

Designation: B 456 – 95

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 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, and copper plus nickel plus chromium coatings on 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 The following hazards caveat pertains only to the test methods portions, Appendix X2, Appendix X3, and Appendix X4, 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.*

Note 1—The ISO standards 1456 and 1457 are not requirements but can be referenced for additional information.

2. Referenced Documents

- 2.1 ASTM Standards:
- B 117 Practice for Operating Salt Spray (Fog) Testing Apparatus²
- B 183 Practice for Preparation of Low-Carbon Steel for Electroplating³
- B 242 Practice for Preparation of High-Carbon Steel for Electroplating³
- B 252 Guide for Preparation of Zinc Alloy Die Castings for Electroplating and Conversion Coatings³

- ² Annual Book of ASTM Standards, Vol 03.02.
- ³ Annual Book of ASTM Standards, Vol 02.05.

- B 253 Guide for Preparation of Aluminum Alloys 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 287 Method of Acetic Acid-Salt Spray (Fog) Testing⁴
- B 320 Practice for Preparation of Iron Castings for Electroplating³
- B 368 Method for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test)³
- B 380 Method of Corrosion Testing of Decorative Electrodeposited Coatings by the Corrodkote Procedure³
- B 487 Test Method for Measurement of Metal and Oxide Coating Thicknesses by Microscopical Examination of a Cross Section³
- B 499 Test Method for Measurement of Coating Thickness by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals³
- B 504 Test Method for Measurement of Thickness of Metallic Coatings by the Coulometric Method³
- B 530 Test Method for Measurement of Coating Thicknesses by the Magnetic Method: Electrodeposited Nickel Coatings on Magnetic and Nonmagnetic Substrates³
- B 537 Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure³
- B 554 Guide for Measurement of Thickness of Metallic Coatings on Nonmetallic Substrates⁵
- B 568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry³
- B 571 Test Methods for Adhesion of Metallic Coatings³
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings³
- B 659 Guide for Measuring Thickness of Metallic and Inorganic Coatings³
- B 697 Guide for Selection of Sampling Plans for Inspection of Electrodeposited Metallic and Inorganic Coatings³

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⁴ Discontinued. See 1986 Annual Book of ASTM Standards, Vol 03.02.

⁵ Discontinued. See 1985 Annual Book of ASTM Standards, Vol 02.05.

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- B 762 Method of Variables Sampling of Metallic and Inorganic Coatings³
- B 764 Test Method for Simultaneous Thickness and Electrochemical Potential Determination of Individual Layers in Multilayer Nickel Deposit (STEP Test)³
- D 1193 Specification for Reagent Water⁶
- D 3951 Practice for Commercial Packaging⁷
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁸
- 2.2 ISO Standards:
- ISO 1456 Metallic coatings—Electrodeposited coatings of nickel plus chromium and of copper plus nickel plus chromium⁹
- ISO 1457 Metallic coatings—Electroplated coatings of copper plus nickel plus chromium on iron or steel⁹

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 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.

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 micrometres (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 micrometres,

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 micrometres (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:

- -ductile copper deposited from acid-type baths а
- b -single-layer nickel deposited in the fully-bright condition р
- -dull or semi-bright nickel requiring polishing to give full brightness
- d -double- or triple-layer nickel coatings
- -regular (that is, conventional) chromium r -microcracked chromium mc
- -microporous chromium mp

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 0.25µ m minimum (micro-cracked) chromium has the classification number: Fe/ Cu15a Ni25d 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 classification numbers corresponding to the specified service condition number, as given in Table 1, Table 2, Table 3, or Table 4. 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 ductility if other than the standard value (see 6.4).

5.1.8 The extent of tolerable surface deterioration after corrosion testing (see 6.6.3).

5.1.9 Sampling methods and acceptance levels (see Section 7).

⁶ Annual Book of ASTM Standards, Vol 11.01.

⁷ Annual Book of ASTM Standards, Vol 15.09.

⁸ Annual Book of ASTM Standards, Vol 03.05.

⁹ Available from International Standards Organization, 1 Rue de Varembe, Geneva 20, Switzerland.



TABLE 1 Nickel Plus Chromium Coatings on Steel

NOTE 1—Results of a test program indicate there is some doubt whether the coating systems described by the classification numbers involving regular chromium are satisfactory for SC 4 and SC 3.

NOTE 2—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 3 may be used to obtain the same service conditions.

Service Condition No.	Classification No. ^A	Nickel Thickness, µm
SC 5	Fe/Ni35d Cr mc	35
	Fe/Ni35d Cr mp	35
SC 4	Fe/Ni40d Cr r	40
	Fe/Ni30d Cr mc	30
	Fe/Ni30d Cr mp	30
SC 3	Fe/Ni30d Cr r	30
	Fe/Ni25d Cr mc	25
	Fe/Ni25d Cr mp	25
	Fe/Ni40p Cr r	40
	Fe/Ni30p Cr mc	30
	Fe/Ni30p Cr mp	30
SC 2 ^B	Fe/Ni20b Cr r	20
	Fe/Ni15b Cr mc	15
	Fe/Ni15b Cr mp	15
SC 1 ^B	Fe/Ni10b Cr r	10

^A When a dull or satin finish is required, unbuffed p nickel may be substituted for a b nickel or for the bright layer of d nickel.

^{*B*} p or d nickel may be substituted for b nickel in Service Condition Nos. 2 and 1, and mc or mp chromium may be substituted for r chromium in Service Condition No. 1.

TABLE 2 Copper Plus Nickel Plus Chromium Coatings on Steel

Service Condition No.	Classification No. ^A	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

^A When a dull or satin finish is required, unbuffed p nickel may be substituted for the bright layer of d nickel.

5.1.10 The minimum values of the electrochemical potential differences between individual nickel layers as measured in accordance with Test Method B 764 within the limits given in 6.7.

5.1.11 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 specified by the purchaser. The electroplated article shall be clean and free of damage.

6.1.2 Defects in the surface of the basis metal, such as scratches, porosity, nonconducting inclusions, roll and die marks, cold shuts, 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.

TABLE 3 Copper Plus Nickel Plus Chromium Coatings on Zinc Alloy

NOTE 1—Results of a test program indicate there is some doubt whether the coating systems described by the classification numbers involving regular chromium are satisfactory for SC 4 and SC 3.

Service Condition No.	Classification No. ^A	Nickel Thickness, µm
SC 5	Zn/Cu5 Ni35d Cr mc	35
	Zn/Cu5 Ni35d Cr mp	35
SC 4	Zn/Cu5 Ni35d Cr r	35
	Zn/Cu5 Ni30d Cr mc	30
	Zn/Cu5 Ni30d Cr mp	30
SC 3	Zn/Cu5 Ni25d Cr r	25
	Zn/Cu5 Ni20d Cr mc	20
	Zn/Cu5 Ni20d Cr mp	20
	Zn/Cu5 Ni35p Cr r	35
	Zn/Cu5 Ni25p Cr mc	25
	Zn/Cu5 Ni25p Cr mp	25
SC 2 ^B	Zn/Cu5 Ni20b Cr r	20
	Zn/Cu5 Ni15b Cr mc	15
	Zn/Cu5 Ni15b Cr mp	15
SC 1 ^B	Zn/Cu5 Ni10b Cr r	10

^A When a dull or satin finish is required, unbuffed p nickel may be substituted for b nickel or for the bright layer of d nickel.

^{*B*} p or d nickel may be substituted for b nickel in Service Condition Nos. 2 and 1, and mc or mp chromium may be substituted for r chromium in Service Condition No. 1.

TABLE 4 Nickel Plus Chromium Coatings on Copper or Copper Alloy

NOTE 1—Although the classification numbers are satisfactory for each of the indicated service condition numbers, systems using microdiscontinuous chromium are generally superior in corrosion resistance to those using regular chromium.

Service Condition No.	Classification No. ^A	Nickel Thickness, µm
SC 4	Cu/Ni30d Cr r	30
	Cu/Ni25d Cr mc	25
	Cu/Ni25d Cr mp	25
SC 3	Cu/Ni25d Cr r	25
	Cu/Ni20d Cr mc	20
	Cu/Ni20d Cr mp	20
	Cu/Ni25p Cr r	25
	Cu/Ni20p Cr mc	20
	Cu/Ni20p Cr mp	20
	Cu/Ni30b Cr r	30
	Cu/Ni25b Cr mc	25
	Cu/Ni25b Cr mp	25
SC 2 ^B	Cu/Ni15b Cr r	15
	Cu/Ni10b Cr mc	10
	Cu/Ni10b Cr mp	10
SC 1 ^B	Cu/Ni5b Cr r	5

^A When a dull or satin finish is required, unbuffed p nickel may be substituted for b nickel or for the bright layer of d nickel.

^{*B*} p or d nickel may be substituted for b nickel in Service Condition Nos. 2 and 1 and mc or mp chromium may be substituted for r chromium in Service Condition No. 1.

NOTE 2—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.



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. Various ASTM practices for the preparation of basis metal are available. See Section 2.

6.2.2 Following the preparatory operations, the parts (articles) to be electroplated are introduced in such plating baths 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, or Table 4 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 micrometres 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 (Note 5):

b for nickel deposited in the fully bright condition.

p for dull or semi-bright nickel requiring polishing to give full brightness. This nickel shall contain less than 0.005 mass % sulfur (Note 3 and Note 4), and have an elongation of not less than 8 %.

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 4), and shall have an elongation of not less than 8 %. The top layer of this system shall contain more than 0.04 mass % sulfur (Note 3 and Note 4), and its thickness shall be not less than 10 % of the total nickel thickness; the thickness of the bottom layer in double-layer coatings shall not be less than 60 % of the total nickel thickness, except for steel, where it shall be not less than 50 % nor more than 70 %. If there are three layers, the intermediate layer shall contain not less than 0.15 mass % sulfur and shall not exceed 10 % of the total nickel thickness. These requirements for multilayer nickel coatings are summarized in Table 5.

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).

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 B 487. 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,¹⁰ in accordance with Test Method B 764.

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

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 over the whole of the significant surface. The cracks shall be invisible to the unaided eye (see 6.9).

mp for microporous chromium containing a minimum of 10 000 pores/10 mm by 10 mm square (10 000 cm²). The pores shall be invisible to the unaided eye (see 6.9).

6.2.5.2 Thickness of Chromium Deposit—The minimum thickness of the chromium deposit shall be 0.25 μ m on significant surfaces (see 3.1), except that for service condition SC 1 (see 4.2.1) the minimum thickness may be reduced to 0.13 μ m. 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.

6.2.5.3 When plating chromium over microdiscontinuous nickel, excessive thickness will bridge the nonconductive particles within the nickel layer. A maximum of 0.5 μ m 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 B 571. The particular test or tests to be used shall be specified by the purchaser.

6.4 *Ductility*—The ductility shall be such that the elongation will not be less than stated in 6.2.3.1 for copper and 6.2.4.1 for nickel 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 Coating Thickness:

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

6.5.2 It is recognized that requirements may exist for thicker coatings than are covered by this specification.

6.5.3 The thickness of a coating and its various layers shall be measured at points on the significant surfaces (See 3.1 and Note 5).

NOTE 5—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

¹⁰ 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.

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			Thickness Relative to Total Nickel Thickness		
Layer Type of Nickel	Specific Elongation	Sulfur Content	Double	-Layer	Triple-Layer
			Steel	Other ^A	Steel and Other ^A
Bottom	≥8 %	<0.005 mass %	≥75 %	≥60 %	50-70 %
Middle (high-sulfur)		>0.15 mass %			≤10 %
Top (b)		>0.04 mass %	10–25 %	10–40 %	≥30 %
Test Method	See Appendix X2	Note 3 ^B			Note 4 ^B

TABLE 5 Summary of the Requirements for Double- and Triple-Layer Nickel Coatings

^A Copper, zinc, and aluminum substrates and their alloys.

^B For Notes 3 and 4, see Section 6.

TABLE 6 Nickel Plus Chromium^A on Stainless Steels, AISI Designated Type 300 and 400 Series,^B and Aluminum Alloys

NOTE 1—Before nickel-chromium plating, the stainless steel surface and the aluminum substrate shall be prepared by a pretreatment from Practice B 254,^C Guide B 253,^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 6 were obtained using only microporous chromium systems. No data are 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 number for specific 300 or 400 alloy.

involve the use of conforming, auxiliary, or bipolar electrodes or nonconducting shields.

6.5.3.1 The coulometric method described in Test Method B 504 may be used to measure thickness of the chromium, the total thickness of the nickel, and the thickness of the copper. The STEP test, Test Method B 764, which is similar to the coulometric method, may be used to closely estimate the thicknesses of individual layers of nickel in a multilayer coating.

6.5.3.2 The microscopial method described in Test Method B 487 may be used to measure the thickness of each nickel layer and of the copper layer.

6.5.3.3 The X-ray method described in Test Method B 568 may be used when the total thickness of a copper/nickel/

chromium composite coating is to be measured, without any indication of the thickness of each individual layer.

6.5.3.4 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 6.5.3. Other methods are outlined in Guide B 659.

6.6 Corrosion Testing:

6.6.1 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 6—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.

6.6.2 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 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

TABLE 7 Corrosion Tests Appropriate for Each Service Condition Number

NOTE 1—The so-called "neutral" salt spray test, Practice B 117 has been generally discredited as an accelerated corrosion test for decorative nickel-chromium coatings largely because of lack of reproducibility of results.^{*A*} It is recognized, however, that the test is still used in some segments of the electroplating industry to check the quality of coatings intended for use under relatively mild service conditions. Accordingly, it is suggested that any use of this test and the requirements to be met be the subject of agreement between the purchaser and the manufacturer and, further, that its use be confined to the coatings indicated as appropriate for Service Conditions Nos. 2 and 1.

NOTE 2—The Acetic-salt Method B 287 has been discontinued.

Basis Metals	Sorvice Condition	Corrosion Test and Duration h		
	No.	CASS Method B 368	Corrodkote Method B 380	Acetic-salt Method B 287
Steel, zinc alloy,	SC 5	44		
or copper and	SC 4	22	Two 16-h cycles	144
copper alloy,	SC 3	16	16	96
stainless steel and	SC 2	4	4	24
aluminum alloys	SC 1			8

^A Mendizza, A., "Standard Salt Spray Test—Is It a Valid Acceptance Test?" Properties, Tests and Performance of Electrodeposited Metallic Coatings, ASTM STP 197, ASTM 1956, p. 107.



function of the electroplated part. A method of rating corrosion is given in Practice B 537.

6.6.3 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.

6.7 STEP Test Requirements:

6.7.1 The electrochemical potential differences between individual nickel layers shall be measured for multilayer coatings corresponding to SC5, SC4, and SC3 in accordance with Test Method B 764 (STEP Test).

NOTE 7—Universally accepted STEP values have not been established but some agreement in the value of ranges has been obtained. The STEP values depend upon which two nickel layers are being measured.

The STEP potential difference between the semi-bright nickel layer and the bright nickel layer has a 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.

The STEP potential difference between the high-activity nickel layer and the bright nickel layer in triple-layer coatings has a potential range of 15 to 35 mV. The high-activity nickel layer is more active (anodic) than the bright nickel layer.

The STEP potential difference between the bright nickel layer and a nickel layer between the bright nickel layer and the chromium layer has a potential range of 0 to 30 mV. The bright nickel layer is more active (anodic) than the nickel layer prior to chromium.

6.8 Sulfur Content:

6.8.1 The sulfur content of the nickel deposit shall meet the maximum or minimum values as stated in 6.2.4.1 and Table 5.

6.8.2 Methods for sulfur determinations are given in Appendix X3.

6.9 Density and Measurement of the Discontinuities in Chromium:

6.9.1 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 mm square (10 000 pores/cm²) in any direction over the whole of the significant surface. The cracks and pores shall be invisible to the unaided eye.

6.9.2 Methods for measuring the discontinuities are given in Appendix X4. See X4.4 in Appendix X4 for a means of determining *active* corrosion sites by corrosion testing.

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 8-Usually, when a collection of coated articles, the inspection

lot (8.2), 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 B 602, Guide B 697, and Method B 762 contain sampling plans that are designed for the sampling inspection of coatings.

Test Method B 602 contains four sampling plans, three for use with tests that are 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 used.

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

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, B762 identifies the plan to be used.

NOTE 9—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, and Appendix X4. 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 D 3951.

9. Keywords

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

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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. DUCTILITY TEST

NOTE X2.1—This test is used to ensure compliance of the type of copper and nickel deposit with the appropriate definition given in 6.4.

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 or nickel to a thickness of 25 μ m 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 11.5 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 6.3.2. 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.

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 E 50 (Apparatus No. 13).

X3.1.5 Reagents:

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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.¹¹ 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 D 1193.

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 (KIO₃) 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 μ m 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 (Na₃PO₄) or 60 g/L of any other suitable anodic alkaline cleaner.

X3.1.7.3 Coat the passivated test panel with 25 to 37 μ m of nickel deposited from the same solution using the same parameters as for the coated articles represented by the test specimen.

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:

Expected Sulfur Content,	Weight of Sample g	
mass %	Weight of Outlipie, 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 X3.3). Add 2 mL of starch solution to the vessel. With the oxygen flow adjusted to 1.0 to 1.5 L/min (Note X3.4), add KIO₃ solution specified until the intensity of the blue color is that which is considered as the end point. Refill the buret.

NOTE X3.3—Always fill the titration vessel to the same point.

NOTE X3.4—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 KIO_3 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 preignited crucible. Cover and proceed as in X3.1.10.3.

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

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

¹¹ 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.

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where:

- A = grams of standard sample used,
- B = percent sulfur in the standard sample
- $C = \text{millilitres of KIO}_3$ solution required for titration of the standard sample (Note X3.5), and
- $D = \text{millilitres of KIO}_3$ solution required for titration of the blank (Note X3.5).

NOTE X3.5—Or apparent percentage of sulfur for "direct-reading" burets.

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

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

where:

- $E = \text{KIO}_3$ solution required for titration of the test sample (Note X3.5), mL,
- $D = \text{KIO}_3$ 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 Method¹²—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:

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.¹¹ 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 D 1193.



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

X3.2.4.3 Ammoniacal Zinc Sulfate Solution—Dissolve 50 g of zinc sulfate ($ZnSO_4 \cdot 7H_2O$) 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 ($H_2PtCl_6 \cdot 6H_2O$) 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 (KIO₃) at 180°C for 1 h. Dissolve 3.570 g of the KIO₃ 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 KIO₃ 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:

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

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.

¹² Luke, C. L., Analytical Chemistry, Vol 29, 1957, p. 1227.

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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 acidhexachloroplatinic 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 X3.6—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 X3.6). Separate the gas delivery tube from the evolution head and remove the receiving flask with the delivery tube.

NOTE X3.7—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:

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

where:

 $A = 0.005 N \text{ KIO}_3$ solution used for the sample titration, mL,

 $B = 0.005 N \text{ KIO}_3$ 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*⁸—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 paint or pressure sensitive tape, including the wire used to make contact to the cathode bar. 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 a 5 % by mass solution of H_2SO_4 .

X4.2.2 Make freshly cleaned sample anodic at 0.8V for 30 s in the copper plating bath, then switch to cathodic (see Fig. X4.1) at approximately 0.2 to 0.4V, for 2 min (see Note X4.1 and Note X4.2). (**Caution**—Do not go beyond the specified anodic voltage or time because nickel will slowly dissolve or become passivated.)



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



Bath formulation—(non-critical) $CuSO_4 \cdot 5H_2O \ 1 \ m \ (250 \ g/L)$ $H_2SO_4(SpG \ 1.95) \ 0.5 \ m \ (20-25^{\circ}C)$ Temperature (room) Anode (copper) Live entry

X4.2.3 Following copper electroplating, carefully remove the specimen, rinse in 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.

X4.2.4 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 X4.4 for a guide to the determination of active: corrosion sites in the chromium layer.)

X4.3.2 Current measured or recorded during the cathodic cycle, or both, serves as a reliable indicator of porosity. If current remains low (<1 mA/cm²) 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/cm²) final current is indicative of high porosity. Use of a strip chart recorder provides a permanent record of the test current. With experience, direct counts of nodules of Cu deposited can be reduced to periodic verifications as the *I-t* signature. A qualitative visual check (microscopically) will then suffice for regular routine use.

NOTE X4.1—Exact potential used is dependent on anode-cathode spacing. At a distance of 8 to 10 cm, 0.2 V usually produces the desired deposit. As spacing increases, the potential can be increased to 0.4 V.

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).

X4.3.3 *Precautions*—Do not exceed 0.6 V cathodic. 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.

X4.4 Determination of Active Corrosion Sites By Corrosion Testing:

X4.4.1 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.

X4.4.2 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. After drying either by hot air or solvent such as methanol, the part should be examined at a magnification between 100 and 200x 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 photographs at a known magnification, the active pore sites can be counted and the active sites per area calculated. Different photographs 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.

X4.4.3 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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