

# Standard Test Method for Measurement of Internal Stress of Metallic Coatings by Split Strip Evaluation (Deposit Stress Analyzer Method)<sup>1</sup>

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  $(\varepsilon)$  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 to 145 000 psi (3.45 to 1000 MPa).
- 1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered 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:<sup>2</sup>

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

<sup>1</sup> 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.

Current edition approved Nov. 1, 2015. Published December 2015. DOI: 10.1520/B975-15.

<sup>2</sup> 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.

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

2.2 *IEC Standard:*<sup>3</sup>

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 3).
- 3.1.2 *constant K, n*—this certifiable calibrated number is determined experimentally for each lot of test strips manufactured to enable simple mathematical calculation of the internal deposit stress while factoring the influence of percent elongation difference between the deposit and the substrate without the use of complicated bent strip formulas. See Note 4.
- 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 0.61 in. (15.5 cm) long with or without a polytetrafluoroethylene (PTFE) coating on the inside surface.
- 3.1.4 *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.
  - 3.1.4.1 Discussion—Stress that develops in a given layer of

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

material is measured as pounds per square inch or megapascals where 1 MPa = 145 psi.

- 3.1.5 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.
- 3.1.6 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, Houssner, Balden and Morse, 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 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

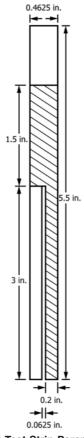


FIG. 1 Test Strip Parameters 1 in. = 2.54 cm

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. 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.

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.
- 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 curve is usually the truest value

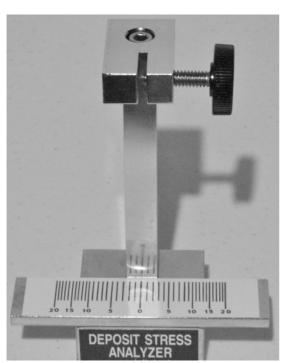


FIG. 2 Deposit Stress Analyzer Measuring Stand



FIG. 3 In-site 1 Plating Device

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 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. See Eq 1 and Eq 2.
- 6.2 On site Plating Device for In-Tank or Laboratory Bench Plating (External Anodes)—This device does not hold a plating bath. It is a 1 in. 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. 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 to each of the two selected tank anodes, and the negative outlet 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

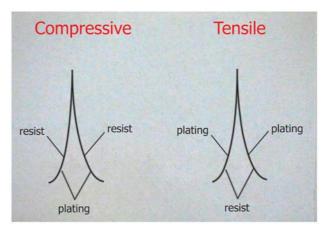


FIG. 4 Compressive and Tensile Stressed Test Strips

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, 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 side opening. See Fig. 4. 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.

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 into a working bath allowing the cell to fill as it is being lowered. The test strip must have its own power supply. In these cells, a test strip is suspended at the center of the cell by clipping it to a stainless steel cross support bar. Two anodes  $2 \times 2 \times \frac{1}{8}$  1/8 in. (5 × 5 × 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 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 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  $\pm$  0.00005 in (0.00508  $\pm$  0.000127 cm) thick. They are applicable for determining internal deposit tensile or compressive

stress values between 1000 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 s at 0.21 amps, 25 asf (2.5 amps/dm²) may be required to activate the surface for metallic deposition.

6.8 Pure Nickel 89 % Cold-Rolled Test Strips—These test strips are  $0.0011 \pm 0.00005$  in.  $(0.00279 \pm 0.000127$  cm) thick. They are useful for internal deposit tensile or compressive stress values between 1000 and 145 000 psi (6.9 and 1000 MPa). 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 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 designated as K is determined by the supplier in a certified manner for each lot of test strips manufactured. Test strips are calibrated as a two step procedure where the deposit stress of a selected nickel plating bath is used to plate five test strips and three helices. The supplier determines the value of 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 for S, the bent test strip value for the constant K can be obtained using the following formula:

$$K = 3 TS \div UM \tag{1}$$

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  $\div$  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  $140^{\circ}F$  (60°C) with stirring 57 oz/gal (1615 g/gal) 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 solution as appropriate, then add deionized water to a final solution volume of 1 gal (3785 mL).

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:

$$S = UKM \div 3T \tag{2}$$

8.5 To calibrate and certify test strips as a manufactured lot, determine the average internal deposit stress of three helices plated on a spiral contractometer and five test strips of the selected material plated in a divided two-section cell with heater and pump in one section and two nickel anodes and a test strip in the other section. Fill the cell to a depth of 3.5 in. (9 cm) with the chosen nickel plating solution. During the test, a pump should provide minimal solution agitation so as not to

TABLE 1 Values for M to Determine Compressive and Tensile Deposit Stress for Various Deposited Coatings

	-		-	
Stock Material		1194	2042B	270NI
$E^{A}$		120 700	144 800	206 900
Stock Thickness, in.		0.0020	0.0015	0.0011
Metal	M <sup>B</sup>	Values for <i>M<sup>C</sup></i>		
Cadmium	31 720	0.26	0.22	0.15
Chromium	248 280	2.1	1.7	1.2
Cobalt	206 897	1.72	1.43	1.0
Copper	117 240	0.97	0.81	0.57
Gold	74 480	0.62	0.51	0.36
Nickel	206 900	1.7	1.42	1.0
Platinum	146 900	1.2	1.02	0.71
Rhodium	289 650	2.4	2.0	1.4
Silver	75 860	0.63	0.52	0.37
Tin	41 370	0.34	0.29	0.2
Zinc	82 760	0.69	0.57	0.4

<sup>&</sup>lt;sup>A</sup>Modulus of elasticity of the substrate material as MPa.

<sup>&</sup>lt;sup>B</sup>Modulus of elasticity of the deposit as MPa.

 $<sup>^{</sup>C}\!Modulus$  of elasticity of the deposit as MPa  $\div$  modulus of elasticity of the substrate as MPa for deposit stress analyzer.

bend the suspended test strip forward. A temperature controller is recommended for critical or certification purposes. The controller probe should be positioned on the test strip side of the cell to maintain the temperature within  $1.5^{\circ}$  F  $(0.5^{\circ}$  C). See 3.1.9, 6.3, and 8.3.

- 8.6 Clean five test strips in an appropriate non-alkaline soak cleaner solution, water rinse, 70% isopropyl alcohol rinse, and blot dry with blotting paper.
- 8.7 Weigh the test strips to the fourth decimal place and record their weights.
- 8.8 Suspend a test strip in the plating cell and attach it with the negative clip from the power supply to the plating cell stainless steel crossbar with the test strip legs centered and barely touching the bottom of the plating cell. About 1/8 in. (0.32 cm) of the test strip will extend beyond the top of the support bar.
- 8.9 Plate each test strip individually in the standard nickel plating bath for 4 min at 0.25 amps, 30 ASF, to obtain a deposit thickness approximately 0.00010 in. (0.00025 cm) thick. See 8.3.
- $8.10\,$  Rinse each test strip after the plating step in water, 70% by volume isopropyl alcohol and blot dry.
- 8.11 Approximately 2 m after the end of the plating cycle, suspend each test strip on the test strip measurement stand and measure the total units spread between the tips of the test strip legs to the right and left of zero. Record this value as the value of U.
- 8.12 For each test strip plated, subtract the initial weight in grams as recorded in subsection 8.7 from the nickel plated weights and record the weight of the nickel deposit in grams.
- 8.13 Calculate the deposit thickness for each test strip using the following equation:

$$T = \frac{W}{D(S) (2.54 \quad c \quad m / i \quad n.)} = inches$$
 (3)

where:

T = deposit thickness in inches,
W = grams deposit,
D = specific gravity as g/cm<sup>3</sup> =
8.9 g/cm<sup>3</sup> for nickel
deposits, and
S = deposit surface area as cm<sup>2</sup>

S = deposit surface area as cm<sup>2</sup>. Test strip plated surface area = 1.2 in<sup>2</sup> (7.74 cm<sup>2</sup>).

8.14 Record the deposit thickness for each test strip.

## 9. Spiral Contractometer Method to Calibrate Test Strips

- 9.1 Prepare three helices for plating a nickel deposit. Refer to Test Method B636 Appendix X1, Procedure for Stress Determination of Nickel Electrodeposits.
- 9.1.1 The preparation steps include mounting helices on a spiral contractometer, cleaning them, applying a nickel strike, rinsing and drying them, removing them for weighing and recording their initial weights, remounting on a spiral contractometer, calibrating each helix at temperature in the plating bath, plating each helix individually and recording the *Kt* and *Kc* values, rinsing in water, 70% isopropyl alcohol and

drying them, weighing them and determining the deposit weight gain for each helix tested.

Note 5—Use helices that have the interior surface masked with a Teflon coating.

Note 6—The nickel strike chemistry may be watts or sulphamate nickel.

Note 7—Nickel plate the helices according to the current density that was used for the test strips: 2.8 amps, 30 asf, for 21 min in the standard plating bath operated at the identical test conditions.

- 9.1.2 Determine the plated surface area for each helix as square meters. See Test Method B636, Appendix X1, Procedure for Stress Determination of Nickel Electrodeposits. Calculate and record the internal deposit stress in MPa as the truer internal stress value corrected for the difference in the modulus of elasticity between the deposit and the helix materials.
- 9.1.3 Calculate the nickel deposit thickness for each helix as square metres.
- 9.1.4 Calculate the internal deposit stress for the plated layer on each helix. See formulas 1 and 2 presented in Test Method B636 Appendix X1.
  - 9.1.5 Average these deposit stress readings.

### 10. Determination of the Constant K Value

- 10.1 Calculate the constant K equation (Eq 1).
- 10.2 Average the value for T (see 8.13).
- 10.3 Average the value for S (see 8.4 and Eq 2).
- 10.4 Average the value for U (see 8.11).
- 10.5 Reference a certified copy obtained from the supplier for the substrate modulus of elasticity value as psi. Divide the modulus of elasticity value of the deposited material, in this case nickel, having a value of 30 000 050 psi (206 843 000 kPa) by the modulus of elasticity of the substrate test strip material to obtain the value for M, which is needed to solve Eq 1 and Eq 2.
- 10.6 Substitute the value for S in psi as determined by the spiral contractometer stress test method into Eq 1 and solve for the value of the constant K. This value for K can then be substituted into Eq 2 to determine the internal deposit stress in applied metallic coatings that are being investigated.

### 11. Stress Test Conditions for Coatings Other than Nickel

11.1 Since metallic deposits, whether pure or alloy in composition, applied to a given test strip are influenced in

TABLE 3 Modulus of Elasticity for Electroless Nickel-Phosphorus and Nickel-Boron Alloy Deposits<sup>A</sup>

	Electroless Nickel-Phosphorus Alloy <sup>A</sup>					
% Phosphorus	High	Mid	Mid-Low	Low	Pure	
					Nickel	
	10–13	7–9	4–6	1–3	0	
Modulus of elasticity as psi × 10 <sup>6</sup>	55–70	5–65	45–65	55–65	30	
	Electroless Nickel-Boron Alloy <sup>A</sup>					
% Boron Mid-Low				Low		
		3–5		0.2	2–1	
Modulus of elasticity as psi × 10 <sup>6</sup>		120				

<sup>&</sup>lt;sup>A</sup>Contact the plating chemicals supplier for more exact values or select the average number for the given range.

regard to the effect of the base material on the internal deposit stress of the plated material, it is recommended that, at least initially, coatings be applied for internal deposit stress evaluation at an average thickness value of  $1 \times 10^{-4}$  in. (2.54 ×  $10^{-3}$  cm). Adjust the plating parameters to maintain a test strip leg tip spread within these recommended limits. See 8.12.

11.2 As an aid in determining test deposition time and current for various deposits, the approximate deposition rates for metals common to electroplating processes are listed in Table 2.

## 12. Modulus of Elasticity Calculation for Stress in Electroless Nickel Phosphorous and Nickel Boron Alloy Deposits

12.1 Calculation example: E = 55 000 000 psi =379 310 MPa. See Table 3 for M and E values for alloy deposits. The value, E, divided by 145 = 379 310 MPa, the modulus of elasticity for the plated alloy deposit,  $E_{Deposit}$ . In the deposit stress analyzer formula, S = UKM/3T, the value M = the modulus of elasticity of the deposit,  $E_{Deposit}$ , divided by the modulus of elasticity of the substrate,  $E_{Substrate}$ . Thus, M, the modulus of elasticity of the nickel test strip = 379 310 divided by 206 900 = 1.833. If pure nickel would be plated over a pure nickel test strip, M would equal 1.0. For this nickel phosphorus alloy, however, M equals 1.833, so the actual internal deposit stress is 1.833 times greater than that of a pure nickel deposit applied under similar conditions. Frequently, the erroneous increase in deposit stress value that is caused by failure to factor into the equation the modulus of elasticity differences between the base material and the substrate material is not recognized. In such cases, the calculated result can be far from the actual value. Also, it is recognized that metal deposits that occur at high bath temperatures over substrates of differing composition may experience a change in deposit stress as a plated part cools or the actual stress value may continue to change for as long as several weeks. In such cases, a room temperature water rinse provides cooling of the test piece. For each case, it is recommended that a deposit stress measurement be determined approximately 2 min after the test plating period ends. If this is not practical, at least choose a set time for the reading to occur and remain consistent with this time for quality control purposes.

**TABLE 2 Approximate Deposition Rates for Metals** 

Electrolyte	% Efficiency	Amps	ASF <sup>A</sup>	μin./min
Cadmium	95	0.33	40	26
Chromium	20	2.90	350	10
Copper (cyanide)	80	0.16	20	36
Copper (acid)	98	0.33	40	36.3
Gold (soft)	93	0.08	10	44
Gold (hardened)	40	0.33	40	45
Nickel	95	0.33	40	32
Palladium	95	0.25	30	38
Silver	99	0.16	20	59
Tin (acid)	98	0.25	20	40
Tin (alkaline)	60	0.25	20	24
Zinc (acid)	85	0.25	30	31
Zinc (cyanide)	98	0.25	30	30

 $<sup>^{</sup>A}\!\text{Convert}$  amps per square foot to amps per square decimetre by dividing the numbers shown here by ten.

### 13. Deposit Stress Analyzer Test Strip

- 13.1 Test Strip Parameters—See Fig. 1.
- 13.2 Test Strip Materials—See 6.6-6.8.

### 14. Recording and Calculating Results

- 14.1 Record the bath temperature as degrees Fahrenheit. Maintain the bath temperature as constant as possible, since temperature differences cause considerable change in stress values.
- 14.2 Record the test piece weight before and after the coating is applied, as well as the weight of the coating in grams.
- 14.3 Determine and record the average deposit thickness by use of Eq 3.
- 14.4 Determine the total increments to which the test strip legs have spread by suspending the coated test strip over the measurement scale of a deposit stress analyzer stand and record this value as U.
- 14.5 Determine the deposit stress value and record the results. See Eq 2.

### 15. Precision and Bias<sup>4</sup>

- 15.1 The precision of this test method is based on an interlaboratory study of B975 Test Method for Measurement of Internal Stress of Metallic Coatings by Split Strip Evaluation (Deposit Stress Analyzer Method) conducted in 2013. Six laboratories tested triplicate specimens of both a bright nickel electroplating solution that produced compressive internally stressed deposits and a matte nickel electroplating solution that produced tensile internally stressed deposits. Every "test result" represents an individual determination. Practice E691 was followed for the design and analysis of the data; the details are given in ASTM Research Report No. B08-1007.
- 15.1.1 Repeatability (r)—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would, in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20.
- 15.1.1.1 Repeatability can be interpreted as maximum difference between two results obtained under repeatability conditions that are accepted as plausible because of random causes under normal and correct operation of the test method.
- 15.1.1.2 Repeatability limits are listed in Table 4 and Table 5.
- 15.1.2 *Reproducibility* (*R*)—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in 1 case in 20.

<sup>&</sup>lt;sup>4</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:B09-1007. Contact ASTM Customer Service at service@astm.org.



TABLE 4 Stress, psi

Material	Average <sup>A</sup>	Repeatability Standard Deviation	Reproducibility Standard Deviation	Repeatability Limit	Reproducibility Limit
	$\bar{X}$	$S_r$	$S_R$	r	R
Bright	-9970	735	2160	2060	6060
Matte	20780	994	1780	2780	4980

<sup>&</sup>lt;sup>A</sup>The average of the laboratories' reported averages.

#### **TABLE 5 Stress. MPa**

	Average <sup>A</sup>	Repeatability Reproducibility		Repeatability Reproducibility	
Material		Standard	Standard	Limit	Limit
		Deviation	Deviation	LIIIII	LIIIII
	$\bar{X}$	$S_r$	$S_R$	r	R
Bright	-68.8	4.9	14.8	13.8	41.4
Matte	143	6.7	12.1	18.8	33.9

<sup>&</sup>lt;sup>A</sup>The average of the laboratories' reported averages.

- 15.1.2.1 Reproducibility can be interpreted as the maximum difference between two results obtained under reproducibility conditions, that is, accepted as plausible because of random causes under normal and correct operation of the test method.
- 15.1.2.2 Reproducibility limits are listed in Table 4 and Table 5.

- 15.1.3 The terms "repeatability limit" and "reproducibility limit" are used as specified in Practice E177.
- 15.1.4 Any judgment in accordance with 15.1.1 and 15.1.2 would have an approximate 95 % probability of being correct.
- 15.2 At the time of the study, there was no accepted reference material suitable for determining the bias for this test method; therefore, no statement on bias is being made.
- 15.3 The precision statement was determined through statistical examination of 60 results, from five laboratories, on two materials.
- 15.4 To judge the equivalency of two test results, it is recommended to choose the metallic coating closest in characteristics to the test material.

Note 8—Because of procedural and equipment issues that surfaced during the most recent interlaboratory study (2013), Committee B08 plans to repeat this work at a later date to update the precision and bias section in this test method.

## 16. Keywords

16.1 deposit stress analyzer method; helix; internal deposit stress; metallic coatings; modulus of elasticity; spiral contractometer method; test strip

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