Designation: B604 – 91 (Reapproved 2015)

Standard Specification for Decorative Electroplated Coatings of Copper Plus Nickel Plus Chromium on Plastics[1]

This standard is issued under the fixed designation B604; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers the requirements for several grades and types of electrodeposited copper plus nickel plus chromium coatings on plateable plastic substrates where appearance, durability and resistance to thermal cycling are important to service performance. Five grades of coatings are provided to correlate with the service conditions under which each is expected to provide satisfactory performance.

1.2 This specification covers the requirements for coatings applied subsequent to the application of metal film by autocatalytic deposition or subsequent to the application of any strike coatings after autocatalytic deposition.

1.3 The following caveat pertains only to the test method portions of Section 6, Annex A1, and Appendix X2, Appendix X3, and Appendix X4 of this specification. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

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

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

B532 Specification for Appearance of Electroplated Plastic Surfaces

B533 Test Method for Peel Strength of Metal Electroplated Plastics

B556 Guide for Measurement of Thin Chromium Coatings by Spot Test

B567 Test Method for Measurement of Coating Thickness by the Beta Backscatter Method

B568 Test Method for Measurement of Coating Thickness by X-Ray Spectrometry

B602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings

B659 Guide for Measuring Thickness of Metallic and Inorganic Coatings

B727 Practice for Preparation of Plastics Materials for Electroplating

B764 Test Method for Simultaneous Thickness and Electrode Potential Determination of Individual Layers in Multilayer Nickel Deposit (STEP Test)

D1193 Specification for Reagent Water

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

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.

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, in

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[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.

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accordance with the following scale:

SC5—extended very severe
SC4—very severe
SC3—severe
SC2—moderate
SC1—mild

4.2.2 Service condition numbers are further defined in Appendix X1 where they are related to the severity of exposure encountered by electroplated articles.

4.3 *Coating Classification Number*— The coating classification number is a means of specifying the types and thicknesses of coatings appropriate for each grade and is comprised of the following:

4.3.1 The symbol for the substrate (PL) indicating it is plateable plastic, followed by a slash mark,

4.3.2 The chemical symbol for copper (Cu),

4.3.3 A number giving the minimum thickness of the copper coating in micrometres,

4.3.4 A lower-case letter designating the type of copper electrodeposit (see 4.4 and 6.3.1),

4.3.5 The chemical symbol for nickel (Ni),

4.3.6 A number giving the minimum thickness of the nickel in micrometres,

4.3.7 A lower-case letter designating the type of nickel electrodeposit (see 4.4 and 6.3.2),

4.3.8 The chemical symbol for chromium (Cr), and

4.3.9 A lower-case letter or letters designating the type of chromium (see 4.4 and 6.3.3).

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 Number*—A coating on plastic comprising 15 µm minimum ductile acid copper plus 15 µm minimum double-layer nickel plus 0.25 µm minimum microporous chromium has the classification number: PL/Cu15a Ni15d Cr mp.

TABLE 1 Copper Plus Nickel Plus Chromium Coatings on Plastic[A]

Service Condition Number	Classification Number	Equivalent Nickel Thickness	
		µm	mils (approx.)
SC 5	PL/Cu15a Ni30d Cr mc	30	1.2
	PL/Cu15a Ni30d Cr mp	30	1.2
SC 4	PL/Cu15a Ni30d Cr r	30	1.2
	PL/Cu15a Ni25d Cr mc	25	1.0
	PL/Cu15a Ni25d Cr mp	25	1.0
SC 3	PL/Cu15a Ni25d Cr r	25	1.0
	PL/Cu15a Ni20d Cr mc	20	0.8
	PL/Cu15a Ni20d Cr mp	20	0.8
SC 2	PL/Cu15a Ni15b Cr r	15	0.6
	PL/Cu15a Ni10b Cr mc	10	0.4
	PL/Cu15a Ni10b Cr mp	10	0.4
SC 1	PL/Cu15a Ni7b Cr r	7	0.3

[A] The minimum copper thickness may be greater in some applications to meet thermal cycling and other requirements.

5. Ordering Information

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

5.1.1 ASTM designation number.

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 number corresponding to the service condition number, as given in Table 1.[3] 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).

5.1.10 Whether thermal cycle and corrosion testing shall be conducted individually on separate specimens as described in 6.6 and 6.7, or sequentially using the same specimens as described in 6.8, and whether the specimens shall be unmounted or mounted in a manner simulating assembly when these tests are conducted.

5.2 The minimum values of the electrochemical potential differences between individual nickel layers as measured in accordance with Test Method B764 within the limits given in 6.10.

6. Product Requirements

6.1 *Visual Defects:*

6.1.1 The significant surfaces of the electroplated articles shall be free of visible 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 free of damage and clean.

6.1.2 Defects in the surface of the molded plastic, such as cold shots, ejection marks, flash, gate marks, parting lines, splay and others, may adversely affect the appearance and performance of coatings applied thereto despite the observance

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[3] "Performance of Decorative Electrodeposited Copper-Nickel-Chromium Coatings on Plastics" is a final report on programs conducted by ASTM and ASEP to evaluate the coating classification numbers. A copy of the report has been filed at ASTM Headquarters as RR B-8-1003.

of the best electroplating practice. Accordingly, the electroplater's responsibility for defects in the coating resulting from the plastic-molding operation shall be waived (Note 1).

NOTE 1—To minimize problems of this type, the specifications covering the items to be electroplated should contain appropriate limitations on the extent of surface defects. Practice B532 distinguishes between defects that arise primarily in molding and those that arise in electroplating operations.

6.2 *Pretreatments*—Proper preparatory procedures are essential for satisfactory performance of electrodeposited coatings on plastics. Procedures described in Practice B727 may be followed. In the case of patented processes, the instructions provided by the suppliers of those processes shall be followed.

6.3 *Process and Coating Requirements*—Following preparatory operations, plastic articles are placed in electroplating solutions as required to produce the composite coating described by the specific coating classification number or by coating one of the specified classification numbers listed in Table 1 appropriate for the specified service condition number.

6.3.1 *Type of Copper*—Ductile copper shall be deposited from acid-type baths containing organic additives that promote leveling by the copper deposit.

6.3.2 *Type of Nickel*—For double- or triple-layer nickel coatings, the bottom layer shall contain less than 0.005 mass % sulfur (Note 2). The top layer shall contain greater than 0.04 mass % sulfur (Note 3), and its thickness shall be not less than 10 % of the total nickel thickness. In double-layer coatings, the thickness of the bottom layer shall be not less than 60 % of the total nickel thickness. In triple-layer coatings, the bottom layer 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 2.

6.3.3 *Thickness of Chromium Deposit*—The minimum permissible thickness of the chromium deposit shall be 0.25 µm on significant surfaces. 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 (see 4.4).

NOTE 2—The sulfur content is specified in order to indicate which type of nickel electroplating solution must be used. Although no simple method is yet 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 X2 for the determination of sulfur in electrodeposited nickel.

TABLE 2 Summary of the Requirements for Double- and Triple-Layer Nickel Coatings

Layer Type of Nickel	Specific Elongation	Sulfur Content	Thickness Relative to Total Nickel Thickness	
			Double-Layer	Triple-Layer
Bottom(s)	8 %	less than 0.005 %	equal to or greater than 50 %	equal to or greater than 50 %
Middle (high-sulfur (b))	...	greater than 0.15 mass %	...	10 % max
Top (b)	...	greater than 0.04 %	equal to or greater than 40 %	equal to or greater than 40 %
Test Method	Appendix X3	[A]	...	[B]

[A] See Note 2 in the text of this specification.
[B] See Note 3 in the text of this specification.

NOTE 3—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 be measured by the STEP test in accordance with Test Method B764.

6.4 *Ductility*—The minimum value of the ductility shall be 8 % for copper and for nickel when tested by the method given in Appendix X3. Greater ductility 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 4.2 and Note 4.)

NOTE 4—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.

6.5.3.1 The coulometric method described in Test Method B504 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 B764, which is similar to the coulometric method, may be used to determine the thicknesses of individual layers of nickel in a multilayer coating.

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

6.5.3.3 The beta backscatter method described in Test Method B567 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 6.4.3. Other methods are outlined in Test Methods B530 and B568 and Guides B556 and B659.

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 3. The test is described in detail in the referenced ASTM standard.

NOTE 5—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

TABLE 3 Corrosion Tests Appropriate for Each Service Condition Number

Service Condition Number	Duration of Corrosion (CASS) Test[A]
SC 5	three 16-h cycles[B]
SC 4	two 16-h cycles[B]
SC 3	one 16-h cycle[B]
SC 2	8 h
SC 1	...

[A] See Method B368.

[B] Each 16-h CASS test cycle shall consist of 16 h of exposure followed by removal from the test cabinet, rinsing in water, and inspection. The test specimen shall not be out of the test cabinet for more than 8 h between cycles.

the relative corrosion resistance of these materials in service.

6.6.2 After subjecting the article to the treatment described in the relevant test method, it shall be examined for evidence of corrosion penetration to the substrate or the copper layer, and for blistering of the coating. Any evidence of copper corrosion, blistering of the coating, or substrate exposure shall be cause for rejection. It is to be understood that occasional widely scattered 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 function of the electroplated part.

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 *Thermal Cycle Testing:*

6.7.1 Coated articles shall be subjected to three cycles of the thermal cycle test as outlined in Annex A1. The specified service condition number of the coating (or the service condition number corresponding to the specified classification number) shall correspond to the service condition number in Annex A1 for determining the temperature extremes as outlined therein.

6.7.2 After having been subjected to three cycles of the appropriate thermal cycle test, the coated article shall show no visible defects, such as cracking, blistering, peeling, sink marks, and distortions.

NOTE 6—There is no direct relation between the results of thermal cycle testing and performance in service, because it is not always possible to predict and control the thermal exposure of the coated article in service or during storage. Therefore, the results of thermal cycling should be used to control the quality of electroplated plastic articles and not as direct guide to performance in service.

6.8 *Combined Thermal Cycle and Corrosion Testing:*

6.8.1 Corrosion testing may be combined with thermal cycle testing for articles electroplated according to the requirements of SC5, SC4, and SC3 by using the same coated articles in each test in sequence as described in this section. The use of combined thermal cycle and corrosion testing obviates the need to conduct the individual tests described in 6.6 and 6.7.

6.8.2 Expose the coated articles to one 16-h cycle according to the procedures outlined in Method B368 (CASS test).

6.8.3 Parts shall be rinsed with demineralized water only after each CASS test cycle.

6.8.4 Subject the electroplated articles to the thermal cycle test procedure given in Annex A1.

6.8.5 Steps 6.8.2 through 6.8.4 represent one cycle of combined thermal cycle and corrosion testing. For articles electroplated to SC5 or SC4, repeat for two additional times. For articles electroplated to SC3, repeat one additional time.

6.8.6 Coated articles shall be examined for defects after each cycle of combined thermal cycle-corrosion testing as indicated in 6.6.2 and 6.7.2.

6.9 *Adhesion*—Test Method B533 provides a procedure for measurement of the peel strength (adhesion) of metal-electroplated plastics using standard specimens. Since there is no direct correlation between results obtained on standard specimens and actual molded parts, the method is useful to determine that processing solutions are capable of giving acceptable results. The thermal cycle test described in 6.7 and the subsequent examination of the electroplated articles described in 6.7.2, or alternatively, the combined thermal cycle test described in 6.7.2, or alternatively, the combined thermal cycle and corrosion tests described in 6.8, are recommended instead of other tests.

6.10 *STEP Test Requirement:*

6.10.1 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 B764 (STEP test). See Note 7.

NOTE 7—Universally accepted STEP values have not been established but some agreement exists for the required ranges. The STEP values depend on which two nickel layers are being measured: (*a*) the STEP potential difference between the semi-bright nickel layer and the bright nickel layer is within the 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; (*b*) the STEP potential difference between the high-activity nickel layer and the bright nickel layer in triple-layer nickel coatings is within the range of 15 to 35 mV. The high-activity layer is more active (anodic) than the bright nickel layer; and (*c*) the STEP potential difference between the bright nickel layer and a nickel layer between the bright nickel layer and the chromium layer is within 0 to 30 mV. The bright nickel layer is more active (anodic) than the nickel layer applied prior to the chromium.

6.11 *Sulfur Content:*

6.11.1 The sulfur content of the nickel deposit shall meet the maximum or minimum values as stated in 6.3.2 and Table 2.

6.11.2 Methods for sulfur determinations are given in Appendix X2.

6.12 *Density and Measurement of Discontinuities in Chromium:*

6.12.1 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 in any direction over the whole of the significant surface. Microporous chromium shall contain a minimum of 100 pores/mm^2 in any direction over the whole of the significant surface. The cracks and pores shall be invisible to the unaided eye.

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

7. Sampling Requirement

7.1 A random sample of the size required by Test Method B602 shall be selected from the inspection lot (see 7.2). The articles in the lot shall be inspected for conformance to the requirements of this specification and the lot shall be classified as conforming or not conforming to each requirement according to the criteria of the sampling plans in Test Method B602.

NOTE 8—Test Method B602 contains three sampling plans for the original inspection of coated articles. Two are to be used where the test methods are nondestructive, that is, the test method does not make the article nonconforming. The third plan is used where the test method is destructive. If it is not clear if the test is destructive or not, the purchaser should identify which test methods are destructive, and which are nondestructive. In some instances, both nondestructive and destructive test methods may exist for the testing of the conformance of a coating to a particular requirement. The purchaser should state which is to be used.

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 at 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 Annex A1 and 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 ductility, sulfur content, the number of discontinuities, thermal cycle and corrosion testing, and STEP testing, separately prepared specimens may be used.

ANNEX

(Mandatory Information)

A1. Thermal Cycling of Electroplated Plastics

NOTE A1.1—This test method is used to ensure compliance of electroplated plastics with the thermal cycle requirements given in 6.7 and 6.8.

A1.1 *Apparatus*—The apparatus shall consist of a circulating air heating chamber and cooling chamber sufficiently powered, insulated, and controlled to closely maintain the preset temperature. The two chambers may be separate, or may be built so as to constitute a single chamber. The controller and recorder used for chamber control, calibration, and records shall be accurate to ±1°C. All points within the working area of the test chamber shall remain within ±3°C of the set temperature. The air circulation shall be controlled to permit a consistent rate of heating or cooling of the parts during the test.

A1.2 *Elapsed Time After Electroplating*—The elapsed time between completion of the electroplating operation and thermal cycle testing may influence the results. The elapsed time shall be 24 ± 2 h.

A1.3 *Procedure:*

A1.3.1 Parts may be introduced into the chamber unmounted, or mounted in a manner simulating assembly as specified by the purchaser.

A1.3.2 Load the chamber with the desired quantity of parts to be tested.

A1.3.3 Record the location of parts within the chamber, the loading and the size of the parts being tested.

A1.3.4 The thermal cycle temperature limits corresponding to the specified service condition number shall be chosen from Table A1.1.

A1.3.5 Each thermal cycle shall consist of either placing the samples in a room-temperature chamber and heating the chamber to the high limit, or placing the samples directly into a chamber at the high limit, and performing the following:

A1.3.5.1 Expose the parts for one hour at the high limit.

A1.3.5.2 Allow the parts to return to 20 ± 3°C and maintain at this temperature for 1 h. This is frequently accomplished by removing the parts from the chamber.

A1.3.5.3 Expose the parts for one hour at the low limit.

A1.3.5.4 Allow the parts to return to 20 ± 3°C and maintain at this temperature for 1 h. Steps A1.3.5.1 through A1.3.5.4 constitute one full thermal cycle.

A1.3.6 When the number of cycles specified in 6.7 and 6.8 has been completed, inspect the parts for coating defects

TABLE A1.1 Recommended Thermal Cycling Temperature Limits

Service Condition Number	Temperature Limits, °C	
	High	Low
SC5 — Extended very severe	85	–40
SC4 — Very severe	80	–40
SC3 — Severe	80	–30
SC2 — Moderate	75	–30
SC1 — Mild	60	–30

produced by thermal cycling. See Specification B532, Table 1, for the limits established for visual defects.

A1.4 *Recording of Test Results*—The recording of the test results shall include the following:

A1.4.1 A statement that the test was performed according to Specification B604, Annex A1.

A1.4.2 The service condition number for which the part was tested.

A1.4.3 The tray construction (if a tray is used) and chamber loading.

A1.4.4 The last calibration date of the controller and records.

A1.4.5 The extent, nature, and location of the defects.

A1.5 *Precision and Bias*—The precision and bias of this test method have not been established.

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-term protection* of the substrate is required; for example, conditions encountered by some exterior components of automobiles.

X1.2 *Service 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)*—Indoor exposure in places where condensation of moisture may occur; for example, in kitchens and bathrooms.

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

X2. DETERMINATION OF SULFUR IN ELECTRODEPOSITED NICKEL

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.

X2.1 Total Sulfur in Electroplated Nickel by Combustion-Iodate Titration

X2.1.1 *Scope*—This method covers the determination of sulfur in concentrations from 0.005 to 0.5 mass %.

X2.1.2 *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 X2.1—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.

X2.1.3 *Interferences*— The elements ordinarily present in electroplated nickel do not interfere.

X2.1.4 *Apparatus*—Induction heating apparatus for determination of sulfur by direct combustion as described in Practices E50 (Apparatus No. 13).

X2.1.5 *Reagents:*

X2.1.5.1 *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.[4] 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.

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[4] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., New York, NY, and the "United States Pharmacopeia."

X2.1.5.2 *Purity of Water*— Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D1193.

X2.1.5.3 *Hydrochloric Acid* (3 + 97)—Mix 3 volumes of concentrated hydrochloric acid (HCl) (sp gr 1.19) with 97 volumes of water.

X2.1.5.4 *Iron (Low-Sulfur) Accelerator*—Chips.

X2.1.5.5 *Iron (Low-Sulfur) Accelerator*—Powder.

X2.1.5.6 *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.

X2.1.5.7 *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 X2.2—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.

X2.1.5.8 *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.

X2.1.5.9 *Tin (Low-Sulfur) Accelerator* —Granular.

X2.1.6 *Standards*—Standards for calibration are National Institute of Standards and Technology (formerly National Bureau of Standards) steels of the proper sulfur content.

X2.1.7 *Sample Preparation:*

X2.1.7.1 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.

X2.1.7.2 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.

X2.1.7.3 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.

X2.1.7.4 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.

X2.1.7.5 Separate 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.

X2.1.8 *Weight for Standards and Samples*—Select and weigh to the nearest 0.1 mg an amount of sample as follows:

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

X2.1.9 *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.

X2.1.10 *Procedure:*

X2.1.10.1 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.

X2.1.10.2 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) (X2.1.5.3) (Note X2.3). Add 2 mL of starch solution to the vessel. With the oxygen flow adjusted to 1.0 to 1.5 L/min (Note X2.4), add KIO_3 solution specified until the intensity of the blue color is that which is considered as the end point. Refill the buret.

NOTE X2.3—Always fill the titration vessel to the same point.

NOTE X2.4—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.

X2.1.10.3 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.

X2.1.10.4 *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 X2.1.10.3.

X2.1.11 *Calculation*— Calculate the sulfur factor of the potassium iodate as follows:

$$\text{Sulfur factor, g/unit volume} = \frac{A \times B}{(C - D) \times 100} \quad \text{(X2.1)}$$

where:

A = standard sample used, g,
B = sulfur in the standard sample, % ,
C = KIO_3 solution required for titration of the standard sample (Note X2.5), mL, and
D = KIO_3 solution required for titration of the blank, mL (Note X2.5).

NOTE X2.5—Use apparent percentage of sulfur for “direct-reading” burets.

X2.1.11.1 Calculate the percentage of sulfur in the test sample as follows:

$$\text{Sulfur, mass \%} = \frac{(E - D)F}{G} \times 100 \quad \text{(X2.2)}$$

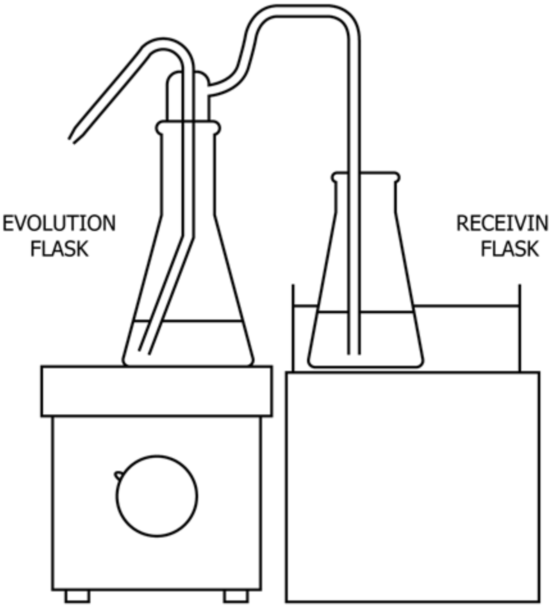

FIG. X2.1 Apparatus for the Determination of Sulfur in Electroplated Nickel Foil by the Evolution Method X2.2

where:

E = KIO_3 solution required for titration of the test sample, mL (Note X2.5),
D = KIO_3 solution required for titration of the blank, mL,
F = average sulfur factor of the KIO_3, g/unit volume, for the standards used (see X2.1.11), and
G = sample used, g.

X2.2 Determination of Sulfur in Electroplated Nickel by the Evolution Method

X2.2.1 *Scope*—This method covers the determination of sulfide sulfur in electroplated nickel in the range from 0.005 to 0.2 mass %.

X2.2.2 *Summary of Method*[5]—Sulfide sulfur is evolved as hydrogen sulfide (H_2S) 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 iodide (KIO_3) as the primary standard.

X2.2.3 *Apparatus:*

X2.2.3.1 The apparatus is shown in Fig. X2.1. It may be assembled using a 50-mL Erlenmeyer flask with a No. 19/38 outer joint. A wash bottle fitted 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.

X2.2.3.2 A nitrogen cylinder with valves and pressure regulator.

X2.2.3.3 Buret, 10-mL.

X2.2.4 *Reagents:*

X2.2.4.1 *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.[4] 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.

X2.2.4.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification D1193.

X2.2.4.3 *Ammoniacal Zinc Sulfate Solution*—Dissolve 50 g of zinc sulfate ($ZnSO_2{\cdot}7H_2O$) in 250 mL of water, add 250 mL of ammonium hydroxide (NH_4 OH sp gr 0.90) and mix. Transfer to a flask and allow to stand about 24 h and filter into a polyethylene bottle.

X2.2.4.4 *Hexachloroplatinic Acid Solution* (10 g/L)—Dissolve 0.5 g of hexachloroplatinic acid ($H_2PtCl_6{\cdot}6H_20$) in about 40 mL of water, add 5 mL of hydrochloric acid (HCl sp gr 1.19), and dilute to 50 mL.

X2.2.4.5 *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.

X2.2.4.6 *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.

X2.2.4.7 *Potassium Iodate, Standard Solution* (0.005 *N*)—Transfer 25 mL of 0.1 *N* KIO_3 solution to a 500-mL volumetric flask with a pipet, dilute to volume, and mix.

X2.2.4.8 *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.

X2.2.5 *Sample Preparation*—Prepare sample as outlined in X2.1.7.

X2.2.6 *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 ± 0.02
0.005 to 0.07	1.0
0.05 to 2	0.4

X2.2.7 *Procedure:*

X2.2.7.1 Weigh the specified amount of sample to the nearest 0.1 mg and transfer to the 50-mL evolution flask.

X2.2.7.2 Add 20 mL of water and 3 mL of ammoniacal zinc sulfate solution to the receiving flask.

X2.2.7.3 Adjust the hot plate to maintain the temperature of 25 mL of water in a 50-mL Erlenmeyer flask at 80°C.

X2.2.7.4 Add 15 mL of the hydrochloric acid-hexachloroplatinic acid solution to the sample. Assemble the apparatus as shown in Fig. X2.1 and start a very gentle stream of nitrogen through the system.

NOTE X2.6—A flow of about 30 cm^3/min is satisfactory. If the sample dissolves rapidly, the flow should be decreased during the time hydrogen is freely liberated.

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[5] Luke, C. L., *Analytical Chemistry*, Vol 29, 1957, p. 1227.

X2.2.7.5 Continue the heating and flow of nitrogen until the sample is completely dissolved, then continue for 5 min (Note X2.6). Separate the gas delivery tube from the evolution head and remove the receiving flask with the delivery tube.

NOTE X2.7—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).

X2.2.7.6 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.

X2.2.7.7 Run a blank titration to the same starch-iodide color on a mixture of 20 mL of water, 3 mL of ammoniacal zinc sulfide, 1 mL of starch-iodide 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.

X2.2.8 *Calculation*— Calculate the mass percent of sulfide sulfur as follows:

$$\text{Sulfide sulfur, mass \%} = \frac{(A - B) \times 0.005 \times 0.016}{W} \times 100 \tag{X2.3}$$

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.

X3. DUCTILITY TEST

NOTE X3.1— This test is used to test ensure compliance of the type of copper and nickel deposit with the appropriate definition given in 6.4. Refer to Practice B489 for details on calculation of percent ductility.

X3.1 *Preparation of Test Piece:*

X3.1.1 Prepare a plated test strip 150 mm long, 10 mm wide, and 1 mm thick by the following method:

X3.1.1.1 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 µm under the same conditions and in the same bath as the corresponding articles.

X3.1.1.2 Cut the test strip from the electroplated sheet with a flat shear. Round or chamfer the longer edges of the test strip, at least on the electroplated side, by careful filing or grinding.

X3.2 *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.

X3.3 *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.

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:*

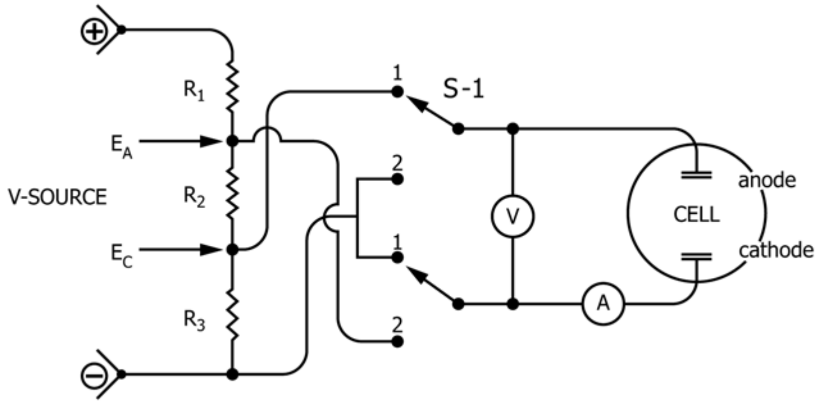
X4.2.1 Mask all edges not covered by the chromium with a nonconductive paint or pressure sensitive tape, including the wire used to make contact to the cathode bar. After masking, clean the specimen by soaking in a hot alkaline cleaner until the surface is free of water breaks. A mild scrubbing with 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_2.

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). (**Warning—**Do not go beyond the specified anodic voltage or time because nickel will slowly dissolve or become passivated.)

Bath formulation—(non-critical)
$CuSO_4 \cdot 5\ H_2O$ 1 M (250 g/L)
H_2SO_4 (sp gr 1.95) 0.5 M (20–25°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.



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

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

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

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 the 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/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. With experience, direct counts of nodules of Cu deposited can be reduced to periodic verifications as the $I \cdot t$ 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 **Warning—**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. After cleaning, examine the part under magnification to determine pore count and size. A magnification between 100 and 200 X is convenient for the size of pores typically found in microdiscontinuous chromium layers. If possible, photograph the part under magnification as a reference. Different photographs should be taken under magnification of all the significant surfaces and current densities to record the difference in pore count and size after corrosion.

X4.4.2 To develop the active corrosion sites, subject the part to between 16 and 24 h of CASS testing. For convenience, subject the part to one cycle of CASS as determined by the corrosion specification for the part. 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 viewed again under the same magnification as previously used and in the same areas in which the pictures were taken. By comparing the pictures of these areas before and after corrosion, it is typically easy to distinguish between the pore sites that have started to corrode and those that have not. The corroding pore sites are typically distinctly larger than the uncorroded sites and have a darker and rougher texture. By means of the photograph at a known magnification, the active pore sites can be counted and the active sites per area can be calculated.

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 one cycle of 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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