INTERNATIONAL

Designation: ~~B975 – 15~~ B975 – 18

Standard Test Method for Measurement of Internal Stress of Metallic Coatings by Split Strip Evaluation (Deposit Stress Analyzer Method)[1]

This standard is issued under the fixed designation B975; 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.

INTRODUCTION

The deposit stress analyzer method provides a rapid, accurate, and economical means for the determination of the internal tensile and compressive stress in metallic and nonmetallic coatings. Internal stress is expressed in pounds per square inch or megapascals. This procedure for measuring internal stress offers the advantages of test specimens that are pre-calibrated by the manufacturer, a small test specimen coating surface area, and rapid determination of the internal stress in the applied coating.

1. Scope

1.1 This test method for determining the internal tensile or compressive stress in applied coatings is quantitative. It is applicable to metallic layers that are applied by the processes of electroplating or chemical deposition that exhibit internal tensile or compressive stress values from ~~500~~200 to 145 000 psi ~~(3.45~~(1.38 to 1000 MPa).

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values ~~given~~stated in ~~parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.~~each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B636 Test Method for Measurement of Internal Stress of Plated Metallic Coatings with the Spiral Contractometer
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

2.2 *IEC Standard:*[3]

IEC 61010 Safety Requirements for Electrical Equipment for Measurement, Control, and Laboratory Use

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

[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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[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.

[3] Available from International Electrotechnical Commission (IEC), 3, rue de Varembé, P.O. Box 131, 1211 Geneva 20, Switzerland, http://www.iec.ch.

3.1.1 *average deposit thickness, n*—average deposit thickness equals the deposit weight in grams divided by the specific gravity of the deposit in grams per cubic centimetre multiplied by the plated deposit surface area per test strip (see Eq 3).

3.1.2 *constant K, n*—this certifiable calibrated number is determined experimentally for each material lot of test strips manufactured to enable simple mathematical calculation of the internal deposit stress while factoring the influence of the percent elongation difference between the deposit and the substrate without the use of complicated bent strip formulas. See Note 4 in Section 8.

3.1.3 *helix, n*—metal strip approximately 0.01 to 0.013 in. (0.025 to 0.033 cm) thick formed as a helix approximately ~~0.9 in.~~ 0.9 in. (2.3 cm) in diameter and ~~0.61 in.~~ 6.1 in. (15.5 cm) long with or without a polytetrafluoroethylene (PTFE) coating on the inside surface.

3.1.4 *in-site device, n*—this device holds a test strip during the application of a coating.

3.1.4.1 *Discussion—*

Anodes are located external to the specimen holder.

3.1.5 *internal stress, n*—stress in a given layer of coating can result from foreign atoms or materials in the layer that stress the natural structure of the deposit as the coating is being formed from sources independent of foreign atoms such as misfit dislocations and the result of additional processing.

3.1.5.1 *Discussion—*

Stress that develops in a given layer of material is measured as pounds per square inch or megapascals where 1 MPa = 145 psi.

3.1.6 *measuring stand, n*—this stand supports the test strip above a logarithmic scale that enables determination of the total number of increments spread between the test strip leg tips.

3.1.7 *modulus of elasticity, n*—stress required to produce unit strain, which may be a change in length (Young's modulus), a twist of shear (modulus of rigidity or modulus of torsion), or a change in volume (bulk modulus).

~~3.1.7 *on site specimen holder, n*—this device holds a test strip during the application of a coating.~~

~~3.1.7.1 *Discussion—*~~

~~Anodes are located external to the specimen holder.~~

3.1.8 *power supply, n*—rectifier to supply amperage for plating.

3.1.9 *self-contained plating cell, n*—this cell contains two anodes within the cell at an equal distance from the test strip that are suspended in electrolyte for deposition to occur. A section for a heating coil and a pump for solution agitation is an option.

3.1.10 *test strip, n*—metal strip formed from flat stock that receives the coating of material being evaluated for internal stress.

4. Summary of Test Method

4.1 The first attempt to measure stress values in applied coatings was the bent strip method, wherein a coating of known thickness was applied to a strip of flat stock material having a known modulus of elasticity, length, width, and thickness. In the test, one end of the strip was held in a fixed position and one end could bend. The degree of bend experienced by the test strip was then measured. Equations were proposed by Stoney, Barklie, and ~~Davies,~~Davies; Houssner, Balden, and ~~Morse,~~ Morse; and Brenner and Senderoff for this method of measurement to calculate the internal deposit stress that was sufficient to cause deflection of the flat stock material.

4.2 Later methods include the use of flat stock material formed into a helix that contracts or expands as a stressed coating is applied to the base material (spiral contractometer as described in United States Patent 4,086,154) and a disk formed from flat stock material that bows outward or inward as a stressed coating is applied to the base material (stress meter).

4.3 The deposit stress analyzer method for determining the internal stress value of a given ~~coating~~plating uses bent strip technology and the formulas devised for calculation of results applicable to this approach. A specific test piece comprises a selected metallic material that exhibits spring-like properties with specified dimensions that define an end area split to give two legs (see Fig. 1). These test strips are coated with a resist, to prevent ~~deposition,~~deposition on the front of one leg and the back side of the other leg and on both sides above where the legs divide, leaving a space uncoated at the top for the purpose of making electrical contact to the test piece during the plating process. ~~As a test piece is plated, the legs bend to relieve the stress that is induced as deposition occurs. Tensile stress bends the test strip legs with the plated deposit on the outside, while compressive stress bends the test strip legs with the resist on the outside.~~ See Fig. 2. Each test is performed at specific operating conditions that are usually selected to approximate the conditions for parts being processed in production mode.

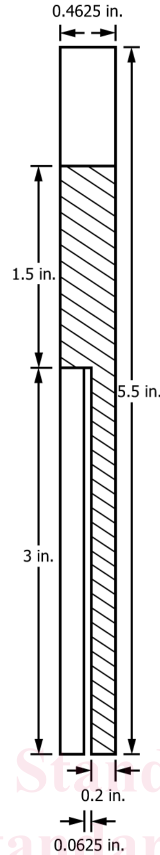

FIG. 1 Test Strip Parameters
1 in. = 2.54 cm

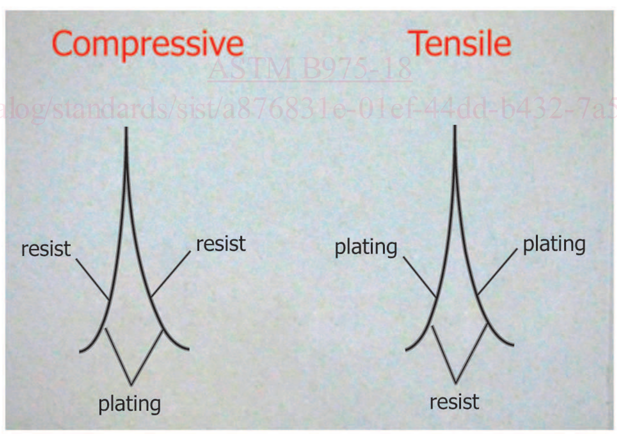

FIG. 2 ~~Deposit Stress Analyzer Measuring Stand~~Compressive and Tensile Stressed Test Strips

4.3.1 The internal deposit stress is calculated based on the total number of increments deflection observed from tip to tip after plating. This value is determined as the test strip is suspended above a measuring stand. See Fig. 3. Results are calculated by use of a simple deposit stress analyzer formula expressed in pounds per square inch. See Eq 2 and Eq 3.

5. Significance and Use

5.1 Internal stress in applied coatings exhibits potential to cause a breakdown of resistance to corrosion and erosion as a result of the formation of fractures from micro-cracking and macro-cracking within the applied coating. This phenomenon can also cause blistering, peeling, reduction of fatigue strength, and loss. The resulting stress can be tensile in nature, causing the deposit to contract, or compressive in nature, causing the deposit to expand.

FIG. 3 In-site ~~1 Plating~~ Device

5.2 To maintain quality assurance by the bent strip method, it is necessary to monitor production processes for acceptable levels of internal deposit stress in applied coatings. Note that the highest value of the internal deposit stress as obtained on a ~~stress-versus-coating-thickness~~stress-versus-plating-thickness curve is usually the truest value of the internal deposit stress. Most low values are false. Initial values tend to be lower than the actual value because of the effect of stock material edge burrs and the resistance of the stock material to bending. Excessive deposit thickness causes lower-than-true ~~values~~value since the coating overpowers and changes the initial modulus of elasticity of the test piece, which becomes more difficult to bend as the coating continues to build upon it. This phenomenon can be corrected considerably by use of a formula that compensates for modulus of elasticity differences between the deposit and the substrate materials, but it does remain a factor. See Eq 2.

6. Apparatus

6.1 *Deposit Stress Analyzer Measuring Stand*—This stand has a scale over which a test strip is suspended to determine the increments of spread as the value of *U* between the test strip leg tips caused by the induced deposit stress. See Fig. ~~2~~4. See Eq 1 and Eq 2.

6.2 *~~On site~~ In-site Plating Device for ~~In-Tank~~In-tank or Laboratory Bench Plating (External Anodes)*—This device does not hold a plating bath. It is a ~~1~~0.875 in. (2.22 cm) diameter, cylindrical tube that is designed with an adjustable bracket to enable placement of the cell in a working tank as a permanently mounted fixture. It is also amenable to laboratory studies where small solution volumes are advantageous. See Fig. ~~4~~3. This device supports a single test strip during the deposition process. To electroplate a test strip, the existing tank anodes may be used for the test if they are of similar composition and size and are located equally distant and parallel to the device open ports. Using a rectifier that is separate from the power supply used to plate the parts, connect the positive ~~outlet~~lead to each of the two selected tank anodes, and the negative ~~outlet~~lead to the top of the test strip at the crossbar that extends over the top of the device. The bottom of the device is sufficiently closed to prevent the test strip from dropping through. It is critical that the test strip legs do not pass through the side openings as a test strip is placed inside the device. Adjust the test strip into position against the bottom of the device and approximately 4 in. (10 cm) below the solution level. A 0-1 to 0-2 amp output constant ~~amperage,~~ amperage and constant voltage power supply is recommended to control the amperage accurately. The negative lead from a power supply is then connected to the test strip at the crossbar located at the top of the device. When using deposition conditions similar to work that is processed in the work tank, the stress measurement result will represent the condition of the work being processed. The device may also be used on a laboratory table in a container for a plating bath as small as 400 mL in which two small nickel anodes are positioned each across from a ~~cell~~device side opening. See Fig. ~~4~~3. This becomes helpful and economical when the plating solution is undergoing laboratory studies in regard to additions of multiple additives, particularly if precious metals are involved. In-tank deposit stress testing yields similar results to those determined on a laboratory bench setup when the test parameters are similar. However, the deposit stress will vary over a given part, particularly over parts that are electroformed where the low-current density area deposits usually exhibit the highest deposit stress. In such cases, the determined deposit stress becomes an approximate average value that serves as a quality control procedure.

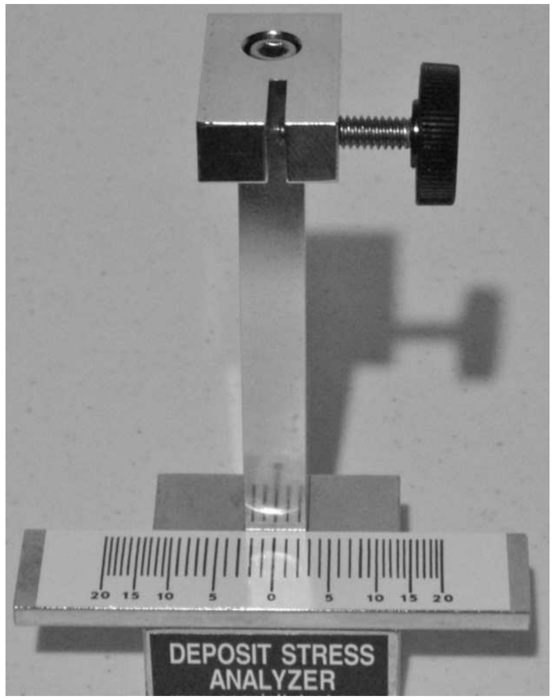

FIG. 4 ~~Compressive and Tensile Stressed Test Strips~~Deposit Stress Measuring Stand

6.3 *Cells for ~~In-Tank~~In-tank Plating or Laboratory Bench Plating (Internal Anodes)*—When agitation and solution temperature are not needed for tests, a test plating cell that includes two anodes of similar size and composition at an equal distance from the test piece is recommended. When solution agitation and elevated bath temperature are required, a two-section cell could be used where one side has a pump and heater. Cells with open low side ports would permit immersion into a working bath allowing the cell to fill as it is being lowered. The test strip ~~must~~shall have its own power supply. In these cells, a test strip is suspended at the center of the cell by clipping ~~it~~ its top end to a stainless steel cross support bar. Two anodes 2⅜ × 2⅜ × ⅛ ~~1/8 in. (5 × 5~~ in. (6 × 6 × 0.3 cm) are positioned along the end of the cell walls where anode pockets are attached. These cells can be designed to be hung directly in a working tank or they could be used in a laboratory setup.

6.4 *Anodes*—When using the deposit stress analyzer method to evaluate the internal deposit stress by electroplating a given metal or metal alloy deposit, two anodes of similar size, shape, and composition are placed at a similar distance from the test strip in a position parallel to the test strip to allow equal exposure of the test strip to the negative current. The positive lead from the power supply shall be connected to each anode.

6.5 *Container*—For tabletop setups, a suitable container can be used to hold a plating bath selected for evaluation when using the in-tank plating cells that have bottom holes for solution flow.

6.6 *Test Strips*—Test strips are used to receive an applied coating that is under investigation for the determination of internal deposit stress. Test strips are shaped similar to a tuning fork so that the test strip legs exist in the same plane geometrically. During the application of a stressed coating, the test strip legs deflect outward in opposite directions. They are made from materials that exhibit spring-like properties so the plated test strip legs will return to the as-plated position if deflected or disturbed by minor mishandling before the degree of deflection is determined. Each test strip is selectively covered with an organic material that is resistant to attack by most solutions to which the test strips are exposed. This coating serves as a mask to define the area to receive ~~metallic deposits~~deposit materials for tests. See Fig. 1.

NOTE 1—Strong alkaline solutions could dissolve away the resist material that covers the areas that do not receive the deposit. If this occurs, a thin coat of high-solids, air-dry lacquer such as Micro-Shield diluted with acetone in a one-to-one ratio is applied by an artist brush over that specific area. When dry, the test can proceed. If lacquer is removed during the test, oven baking at ~~180°F (82°C)~~180 °F (82 °C) for two hours will increase the adhesion of the lacquer.

NOTE 2—If the deposit stress is tensile in nature, the test strip legs will deflect with the deposit facing outward. If the deposit stress is compressive, the deposit will face inward. See Fig. 2.

NOTE 3—After a test has been completed, a measurement of total deflection at the test strip leg tips is determined and the stress value is calculated by the use of a simple equation. See Eq 2.

6.7 *Copper-iron Alloy Test Strips*—These strips are made from UNS Alloy C19400-H02 material. These are ~~0.002~~0.00200 ± 0.00005 in (0.00508 ± 0.000127 cm) thick. They are applicable for determining internal deposit tensile or compressive stress values between ~~1000~~1500 and 145 000 psi (6.9 and 1000 MPa). When used to evaluate chemically induced electroless deposits, a watts or sulfamate nickel strike for ~~5~~30 s at ~~0.21~~0.25 amps, ~~25~~33 asf ~~(2.5 amps/dm²)~~ may be required to activate the surface for metallic deposition.

6.8 *Pure Nickel ~~89~~99 % ~~Cold-Rolled~~Cold-rolled Test Strips*—These test strips are 0.0011 ± 0.00005 in. (0.00279 ± ~~0.000 127~~0.000127 cm) thick. They are useful for internal deposit tensile or compressive stress values between ~~1000 and 145 000 psi (6.9 and 1000 MPa).~~ 200 and 60 000 psi (1.38 and 413.69 MPa). They are the most sensitive test strip choice for low stress conditions and have a wide range of applications, the primary one being electroless nickel deposits that can be applied by a chemical reduction process. For some bath formulations, an activation step may be required, such as a brief dip in diluted hydrochloric acid or plated in a woods nickel strike. When these test strips are used for testing nickel deposits, in a nickel-over-nickel situation, the substrate has little influence on the initial internal deposit stress of the applied coating.

7. Preparation of Test Strips for Calibration and Use

7.1 Test strips must be in a precleaned condition with soils and oils removed prior to plating. Immerse the areas for coating on the test strip legs in a mild aqueous soak cleaning solution for 30 s. This step is followed with a water rinse. Immerse the test strip in a dilute mineral acid solution such as 10 % by volume hydrochloric acid for 30 s to remove surface oxides, and then water rinse.

8. Calibration of Test Strips

8.1 To determine the internal deposit stress in metallic coatings applied to test strips, it is necessary to establish a standardized deposit stress value from which a constant designated as *K* can be assigned. This value includes and combines the various forces that induce stress and strain and influence the bendability of the test strip legs. When used to determine stress values, each material lot of test strips manufactured responds differently because of slight ~~variances in stock material edge characteristics, small~~ variations in stock thickness that occur during the rolling process, temper, and particularly the large differences in material percentages of elongation over the 3 in. (7.6 cm) length of the test strip legs. To compensate for these differences, the constant is designated as *K* ~~is determined by the supplier~~ in a certified manner for each material lot of test strips manufactured. Test strips are calibrated by the manufacturer as a ~~two step~~ two-step procedure where the deposit stress of a selected nickel plating bath is used to plate ~~five~~three test strips and ~~three helices. The supplier determines the value of~~ two helices. ~~K after the deposit stress is known.~~ This *K* value is included in the formulas that are used to determine the internal stress of applied coatings in pounds per square inch.

~~NOTE 4—Calibration of test strips at the work place is not necessary.~~

8.2 When the internal deposit stress value has been determined ~~by obtaining the average of three stress test results performed by plating helices using the spiral contractometer method, the average internal deposit stress test strip spread for five test strips has been determined, the average deposit thickness for both test methods has been calculated, and the results of both methods have been averaged together as the value~~as in 8.1 ~~for~~the ~~S, the bent test strip value for the~~ constant *K* can be obtained using the following formula:

~~$K = 3TS \div UM$ (1)~~

$$K = 3TS \div UM \quad (1)$$

where:

K = calibration constant,
T = average deposit thickness in inches,
S = internal deposit stress as psi, as determined by use of a spiral contractometer test method,
U = average number of increments spread between the test strip leg tips as measured over the deposit stress analyzer scale, and
M = correction for modulus of elasticity differences = modulus of elasticity of the deposit ÷ modulus of elasticity of the substrate (see Table 1).

8.3 To determine the *K* factor calibration constant in the split strip equation, $K = 3TS/UM$, a high nickel sulfate, low nickel chloride, and low boric acid chemistry is used. For bath makeup, slowly add to 0.80 gal (3030 mL) deionized water at ~~140°F (60°C)~~140 °F (60 °C) with stirring 57 oz/gal (1615 g/gal) ~~reagent-grade~~reagent-grade, nickel sulfate, 4.4 oz/gal (125 g/gal) reagent-grade nickel chloride, and 6 oz/gal (170 g/gal) reagent-grade boric acid. Adjust the pH to 3.9 to 4.0 using 10 % by volume reagent-grade ~~sulfuric acid or 20 % reagent-grade sodium hydroxide~~sodium hydrozide solution as appropriate, then add deionized water to a final solution volume of 1 gal (3785 mL).

NOTE 4—The above information explains the process by which "*K*" is determined. The Test strip "*K*" value will be supplied by the manufacturer.

8.4 When the value for *K* is determined by use of the Spiral Contractometer Method (see Test Method B636, Appendix X1), subsequent tests in the field can be made to determine stress in deposits using the following formula:

~~$S = UKM \div 3T$ (2)~~

$$S = UKM \div 3T \quad (2)$$