



Designation: ~~B975 – 18~~^{ε1} 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

^{ε1} NOTE—Footnote 5 was editorially corrected in February 2020.

INTRODUCTION

The ~~deposit stress analyzer split strip~~ 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—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~~200 psi to 145 000 psi (~~1.38~~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, and values from the two systems shall not be combined. 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

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

[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:*

~~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 32](#)).~~

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 [810](#).

~~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. (2.3 cm) in diameter and 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.~~

3.1.3 *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.

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.

~~3.1.6 *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.~~

3.1.4 *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).

~~3.1.8 *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.~~

~~3.1.5 *sample test strip, n*—metal strip formed from flat stock that receives the coating of material being evaluated for internal stress—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 Houssner;⁴ Heussner, Balden, and Morse;⁴ and Brenner and Senderoff⁵ 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.

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 split strip formulas expressed in pounds per square inch. See (See Eq 21 and Eq 3.)

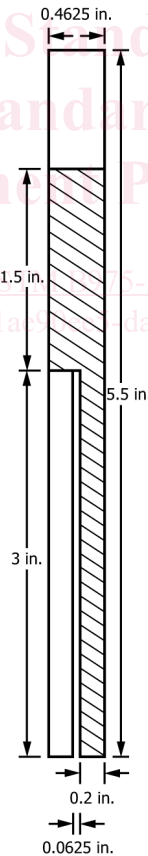


FIG. 1 Test Strip Parameters
1 in. = 2.54 cm

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

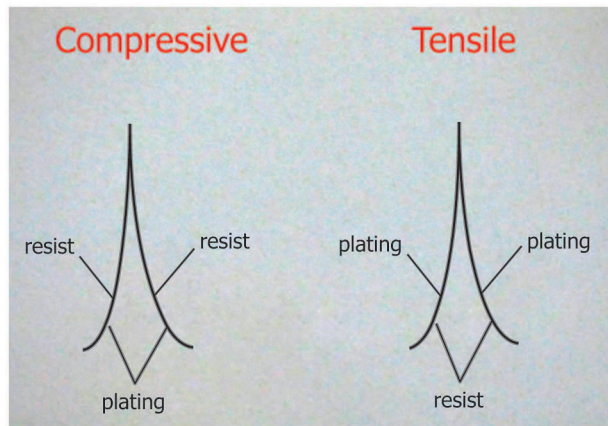


FIG. 2 Compressive and Tensile Stressed Test Strips (provided by the Specialty Testing and Development Company, PO Box 296, Seven Valleys, PA 17360)

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.

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-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 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 23.

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 *Deposit Stress Analyzer-Measuring Stand*—This stand has a logarithmic scale over which a test strip is suspended to determine the increments of units spread as the value of U between the test strip leg tips caused by the induced deposit stress. See (See Fig. 43. See, Eq 14, and Eq 23.)

6.2 *In-site Plating Device for In-tank or Laboratory Bench Plating (External Anodes)*—*In-tank*—This device does not hold a plating bath. It is a 0.875 in. (2.22 cm) diameter, 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 amenable to laboratory studies where small solution volumes are advantageous. See Fig. 34. 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 lead to each of the two selected tank anodes, and the negative 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 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 device side opening. See Fig. 3. This becomes helpful and

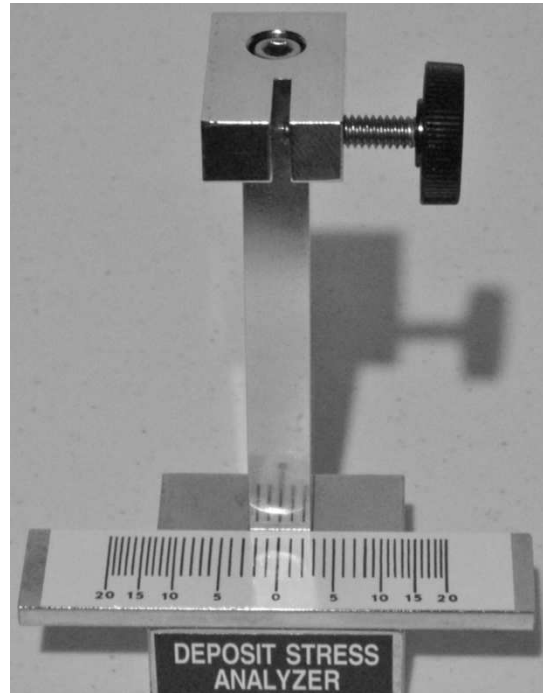


FIG. 43 Deposit Stress Measuring Stand (provided by the Specialty Testing and Development Company, PO Box 296, Seven Valleys, PA 17360)



FIG. 34 In-site Device (provided by the Specialty Testing and Development Company, PO Box 296, Seven Valleys, PA 17360)

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.

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

6.3 Cells for 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 strip, which can be immersed into a working bath allowing the cell to fill as it is being lowered. bath. The test strip shall have its own power supply. In these cells, a test strip is suspended at the center of the cell by clipping its top end to a stainless steel cross support bar. Two anodes $2\frac{3}{8} \times 2\frac{3}{8} \times \frac{1}{8}$ in. ($6 \times 6 \times 0.3$ 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 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 deposit stress analyzer 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 from the test strip in a position and 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.

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

6.7 Test Strips—Strip—Test strips are used to receive an applied coating that is under investigation for the determination of A metal strip formed from flat stock that receives the plating of material being evaluated for internal deposit stress. Test strips are shaped similar to a tuning fork so that During plating, if the test strip legs exist in the same plane geometrically. During the application of a stressed coating, the test strip legs has two legs they will 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. directions because of their spring-like properties. Each test strip is should be selectively covered coated with an organic A material that is resistant to attack by most solutions to which the test strips are exposed. solutions. This coating serves as a mask to define the area to receive 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) for two hours will increase the adhesion of the lacquer.

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 a simple equation. equations. See Eq 23.

6.8 Copper-iron Alloy Test Strips—These strips are made from UNS Alloy C19400-H02 material. These They are $0.00200.00200$ in. ± 0.00005 in. (0.00508 in. (0.00508 cm ± 0.000127 cm) thick. They thick and are applicable for determining internal deposit tensile or compressive stress values between ± 5001500 psi and 145 000 psi ($6.9(6.9$ MPa and 1000 MPa). When used to evaluate chemically induced electroless deposits, a watts or sulfamate nickel strike for 30 s at 0.25 amps, 33 asf may be required to activate the surface for metallic deposition.

6.9 Pure Nickel 99% Cold-rolled Test Strips—These test strips are $0.00110.0011$ in. ± 0.00005 in. ($0.00279(0.00279$ cm ± 0.000127 cm) thick. They thick, and they are useful for internal deposit tensile or compressive stress values between 200200 psi 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. (1.38 MPa and 413.69 MPa).

6.10 Temperature Controller—A device 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. Preparation of Test Strips for Calibration and Use

7.1 Test strips must be in a preecleaned 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.

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. 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 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 in a certified manner for each material lot of test strips manufactured. Test strips are 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.

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

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

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