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Standard Test Method for Measurement of Internal Stress of Plated Metallic Coatings with the Spiral Contractometer¹

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^{ε1} NOTE—Section 12 was added, the title changed to “Test Method,” and other editorial changes made in January 1992.

1. Scope

1.1 This test method covers the use of the spiral contractometer for measuring the internal stress of metallic coatings as produced from plating solutions on a helical cathode. The test method can be used with electrolytic and autocatalytic deposits.

1.2 *This standard does not purport to address all of the safety problems, 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. Terminology

2.1 Definitions:

2.1.1 *compressive stress* (–)—stress that tends to cause a deposit to expand.

2.1.2 *internal stress*—the net stress that remains in a deposit when it is free from external forces. The internal stress tends to compress or stretch the deposits.

2.1.3 *tensile stress* (+)—stress that tends to cause a deposit to contract.

3. Summary of Test Method

3.1 The test method of measuring stress with the spiral contractometer is based on plating on the outside of a helix. The helix is formed by winding a strip around a cylinder, followed by annealing. In operation, one end of the helix is fixed and the other is allowed to move as stresses develop. The free end is attached to an indicating needle through gears that magnify the movement of the helix. As the helix is plated, the stress in the deposit causes the helix to wind more tightly or to unwind, depending on whether the stress is compressive (–) or tensile (+). From the amount of needle deflection and other data, the internal stress is calculated.

3.2 With instrument modifications, the movement of the helix can be measured electronically instead of mechanically as described in 3.1.

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4. Significance and Use

4.1 The spiral contractometer, properly used, will give reproducible results (see 8.5) over a wide range of stress values. Internal stress limits with this method can be specified for use by both the purchaser and the producer of plated or electroformed parts.

4.2 Plating with large tensile stresses will reduce the fatigue strength of a product made from high-strength steel. Maximum stress limits can be specified to minimize this. Other properties affected by stress include corrosion resistance, dimensional stability, cracking, and peeling.

4.3 In control of electroforming solutions, the effects of stress are more widely recognized, and the control of stress is usually necessary to obtain a usable electroform. Internal stress limits can be determined and specified for production control.

4.4 Internal stress values obtained by the spiral contractometer do not necessarily reflect the internal stress values found on a part plated in the same solution. Internal stress varies with many factors, such as coating thickness, preparation of substrate, current density, and temperature, as well as the solution composition. Closer correlation is achieved when the test conditions match those used to coat the part.

5. Apparatus

5.1 The spiral contractometer is described by A. Brenner and S. Senderoff.²

NOTE 1—Spiral contractometers are available from many of the suppliers of nickel sulfamate.

5.2 Helices shall be stopped-off on the inside to prevent plating. Helices are available with or without a permanent inert coating on the insides (see Appendix X1).

5.3 The clamps holding the helix to the contractometer shall be coated with an inert nonconductive coating to prevent their plating and acting as thieves.

5.4 For testing electroplating solutions, anodes are placed equidistant from the helix and symmetrically positioned to produce even plate distribution. A minimum of four anodes is required. A concentric anode arrangement is preferred.

² Brenner, A., and Senderoff, S., *Proceedings of the American Electroplaters Society*, Vol 35, 1948, p. 53.

5.5 Laboratory tests on electroplating solutions shall utilize at least 3.7 L of solution. A 4-L beaker with an annular anode arrangement is convenient. Use of this volume or larger will minimize solution changes due to electrolysis during the test.

5.6 Laboratory tests on autocatalytic plating solutions are done in a 1-L, tall-form beaker. Obviously, no anodes are used.

6. Factors Affecting Accuracy

6.1 Variations in the preparation of the helix may cause substantial variations in results.

6.1.1 Stop-off material shall be applied properly to the interior of the helix. The stop-off material shall be thin and flexible to permit the helix to move freely during the test. A coating weight of less than 50 mg/dm² is satisfactory.

NOTE 2—The inside must be stopped-off with some inert, flexible coating. One acceptable stop-off material is “Microstop.”³ One part of “Microstop” is diluted with two parts of acetone before use. Any nickel deposited on the inside of the helix will exhibit an opposing effect.

6.1.2 Helices that have been permanently coated on the inside with TFE-fluorocarbon may give variable results when testing near-zero stresses.

6.1.3 Cleaning variations and surface preparation of the helix before the test can produce varying results. For example, electrocleaning of the helix shall always be cathodic and controlled with respect to current, time, and temperature. Anodic cleaning at this stage can give wide variations. Abrasive cleaning of the helix and the use of etchants shall be avoided.

6.1.4 Very thin deposits of less than about 3 μm are influenced more by the surface conditions and preparation of the helix than are thicker deposits.

6.2 Internal stress varies with current density used in electroplating. The variation is not predictable and depends on the metal being deposited, impurities or additives, and the current density range under consideration. It is important that the current be measured and controlled closely throughout the stress test. Variations in currents shall be held to less than 2 %.

6.3 Because the temperature of the plating solution may affect the internal stress, it shall be maintained within 2°C during the test. The initial rest point of the indicator and the final rest point are both taken at the operating temperature of the plating solution to eliminate thermal stresses.

6.4 The solution composition shall not vary during the test. Usually, if the repeatability tests in 8.5 are met, the solution can be assumed to be unchanged during the test runs. Conversely, when the repeatability tests are not met, the plating solution shall be analyzed to determine if any changes in solution composition have occurred during the test.

6.4.1 Tests run on electroplating solutions using insoluble or inefficient anodes could result in significant solution changes during the test.

6.4.2 When testing autocatalytic plating solutions, the constituents of the plating solution may be significantly depleted during the test, unless replenished.

6.5 A relationship between the surface area to be plated and the volume of autocatalytic plating solutions exists that may

affect the character of the deposit. In testing autocatalytic plating solutions, the ratio of plated surface area to the volume of solution that is normally used in the plating tank shall be maintained. When using proprietary solutions, the supplier’s recommendation shall be followed.

7. Calibration

7.1 Calibrate the instrument as directed in the manufacturer’s instructions.

7.2 The frequency of calibration will vary with use and extent of attack on the helices from the chemical stripping. When visible attack is noted, discard the helix.

7.3 The calibration procedure consists essentially of determining the force required per degree of dial deflection. A known mass is suspended over a small pulley on a lever arm with the helix mounted in place. The degree of deflection is read from the dial. The data required for the calibration calculations as expressed in metric units are as follows:

w	= mass used in calibrating, kg,
a	= length of lever arm, m,
p	= pitch of helix, m,
t	= thickness of the strip used to make the helix, m,
deg_{def}	= degree deflection; difference in dial readings caused by mass,
g	= 9.8 m/s ² (acceleration of free fall), and
Z	= calibration constant of the helix $\left(\frac{\text{MPa}}{\text{m deg}_{\text{def}}}\right)$

where

$$Z = \frac{2(w)(a)(g)}{p(t)\text{deg}_{\text{def}}} \times 10^{-6}$$

8. Procedure

8.1 The procedure will vary with the solution being tested. Follow the instructions given by the supplier carefully. Variations in the procedure can produce variations in results. Give appropriate attention to the factors in Section 6. A detailed procedure for nickel plating solutions appears in Appendix X1.

8.2 Position the spiral contractometer in electroplating solutions so that it is equidistant from the anodes. Position the anodes on at least four sides when they are used in a production tank or use a concentric anode arrangement. Do not place the spiral contractometer between the tank anodes and the work being plated in a production tank. A separate ammeter and current control are required. If the test is run on a sample of the solution, use a 4-L beaker.

8.3 When testing autocatalytic plating solutions, maintain the proper surface area-to-solution volume ratio (see 6.5).

8.4 The conditions of the test are usually chosen to match those of the production parts as closely as practicable.

8.4.1 The thickness of the deposit plated on the helix may not be required to be the same as the thickness plated on production parts. This is especially true when production part thickness requirements are greater than about 15 μm.

8.4.2 The current density used for the spiral contractometer is sometimes lower than that of production parts. Electroplating at the lower current density will often indicate the cumulative effects of solution impurities before these have much effect on the work being plated at the higher currents.

³ Manufactured by Michigan Chrome and Chemical Co., 8615 Grinell Ave., Detroit, MI 48174.

This information can be used as a measure of the solution purity and will allow planning for corrective or purification procedures.

8.5 Repeatability:

8.5.1 Consider the procedure repeatable if the results of three consecutive tests on the same solution sample produce internal stress values that do not vary more than $\pm 10\%$ from the mean of the three values.

8.5.2 For near zero stress values, -10 to $+10$ MPa, it is more difficult to detect small changes with the contractometer and an acceptable range for repeatability is ± 5 MPa.

8.6 Solution adjustments may be made during the test, especially when testing autocatalytic solutions and electroplating solutions using insoluble anodes.

8.7 Clean the instrument after each use and before storage. This is to prevent solution that may creep up the shaft from crystallizing and binding or corroding the instrument.

9. Stress Calculations

9.1 Data Required:

Z = calibration constant for the helix, MPa/(m deg_{def}),

D = change in dial reading during the test, deg, and

τ = thickness of the deposit on the helix, m.

τ can be calculated from the additional data:

A = plated area, m²,

W = mass of deposit, kg, and

d = density of deposit, kg/m³.

Then $\tau = W/(d)A$

$$\text{Internal stress, } S, \text{ in MPa} = \frac{Z \times D}{\tau} \quad (1)$$

NOTE 3—This value is an average stress and is uncorrected for the effects of the differences in Young's modulus between the helix and the

deposit. It also assumes a relatively thin deposit on the helix. If a corrected or truer stress, S_T , is desired:

$$S_T = S \left[1 + \left(\frac{E_0 \times t}{E \times \tau} \right) \right] \quad (2)$$

where:

E_0 = Young's modulus of the deposit, and

E = Young's modulus of the helix.

Normally, the correction made above is less than the experimental errors in the procedure and is infrequently used.

10. Report

10.1 When reporting internal stress values, it is necessary to include the following:

10.1.1 Metal deposited and the plating solution composition.

10.1.2 Instrument used (spiral contractometer).

10.1.3 Thickness of deposit on helix.

10.1.4 Current density (omit if autocatalytic deposits are being tested).

10.1.5 Temperature.

10.1.6 pH (if it is a controllable variable).

10.2 The unit for expressing internal stress is the megapascal (MPa). One megapascal is one meganewton per square metre (MN/m²), approximately 145 psi.

11. Precision and Bias

11.1 Results are reproducible to about $\pm 10\%$ except for the near-zero range of ± 10 MPa. In the near-zero range, results are reproducible to about ± 5 MPa.

12. Keywords

12.1 internal stress test; metallic deposit

APPENDIX

(Nonmandatory Information)

X1. PROCEDURE FOR STRESS DETERMINATION OF NICKEL ELECTRODEPOSITS

X1.1 Scope

X1.1.1 The following is a detailed procedure for the determination of internal stress using the spiral contractometer on nickel electroplating solutions. Some modifications can be made to adapt this procedure for use with other metal plating solutions.

X1.2 Preparation of Helix (use a stainless steel helix)

X1.2.1 Clean helix (as cathode) by electrocleaning at 4.0 to 5.0 A for 15 s in an alkaline steel cleaner at $50 \pm 2^\circ\text{C}$.

X1.2.2 Rinse.

X1.2.3 Immerse in 50 %, v/v, hydrochloric acid for 3 to 5 s.

X1.2.4 Rinse.

X1.2.5 Nickel strike at 5.0 A for 1 min in a standard Wood's nickel solution. The nickel strike is optional. If omitted, proceed to X1.2.9.

NOTE X1.1—The nickel strike solution contains 240 g/litre of nickel chloride hexahydrate and 12.5 % v/v of concentrated hydrochloric acid. It is operated at room temperature using nickel anodes.

X1.2.6 Rinse.

X1.2.7 Immerse in 50 %, v/v hydrochloric acid for 3 to 5 s.

X1.2.8 Rinse.

X1.2.9 Dry the helix with acetone and weigh to nearest 1 mg.

X1.2.10 Stop-off the inside of the helix by dipping in a test tube containing a stop-off lacquer solution (see Note 2). If the helix has a permanently bonded, inert coating, omit this step, the step described in X1.2.11, and the stripping of the stop-off.

X1.2.11 Remove the lacquer from the outside of the helix by wiping with acetone and then trichloroethylene. Do not touch the cleaned helix with bare fingers.

X1.2.12 Mount the helix with clamps. Stop-off the outside

of the bottom clamp or use a bottom clamp, bolt, and nut made of resistant plastic such as TFE-fluorocarbon. The top clamp need not be coated because it is not immersed.

NOTE X1.2—Since the clamp is insulated, the cathode efficiency can be calculated and even current distribution over the helix is obtained.

X1.2.13 Clean cathodically at 0.5 to 1.0 A for 5 s in the cleaner used in X1.2.1.

X1.2.14 Rinse.

X1.2.15 Immerse 50 %, v/v, hydrochloric acid for 3 to 5 s.

X1.2.16 Rinse.

X1.2.17 Inspect the stop-off coating on the inside of the helix. If it is peeling, strip it with acetone and re-coat (from X1.2.10) the helix.

X1.2.18 Mount the helix on the instrument in a no-stress state. Adjust the pointer gear to near its center.

NOTE X1.3—If the instrument is used daily, calibrate the helix once a week. For calibration, see regular instructions.

X1.3 Procedure

X1.3.1 Immerse the assembled instrument into the plating solution so that the dial face is horizontal and the solution level is just below the top clamp. The helix is equidistant from the anode surface. Use at least four anodes surrounding the helix or a concentric anode.

NOTE X1.4—An annular basket is convenient for use in nickel solutions and can be used in either laboratory or production tank tests. The basket is constructed of titanium expanded metal or mesh filled with segmented

nickel anodes. For nickel electroplating solutions containing less than about 15 g/litre of halide, sulfur depolarized nickel anode material is required.

X1.3.2 Allow a few seconds for the helix to reach the solution temperature and then record the initial dial reading. Temperature equilibrium is reached when the dial reading does not change after slightly tapping the instrument.

X1.3.3 Connect the electrical contacts and adjust the current to 2.0 A dc. Use a filtered rectifier or battery for the power source.

X1.3.4 Set the timer for 30 min.

NOTE X1.5—The thickness of the nickel plate should be 13 to 16 μm . This thickness minimizes the effect of the initial high stresses due to basis metal and basis metal preparation.

X1.3.5 Maintain the current within 0.05 A and the temperature within 2°C throughout the test. Tap the dial intermittently throughout the test to ensure that the instrument is not bound.

X1.3.6 Disconnect from the d-c power source after 30 min and record the final dial reading after tapping to ensure that the needle has reached a steady state.

X1.3.7 Remove the instrument and rinse it in hot water.

X1.3.8 Remove the helix from the instrument.

X1.3.9 Remove stop-off lacquer by immersion in acetone. Use succeeding amounts of fresh acetone until the wash solution is no longer colored.

X1.3.10 Allow the helix to dry and weigh it to the nearest 1 mg.

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