Designation: B636/B636M – 15

Standard Test Method for Measurement of Internal Stress of Plated Metallic Coatings with the Spiral Contractometer[1]

This standard is issued under the fixed designation B636/B636M; 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 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 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with 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:*

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

3.1.1 *compressive stress (−)*—stress that tends to cause a deposit to expand.

3.1.2 *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.

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

4. Summary of Test Method

4.1 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.

4.2 With instrument modifications, the movement of the helix can be measured electronically instead of mechanically as described in 4.1.

5. Significance and Use

5.1 The spiral contractometer, properly used, will give reproducible results (see 9.5) 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.

5.2 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.

5.3 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.

5.4 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.

[1] This test method is under the jurisdiction of ASTM Committee B08 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.10 on Test Methods.

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6. Apparatus

6.1 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.

6.2 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).

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

6.4 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.

6.5 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.

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

7. Factors Affecting Accuracy

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

7.1.1 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 3 mg/in.^{2} is satisfactory.

NOTE 2—The inside shall be stopped-off with some inert, flexible coating. One acceptable stop-off material is "Microstop." 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.

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

7.1.3 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.

7.1.4 Very thin deposits of less than about 3 µm [1.18×10^{-4} in.] are influenced more by the surface conditions and preparation of the helix than are thicker deposits.

7.2 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 %.

7.3 Because the temperature of the plating solution may affect the internal stress, it shall be maintained within 2°C [6.5°F] 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.

7.4 The solution composition shall not vary during the test. Usually, if the repeatability tests in 9.5 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.

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

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

7.5 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.

8. Calibration

8.1 Calibrate the instrument as directed in the manufacturer's instructions.

8.2 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.

8.3 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

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

9. Procedure

9.1 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

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