nickel in the range from 0.005 to 0.2 mass %.

X3.2.2 *Summary of Method*⁶—Sulfide sulfur is evolved as hydrogen sulfide (H₂S) on dissolving the sample of hydrochloric acid (HCl) containing a small amount of platinum as an accelerator for dissolution. The sulfur is precipitated as zinc sulfide (ZnS) in the receiving vessel and then titrated with standard potassium iodate solution. Values are based on potassium iodate (KIO₃) as the primary standard.

X3.2.3 *Apparatus:*

X3.2.3.1 The apparatus is shown in Fig. X3.1. It may be assembled using a 50-mL Erlenmeyer flask with a No. 19/38 outer joint. A wash bottle fitting with a No. 19/38 inner joint can be cut to fit the 50-mL flask. The exit tube can be bent and connected to the 6-mm gas tube with tubing.

X3.2.3.2 A nitrogen cylinder with valves and pressure regulator.

X3.2.3.3 Buret, 10-mL.

X3.2.4 *Reagents:*

⁶ Luke, C. L., *Analytical Chemistry*, Vol 29, 1957, p. 1227.

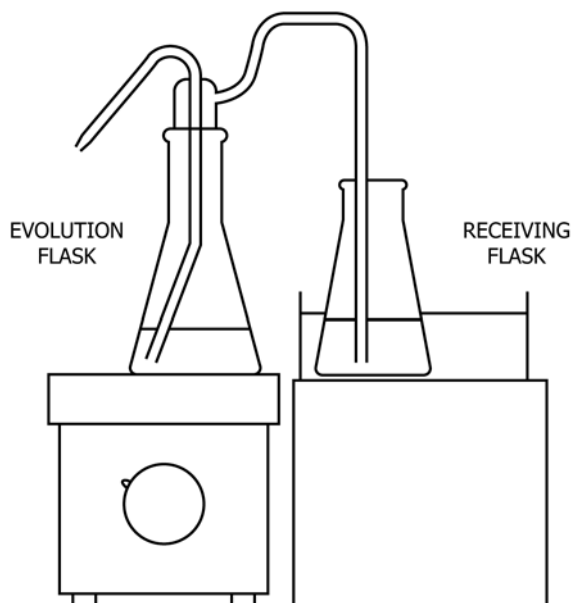


FIG. X3.1 Apparatus for the Determination of Sulfur in Electroplated Nickel Foil by the Evolution Method X 3.2

X3.2.4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

X3.2.4.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification **D1193**.

X3.2.4.3 *Ammoniacal Zinc Sulfate Solution*—Dissolve 50 g of zinc sulfate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) in 250 mL of water, add 250 mL of ammonium hydroxide (NH_4OH , sp gr 0.90) and mix. Transfer to a flask and allow to stand about 24 h and filter into a polyethylene bottle.

X3.2.4.4 *Hexachloroplatinic Acid Solution (10 g/L)*—Dissolve 0.5 g of hexachloroplatinic acid ($\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$) in about 40 mL of water, add 5 mL of hydrochloric acid (HCl sp gr 1.19), and dilute to 50 mL.

X3.2.4.5 *Hydrochloric Acid-Platinum Chloride Solution*—Prepare 500 mL of diluted hydrochloric acid (HCl sp gr 1.19 1 part acid in 1 part water). Add 2.5 mL of the hexachloroplatinic acid solution and mix.

X3.2.4.6 *Potassium Iodate, Standard Solution (0.1 N)*—Dry the crystals of potassium iodate (KIO_3) at 180°C for 1 h. Dissolve 3.570 g of the KIO_3 in about 200 mL of water, transfer to a 1-L volumetric flask, dilute to volume, and mix.

X3.2.4.7 *Potassium Iodate, Standard Solution (0.005 N)*—Transfer 25 mL of the 0.1 N KIO_3 solution to a 500-mL volumetric flask with a pipet, dilute to volume, and mix.

X3.2.4.8 *Starch Solution (10 g/L)-Potassium Iodide (50 g/L) Solution*—Add about 5 mL of water to 1 g of soluble starch with stirring until a paste is formed and add to 100 mL of boiling water. Cool, add 5 g of potassium iodide (KI), and stir until the KI is dissolved.

X3.2.5 *Sample Preparation*—Prepare sample as outlined in **X3.1.7**.

X3.2.6 *Weight of Sample*—Select and weigh to the nearest 0.1 mg an amount of sample as follows:

Expected Sulfur Content, mass %	Weight of Sample, g
0.005 to 0.07	1.0 ± 0.02
0.05 to 2	0.4 ± 0.02

X3.2.7 *Procedure*:

X3.2.7.1 Weigh the specified amount of sample to the nearest 0.1 mg and transfer to the 50-mL evolution flask.

X3.2.7.2 Add 20 mL of water and 3 mL of ammoniacal zinc sulfate solution to the receiving flask.

X3.2.7.3 Adjust the hot plate to maintain the temperature of 25 mL of water in a 50-mL Erlenmeyer flask at 80°C.

X3.2.7.4 Add 15 mL of the hydrochloric acid-hexachloroplatinic acid solution to the sample. Assemble the apparatus as shown in **Fig. X3.1** and start a very gentle stream of nitrogen through the system.

NOTE X3.7—A flow of about 30 cm³/min is satisfactory. If the sample dissolves rapidly, the flow should be decreased during the time hydrogen is freely liberated.

X3.2.7.5 Continue the heating and flow of nitrogen until the sample is completely dissolved, then continue for 5 min (**Note X3.7**). Separate the gas delivery tube from the evolution head and remove the receiving flask with the delivery tube.

NOTE X3.8—The solution in the receiving flask will remain alkaline throughout the dissolution period if the hot plate temperature and the nitrogen flow are properly adjusted. Additional ammoniacal zinc sulfate solution may be added, if necessary, but the sample should be discarded if the receiving solution becomes acidic (less than pH 7 by test paper).

X3.2.7.6 Add 1 mL of the starch-iodide solution and 5 mL of diluted HCl (1 + 1) and mix. Titrate immediately with standard potassium iodate from a 10-mL buret to the first blue color. Draw some of the solution into the delivery tube with a rubber bulb and release along the neck of the flask to wash down any adhering zinc sulfide. Swirl the solution to wash the outside of the tube. Continue the titration to a permanent blue color.

X3.2.7.7 Run a blank titration to the same starch-iodine color on a mixture of 20 mL of water, 3 mL of ammoniacal zinc sulfate, 1 mL of starch-iodate solution and 5 mL of diluted hydrochloric acid (1 part HCl sp gr 1.19 and 1 part water) in a 50-mL Erlenmeyer flask.

X3.2.8 *Calculations*—Calculate the mass percent of sulfide sulfur as follows:

$$\text{Sulfide sulfur, mass \%} = \frac{(A - B) \times 0.005 \times 0.016}{W} \times 100 \quad (\text{X3.3})$$

where:

- A = 0.005 N KIO_3 solution used for the sample titration, mL,
- B = 0.005 N KIO_3 solution used in the blank, mL, and
- W = sample used, g.

X4. DETERMINING THE NUMBER OF DISCONTINUITIES IN CHROMIUM ELECTROPLATING (DUBPERNELL TEST)

X4.1 *Principle of the Method*—Copper will be deposited on nickel exposed through discontinuities in chromium but not on the chromium, provided that potential is properly controlled (kept low enough to avoid activation of passive chromium).

X4.2 *Preparation of Test Piece:*

X4.2.1 Mask all edges not covered by the chromium with a nonconductive coating such as paint, wax, or pressure sensitive tape, including the wire used to make contact to the cathode bar.

X4.2.2 After masking, clean the specimen by soaking in a hot alkaline cleaner until the surface is free of water breaks. A mild scrubbing with a soft brush is helpful. Follow the cleaning by a thorough rinse in cold deionized water, then a dip in an approximately 5% by mass solution of H₂SO₄. Thoroughly rinse in deionized water and transfer wet into the copper solution using live entry.

NOTE X4.1—If the chromium deposit on the test piece is several days old (aged), immerse for approximately four min. in a solution of 15 ± 5% nitric acid at approximately 65 °C. Longer immersion times may be required to obtain a no-water-break surface after rinsing. An alternative activation for aged chromium is to scrub with magnesium oxide slurry until the surface has no water breaks after rinsing. Follow either approach with step X4.2.2.

X4.2.3 Make freshly cleaned sample anodic at 0.8V for 30 s in the bright acid copper plating bath with copper anodes (see Note X4.2), then switch to cathodic (see Fig. X4.1) at 3 A/dm² for 2 min (see Note X4.2). (**Warning**—Do not go beyond the specified anodic voltage or time because nickel will slowly dissolve or become passivated.)

NOTE X4.2—After cleaning, anodic treatment to repassivate chromium is essential. Plating time can be varied from 1 to 5 min. Two minutes has been found to be near optimum. With highly porous chromium, longer

times incur risk of merging the deposit nodules, giving rise to ambiguities in counting pores (nodules).

NOTE X4.3—Most bright acid copper formulations will work in this application. If desired, the following formulation can be used.

- Bath formulation
- CuSO₄·5H₂O 1 m (250 g/L)
- H₂SO₄ (SpG 1.84) 0.5 m (50 g/L)
- Temperature (20 to 25°C)
- Anode (copper)
- Live entry

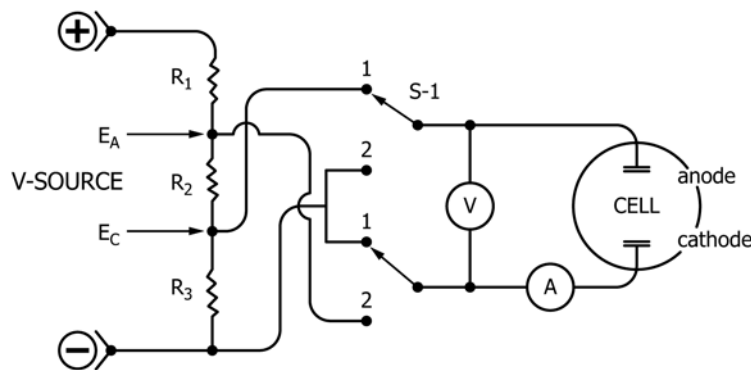
X4.2.4 Following copper electroplating, carefully remove the specimen, rinse in low-pressure cold then hot deionized water, and air dry. The specimen should not be wiped where pores or cracks are to be counted, nor should the part be force air dried. Drying can be accelerated by following the last water rinse by a rinse with alcohol (ethanol) or other volatile water miscible solvent.

X4.2.5 The copper deposits only on the underlying nickel that is exposed through discontinuities (pores and cracks) in the chromium.

X4.3 *Assessment:*

X4.3.1 The number of discontinuities in the chromium can be estimated by counting the copper nodules deposited within a known area of the specimen or the number of cracks in a known length. These determinations are facilitated with a metallurgical microscope fitted with a calibrated reticle in the eyepiece, or from photomicrographs taken of a representative field of the specimen. (See Appendix X5 for a guide to the determination of active corrosion sites in the chromium and underlying nickel layers.)

X4.3.2 Current measured or recorded during the cathodic cycle, or both, serves as a reliable indicator of porosity. If



$$E_A = \frac{R_2 + R_3}{R_1 + R_2 + R_3}$$

$$E_C = \frac{R_3}{R_1 + R_2 + R_3}$$

S - 1 = DPDT switch
position 1 - cathodic
position 2 - anodic

NOTE: Ammeter and voltmeter can be replaced by a 2 - channel strip chart recorder.

FIG. X4.1 Schematic Diagram of a Switching Apparatus to Conveniently Control Polarity and Voltage During Porosity Testing via Copper Deposition

current remains low ($<1 \text{ mA/cm}^2$) during the cathodic cycle, porosity is low. Rapidly rising current ($\Delta I/\Delta t \approx 1$ to 2 mA/min) and high (2 to 4 mA/cm^2) final current is indicative of high porosity. Use of a strip chart recorder provides a permanent record of the test current. A qualitative visual check (microscopically) is used to count the number of pores or cracks.

NOTE X4.4—100× magnification is specified in many standards requir-

ing the measuring of Dubpernell pore count.

X4.3.3 *Precautions*—High cathodic potentials can activate chromium locally, giving rise to spuriously high nodule counts. If this condition is suspected, it can be tested by gently wiping the copper off with a tissue. If copper adheres to specimen, it is probable the cathodic potential was too high, thus depositing copper on the chromium instead of just in the pores.

X5. DETERMINING THE NUMBER OF ACTIVE SITES AFTER CORROSION

X5.1 Principle of the Method—Some of the discontinuities determined in Appendix **Appendix X4** might not become corrosion sites. The sites that actually corrode are determined by stripping the chromium after corrosion and the surface pits in the nickel are counted and referred to as “active sites.”

X5.2 Before testing, the part should be cleaned to eliminate water breaks. Magnesium oxide, warm water and soap, or solvents, or a combination thereof, might be necessary for thorough cleaning.

X5.3 To develop the active corrosion sites, subject the part to the number of hours of CASS exposure corresponding to the corrosion specification for the required service condition and thickness specification. After CASS testing, rinse the part in warm water to remove the salt layer. If the part contains corrosive product staining, it can be washed with a very mild sponge in warm water but the part should not be subjected to any abrasive cleaning. The chromium deposit should be stripped and the part rinsed at this point. After drying either by hot air or solvent such as methanol, the part should be examined at a magnification between 100 and 200× and the active pore size and density determined. The corroding pore sites are typically distinctly larger than the uncorroded sites and have a darker and rougher texture. By means of images at

a known magnification, the active pore sites can be counted and the active sites per area calculated. Different images should be taken at the same magnification at all significant surfaces and current densities to record the differences in pore density and size after corrosion. As a guide, the average diameter of the active sites should not exceed 0.032 mm (0.00125 in.) and the largest individual site should not exceed 0.0635 mm (0.0025 in.). Comparisons with known pore density charts can be used for determination of the sites per square centimetre.

X5.4 Even though the necessary work has not been conducted to establish a correlation between active corrosion sites and starting sites as measured by Dubpernell, it appears that for a given current density and deposit system there is a correlation between the number of pores before corrosion and the number of active corrosion sites after CASS testing. Once this correlation has been established at a given installation, a simple 100 to 200 X viewing of a part might nondestructively indicate the service performance of the part after corrosion. The desired number of active corrosion sites has not been definitely established. This number depends somewhat on the size of the pores before and after corrosion, the thickness and potentials of the deposit system, and the desired trade-off between basis metal corrosion and staining in CASS.

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