Designation: B 651 – 83 (Reapproved 2001)

Standard Test Method for
Measurement of Corrosion Sites in Nickel Plus Chromium or Copper Plus Nickel Plus Chromium Electroplated Surfaces with Double-Beam Interference Microscope[1]

This standard is issued under the fixed designation B 651; 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 test method provides a means for measuring the average dimensions and number of corrosion sites in an electroplated decorative nickel plus chromium or copper plus nickel plus chromium coating on steel after the coating has been subjected to corrosion tests. This test method is useful for comparing the relative corrosion resistances of different electroplating systems and for comparing the relative corrosivities of different corrosive environments. The numbers and sizes of corrosion sites are related to deterioration of appearance. Penetration of the electroplated coatings leads to appearance of basis metal corrosion products.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *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:*

B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section[2]

3. Summary of Test Method

3.1 The depths and diameter of corrosion pits or the widths of corrosion crevices, and the number of pits per square millimetre or crevices per linear millimetre on a specimen surface, are determined using optical aids (magnifier, microscope, and interference microscope). The values are compared to dimensions and numbers of corrosion sites obtained from other specimens.

4. Significance and Use

4.1 Different electroplating systems can be corroded under the same conditions for the same length of time. Differences in the average values of the radius or half-width or of penetration into an underlying metal layer are significant measures of the relative corrosion resistance of the systems. Thus, if the pit radii are substantially higher on samples with a given electroplating system, when compared to other systems, a tendency for earlier failure of the former by formation of visible pits is indicated. If penetration into the semi-bright nickel layer is substantially higher, a tendency for earlier failure by corrosion of basis metal is evident.

5. Apparatus

5.1 *Double-Beam Interference Microscope* (lateral magnification about 100×), capable of producing, with white light, a visible group of interference fringes, and equipped with a calibrated fine focus and a graduated bifilar (movable cross hair) eyepiece.

5.2 *Magnifier or Microscope* (10× to 20×), with light source.

5.3 *Rule*, graduated in millimetres, and a *scriber* for producing visible lines on the specimen surface.

5.4 *Microscope*, with a magnification capability of 500×, equipped with a bifilar eyepiece, for making measurements on opaque surfaces.

5.5 Equipment for mounting and polishing of specimens for microscopical cross-sectional measurements.

6. Specimen Preparation

6.1 Clean the corroded specimen surface with an agent or agents that remove soil and corrosion products, but do not significantly change the surface of the corrosion sites. Scouring powder may be used to remove insoluble corrosion products, organic solvent to remove road tar, water accompanied by gentle abrasion with a cloth to remove lightly adherent soil, etc.

6.2 Mask with paint or tape that portion of the specimen surface on which no measurements of pits or cracks will be made. Alternatively, a gasketed cell pressed onto the surface may be used. The opening in the gasket will define the area to be stripped.

NOTE 1—If pitted, the area selected for measurement should contain at least 100 pits or be as large as 50 by 50 mm. If the area contains cracks, the location for measurement should contain at least 100 cracks, or be at least 50 mm long.

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[1] This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.08.03 on Decorative Coatings.

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[2] *Annual Book of ASTM Standards*, Vol 02.05.

6.3 Strip the chromium anodically at 6 to 8 V in a solution containing about 50 g/L of sodium carbonate (Na_2CO_3).

6.4 Remove masking material, if desired.

NOTE 2—If tape was employed for masking, its removal is recommended. When the specimen rests on tape, it will allow the specimen to settle slowly. This gradual movement interferes with measurements of penetration with the interference microscope.

7. Procedure for Determination of Average Number of Pits or Cracks

7.1 Using the 10× to 20× magnifier, count the number of pits in a known area or the number of cracks intersecting a line of known length. Where uncertainty exists as to whether localized blemishes are corrosion sites when the magnifier is employed, use the 100× microscope for verification. Extreme accuracy is not necessary; values within ±10 % of the true value are adequate.

7.1.1 For surfaces where the number of pits is more than about 1000/cm^2, count the pits bounded by lines seen in the eyepiece reticle of the 100× microscope enclosing a known area of specimen surface (probably about 0.5 mm^2).

7.1.2 For surfaces where the number of pits is less than about 1000/cm^2, lightly scribe lines 10 mm or less apart to form a rectilinear grid on the surface. Count the number of pits within a scribed area, by using the magnifier, or the 100× microscope, whichever has the necessary resolution to assure pit identification. Determine the area that contains about 100 pits, or, if the area exceeds 25 cm^2, count the number of pits in a 25-cm^2 area.

7.1.3 For surfaces with more than about 5 cracks per millimetre, count the number of cracks on the surface image that cross a 100× microscope reticle line of known length.

7.1.4 For a surface with fewer than about 5 cracks per millimetre, lightly scribe a straight line up to 50 mm long on the specimen surface. Using a magnifier or, if necessary, a 100× microscope, count the number of cracks in a known length of line, or all the cracks in 50-mm length, whichever comes first.

NOTE 3—If the cracks tend to be oriented, scribe the line approximately perpendicular to the predominant crack direction.

7.2 Calculate the number of pits as pits per square millimetre, or the number of cracks as cracks per millimetre. Enter result in Table 1 under "pit density" or "crack density."

8. Determination of Mean Dimensions of Pits or Cracks

8.1 Observe one pit or crack with the interference microscope.

8.1.1 Using the bifilar eyepiece, count the number of eyepiece scale units occupied by the major diameter of the pit, or by the width of the crack. If the crack width varies, or if the pit outline is irregular, estimate the average. Enter "width" value in Table 1.

8.1.2 Adjust the elevation of the microscope tube so that interference fringes appear in the deepest part of the pit or the crack (the portion seen in the field of view) being measured; enter the reading on fine-focus knob under *B* in Table 1. Using the fine-focus knob only, raise the tube so that the fringes appear on the uncorroded surface surrounding the corroded site, and so that the center of the fringe group is aligned with the location of the penetration measurement. The best fringe orientation is perpendicular to the major pit diameter or crack direction. Enter the reading on fine-focus knob under *A* in Table 1. Subtract *B* from *A* to obtain penetration *P* and enter the value into Table 1. (Fine-focus knobs are generally calibrated directly in micrometres, necessitating no further conversion.)

NOTE 4—If the bottoms of the corrosion sites do not produce visible fringes, treat the specimen with a suitable agent to clean or to brighten the sites. Then repeat the steps in 8.1 on ten pits or cracks. A 15-s soak in a water solution of 5 % H_2SO_4, by weight, followed immediately with a water rinse, is often helpful.

8.2 Section the specimen in the location of the above measurements. Mount, polish, etch, and measure (Note 5) the thickness of each deposit; enter the values into Table 1 under respective values of "Thickness." Employ a microscope having a magnification capability of at least 500×.

NOTE 5—For a guide to the procedure for measuring the thickness of each deposit consult Test Method B 487.

NOTE 6—A suggested etchant is 1 part by volume glacial acetic acid, 1

TABLE 1 Measurements of Corrosion Pits and Cracks in Nickel Plus Chromium or Copper Plus Nickel Plus Chromium Plated Surfaces

SAMPLE NO. ____________	EXPOSURE MEDIUM __________	EXPOSURE TIME ________
Pit or crack density (7.2)	___ pits/mm^2 ___ cracks/mm^2	
Pit or crack dimensions (8.1.1)	pit diameter ___ µm crack width ___ µm	
Thicknesses of deposits (8.2)	copper	____ µm
	semibright nickel	____ µm
	bright nickel	____ µm
	other nickel layer	____ µm
	chromium	____ µm

Penetration of Crack or Pit into the Semibright Nickel or Copper Layers

Total Penetration (*P*) (8.1.2)		Penetration into Semibright Nickel (8.3)	Penetration into Copper (8.4)
A	____ µm	___ µm	___ µm
B	____ µm		
A-B	____ µm	mean penetration into semibright	mean penetration into copper
		___ µm	___ µm

part concentrated nitric acid, (sp gr 1.42) and 1 part glycerin. Approximate etch time is 30 s.

8.3 For the determination of penetration by corrosion into the semi-bright nickel layer, subtract the value of thickness of all deposits above the semi-bright layer (obtained in 8.2) from each value of penetration *P* (obtained in 8.1.2); enter each value of penetration into Table 1 into the column designated" semi-bright penetration." Calculate the arithmetic mean of the values and enter the "Mean Penetration," P_s into Table 1.

8.4 For determination of penetration into the copper layer, subtract the thickness of all the layers above copper from the 1 penetration value ($A - B$) in Table 1. Calculate the arithmetic mean of the differences, and enter "Mean Penetration," P_{cu} into Table 1.

9. Report

9.1 Report the following information:
9.1.1 Sample number or identification,
9.1.2 Exposure medium,
9.1.3 Exposure time,
9.1.4 Thickness of deposits,
9.1.5 Pit or crack density,
9.1.6 Pit or crack dimensions: width and penetration,

NOTE 7—Level *A* (Table 1) is the reading on the calibrated fine-focus knob corresponding to the specimen surface plane. Level *B* is the reading on the calibrated fine-focus knob corresponding to the maximum depth of the pit or crack. $A - B$ is the depth of the pit or crack.

9.1.7 Semi-bright penetration, and
9.1.8 Copper penetration.

10. Precision and Bias

10.1 Precision of individual penetration measurements can be as good as ±1 μm. It is determined by the care with which the interference fringes, which move laterally across the field of view as the focus knob is turned, are positioned (either in the corrosion site or on the top surface).

10.2 Bias of individual penetration measurements can be as good as the precision, since the method has no built-in bias; it can be even better than the precision when computed as a statistical average. Bias is determined by the precision and by the care with which the pits are cleaned of foreign material but have had no metal removed by etching of any cleaning agent.

10.3 Bias of individual pit diameter measurements and thickness measurements can be as good as ±1 μm. Suggestions for obtaining measurements of best bias are found in Test Method B 487.

10.4 Biases of the means obtained for penetration, site diameter or width, and number are determined by the uniformity of distribution of pit size and density across the surface and by the number of individual pits measured that are used to obtain the mean. Values of number and size of corrosion sites need not be more accurate than ±10 % of the true average value.