Designation: B975 − 22

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 split strip method provides a determination of the internal tensile and compressive stress in metallic and nonmetallic coatings. Internal stress is expressed in pounds per square inch or megapascals.

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 200 psi to 145 000 psi (1.38 MPa to 1000 MPa).

1.2 *Units*—The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other. Conversion between unit systems may result in errors that can cause confusion and should be avoided.

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, health, and 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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *average deposit thickness, n*—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 2).

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 Section 10.

3.1.3 *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.

3.1.3.1 *compressive stress (-), n*—stress that tends to cause a deposit to expand.

[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.

*A Summary of Changes section appears at the end of this standard

3.1.3.2 *tensile stress (+), n*—stress that tends to cause a deposit to contract.

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

3.1.4 *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).

3.1.5 *sample test strip, n*—test strip that is used to set the desired amperage on the power supply. This can be a previously used test strip.

3.1.6 *units spread, n*—the amount of deflection between test strip legs is the value of U. Plating test should be continued until the test strip legs deflect from 2-20 total units spread for the most accurate results. The U is measured on the measuring stand.

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[3]; Heussner, Balden, and Morse[4]; and Brenner and Senderoff[5] 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 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 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. 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.

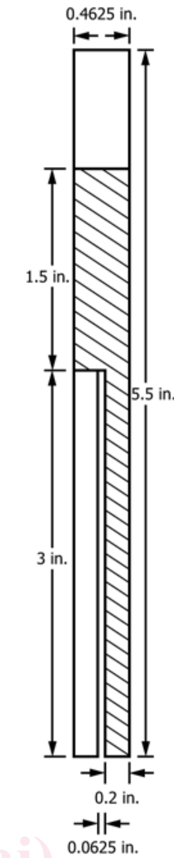

FIG. 1 Test Strip Parameters
1 in. = 2.54 cm

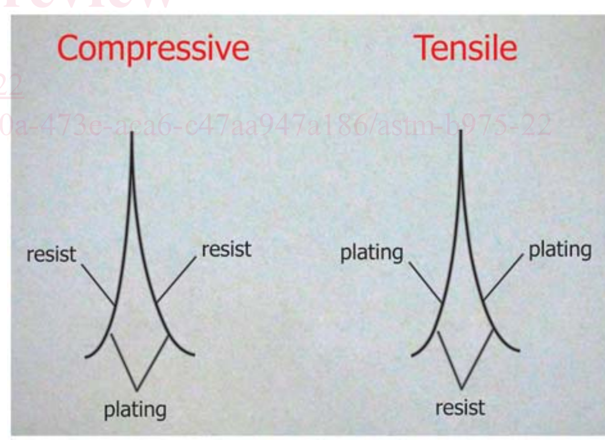

FIG. 2 Compressive and Tensile Stressed Test Strips (provided by the Specialty Testing and Development Company, PO Box 296, Seven Valleys, PA 17360)

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 split strip formulas expressed in pounds per square inch. (See Eq 1 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

[3] Stoney, G. G., *Proceedings of the Royal Society A*, Vol 82, No. 172, 1909, p. 553.

[4] Heussner, Balden, and Morse. "Some Metallurgical Aspects of Electrodeposits," *Plating*, Vol 35.

[5] Brenner, A., and Senderoff, S. *Journal of Research of the National Bureau of Standards*, Vol 42, No. 89, 1949.

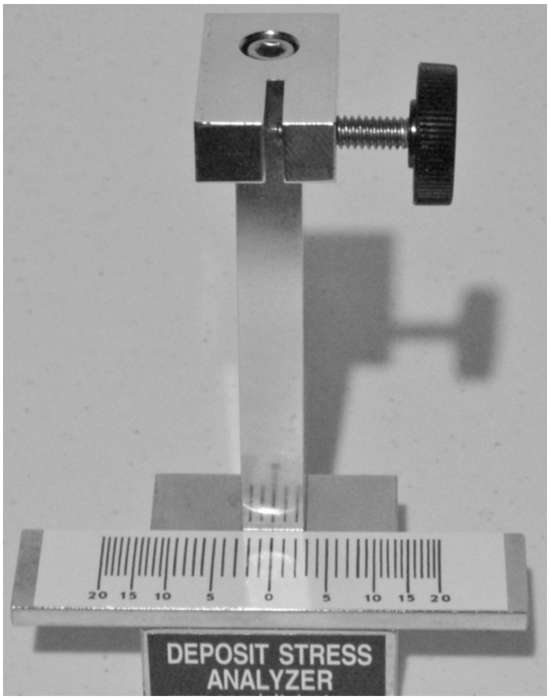

FIG. 3 Deposit Stress Measuring Stand (provided by the Specialty Testing and Development Company, PO Box 296, Seven Valleys, PA 17360)

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.

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. 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 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 3.

NOTE 1—The highest value of the internal deposit stress as obtained on a stress-versus-plating-thickness curve is usually the truest value of the internal deposit stress.

6. Apparatus

6.1 *Measuring Stand*—This stand has a logarithmic scale over which a test strip is suspended to determine the units spread as the value of *U* between the test strip leg tips caused by the induced deposit stress. (See Fig. 3, Eq 4, and Eq 3.)

6.2 *In-site Plating Device for In-tank*—This device is a 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 adaptable to laboratory studies where small solution volumes are advantageous. See Fig. 4. This device supports a single test strip during the deposition process.

NOTE 2—Anodes are located external to the In-site Plating Device.

6.3 *Cells for In-tank Plating (Internal Anodes)*—A test plating cell that includes two anodes of similar size and composition at an equal distance from the test strip, which can be immersed into a working bath. The test strip shall have its own power supply.

6.4 *Cells for Laboratory Bench Plating (Internal Anodes)*—A two-section cell used in a laboratory. Where one section holds the test strip, and two anodes of similar size and composition at an equal distance from the test strip, and the other section has a pump and heater. The test strip shall have its own power supply.

6.5 *Anodes*—When using the split strip 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 and parallel to the test strip to allow equal exposure to the negative current.

6.6 *Power Supply*—Rectifier to supply amperage for plating.

6.7 *Test Strip*—A metal strip formed from flat stock that receives the plating of material being evaluated for internal deposit stress. During plating, if the test strip has two legs they will deflect outward in opposite directions because of their spring-like properties. Each test strip should be selectively coated with a material that is resistant to attack by most solutions. This coating serves as a mask to define the area to receive deposit materials for tests. See Fig. 1.

FIG. 4 In-site Device (provided by the Specialty Testing and Development Company, PO Box 296, Seven Valleys, PA 17360)

NOTE 3—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 4—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 equations. See Eq 3.

6.8 *Copper-iron Alloy Test Strips*—These strips are made from UNS Alloy C19400-H02 material. They are 0.00200 in. ± 0.00005 in. (0.00508 cm ± 0.000127 cm) thick and are applicable for determining internal deposit tensile or compressive stress values between 1500 psi and 145 000 psi (6.9 MPa and 1000 MPa).

6.9 *Pure Nickel Test Strips*—These test strips are 0.0011 in. ± 0.00005 in. (0.00279 cm ± 0.000127 cm) thick, and they are useful for internal deposit tensile or compressive stress values between 200 psi and 60 000 psi (1.38 MPa and 413.69 MPa).

6.10 *Temperature Controller*—A devise that regulates the temperature of the plating solution.

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

7. Equipment Set Up For Laboratory Settings (See Fig. 5)[6]

7.1 Plug the rectifier into the automatic timer.

7.2 Place the anodes in the double section plating cell anode pockets and connect the red positive leads to the anodes.

7.3 Place the heater, if needed, in the double section plating cell. Plug the heater into an electrical source or a temperature controller.

7.4 Set the pump, if needed, to its lowest setting and fasten it to the non-testing side of the double section plating cell so the bath will circulate. Agitation in the plating side of the cell must be limited to prevent the test strip legs from swaying to favor one anode over the other.

7.5 Fill the cell with the plating solution to within a ½ in. of the top of the double section plating cell.

7.6 Plug the pump into an electrical source.

7.7 Heat the plating solution to the required operating temperature.

7.8 Connect the red positive lead from the power supply to the anode contact provided on the cell (lug to connect red wires).

7.9 Follow the test procedure (see Section 8 and Fig. 5).

7.10 Then use the black negative lead to fasten a test strip to the test strip clip on the double section plating cell.

8. Test Procedure[7]

8.1 The average of 3 test strips minimum should be used for more precise results. Use the manufacturer's instructions.

8.2 Put a test strip in a soak cleaner solution at 110 °F – 120 °F for 30 seconds, then water rinse.

8.3 Immerse the test strip in a 10 % by volume hydrochloric acid solution for 15 seconds at room temperature, then water rinse and isopropyl alcohol rinse. Dry the test strip gently lying flat with a paper towel and weigh the test strip. Record the individual test strip starting weight (SW) in grams to the fourth decimal place.

8.4 Turn the agitation pump ON and set the timer for the required plating time.

8.5 Attach the weighed test strip so it is centered between the cell walls.

8.6 Set the timer for the desired test time.

8.7 After the test strip is plated, remove the test strip.

8.8 Rinse the test strip in water, and then rinse it in isopropyl alcohol.

8.9 Dry the test strip gently lying flat with a paper towel.

8.10 Place the test strip on the Measuring Stand and record the total units spread as the value for U. This will be used in Eq 3 below. Example: 2.4 + 3.1= 5.5 (U)

8.11 Weigh the plated test strip and record the final weight (see 8.3). Subtract the starting weight (SW) from the final weight (FW) where the difference = W. This is the W value in the T=formula (Eq 2).

$$FW - SW = W \quad (1)$$

9. Test Strip Calculations[8]

9.1 Calculate the deposit thickness for each test strip using the following equation:

$$T = \frac{W}{D(7.74\ \text{cm}^2)(2.54\ \text{cm/in.})} = \text{inches} \quad (2)$$

where:

T = deposit thickness in inches,
W = weight in grams of the deposit,
D = density of the plated material specific gravity as g/cm^3, and
$7.74\ cm^2$ = the plated surface area of the Test strip is 1.2 $in.^2$ (7.74 cm^2).

NOTE 5—The 7.74 cm^2 is based off the measurements in Fig. 1.

9.2 After the deposit thickness and the number units spread has been determined, the deposit stress can be calculated thus:

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

where:

S = stress in pounds per square inch,
U = number of units spread,
T = deposit thickness in inches,
K = the strip calibration constant (provided by the manufacturer), and
M = modulus of elasticity of the deposit divided by the modulus of elasticity of the substrate material (see Table 1).

[6] The content of this section was provided by Specialty Testing and Development Company, PO Box 296, Seven Valleys, PA 17360.

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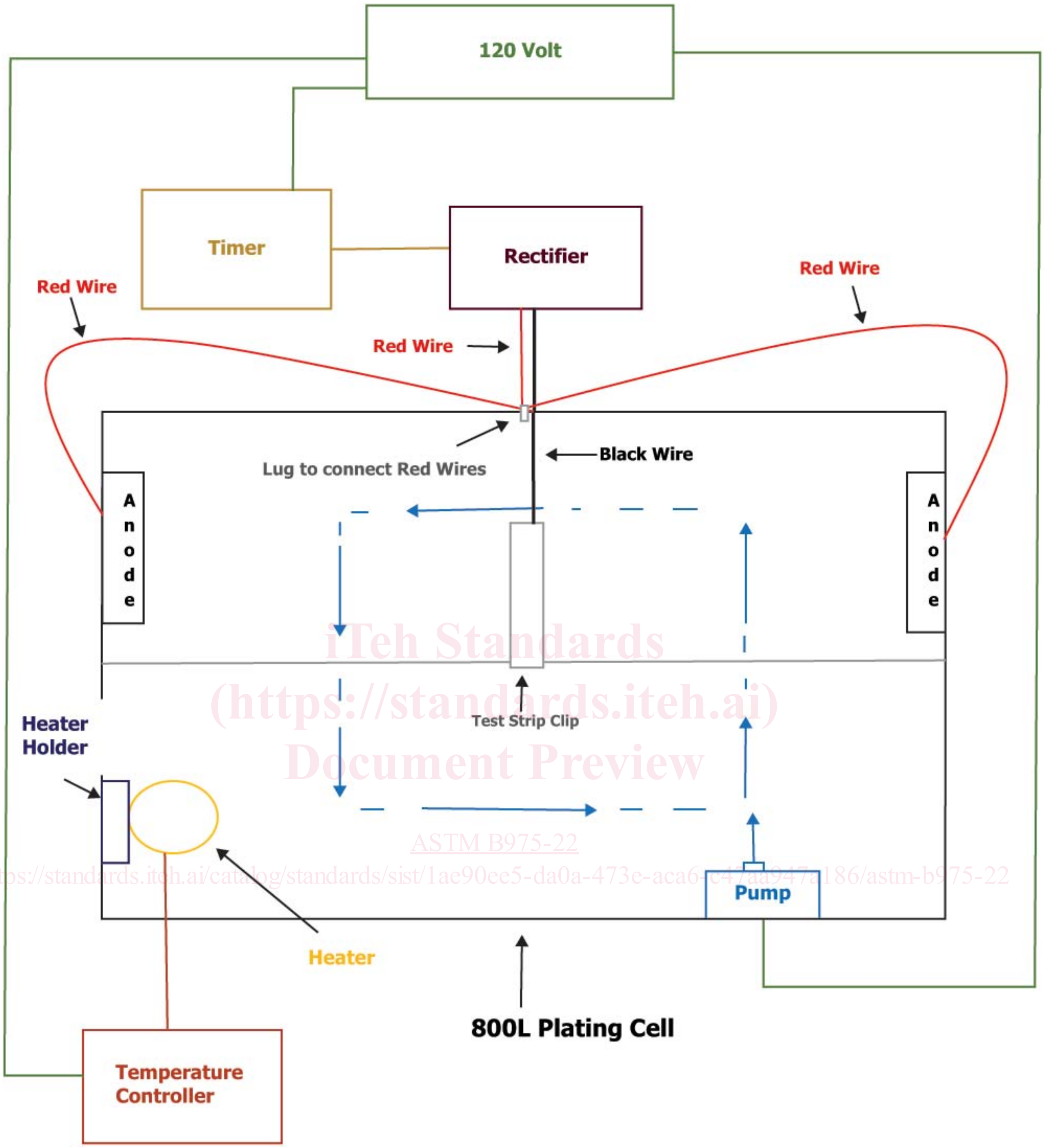

FIG. 5 Double Cell Set Up (provided by Specialty Testing and Development Company, PO Box 296, Seven Valleys, PA 17360)

10. Calibrating The "K" Value for Test Strips

10.1 The "K" Value for test strips is calibrated by the manufacturer as a two-step procedure where the deposit stress of a selected nickel plating bath is used to plate three test strips and two helices. This K value is included in the formulas that are used to determine the internal stress of applied coatings in pounds per square inch.

10.2 When the internal deposit stress value has been determined as in 8.1, the constant "K" can be obtained using the following formula: