

# **A NEW FRONTIER FOR DEPOSIT STRESS MEASUREMENTS**

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## **ABSTRACT**

Internal stress exists as an inherent force within electroplated and chemically applied coatings, resulting from the inclusion of foreign organic and metallic contaminants. These materials disrupt the orderly building of the respective atoms according to their natural lattice structures and create internal stress. The induced stress can be tensile or compressive in nature, causing the deposit to contract or expand in relation to the basis substrate. High internal stress levels produce fractures in coatings that cause deposit flaking, blistering, peeling, and the distortion of electroformed products. These deposit failures accelerate corrosion of the substrate materials significantly. This paper discloses a new development that enables accurate deposit stress measurements at values near zero stress, whether said stress is tensile or compressive in nature. Common errors and misconceptions in regard to deposit measuring techniques are explained and fallacies concerning the interpretation of test results are also addressed. For example, electroformed nickel deposit stress is frequently being determined at much lower numbers than the actual values. Internal deposit stress values are presented for the following electroplating baths: cobalt-hardened gold, selenium brightened silver (99.9% deposit purity), semi-bright nickel sulfamate with bromide ion, semi-bright acid copper, and bright acid tin. The deposit stress tests determined in this report include the employment of the bent strip, the spiral contractometer, and the stress-meter methods.

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## Introduction and background

All metallic and non-metallic coatings applied by electrolytic or electroless methods to a foreign substrate exhibit a degree of stress that may be compressive or tensile in nature. The degree of stress within these coatings may be small or great. In applications where the stress levels are high, it is common for such coatings to crack, blister and peel away from the substrate material. Thus, protection of the basis material is lost and the rate of basis metal corrosion is accelerated. High stress levels in electroformed deposits can even cause electroforms to warp.

Internal stress as a force within metallic coatings results from the inclusion of materials foreign to the pure element that is being deposited. The foreign matter becomes entrapped in the matrix and disrupts the orderly building of the pure element atoms into their natural lattice structure. As the natural lattice formation is altered, the internal deposit stress increases. This phenomenon has been recognized since the mid-1800s. A given basis material will undergo distortion when a coating of another material is applied to it. The challenge for the plating industry has been two-fold: to minimize coating stress levels and to accurately measure coating stress values.

## Methods of stress measurement

The first attempt to measure stress values in applied coatings was the bent strip method. A coating of known thickness was applied to a strip of material having a known modulus of elasticity, length, width and thickness. The degree of bend experienced by the test strip was then measured. For this method of measurement, equations were proposed to calculate the deposit stress by using the above data. . Figure 1 illustrates curvature of the basis material caused by the application of a stressed coating.

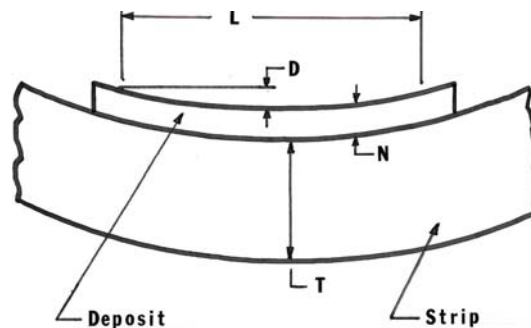


Figure 1. Simple Beam Theory

From this figure, the following equation can be derived to determine the value of coating internal stress.

$$S = \frac{4E(N+T)D}{3NTL}$$

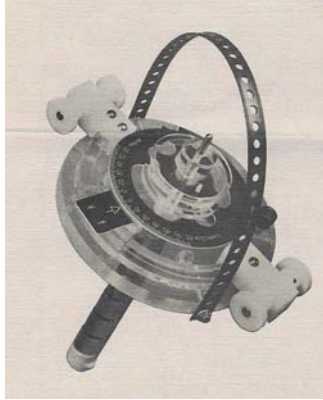
Where,

- N = Thickness of the plated coating (inches)
- T = Thickness of the test strip (inches)
- D = Deflection of the strip due to bending (inches)
- L = Length of the test section (inches)
- E = Modulus of elasticity of the test strip (lb/in<sup>2</sup>)
- I = Moment of inertia of the cross section of the test strip about its neutral axis
- S = Stress in plated layer (lb/in<sup>2</sup>)

More equations for this method are presented in the General Motors Engineering Standards.<sup>1</sup> General Motors used this method for stress tests for many years. However, the measurement of the degree of deflection is difficult to determine and accuracy remains questionable.

Several other methods for deposit stress measurement include the spiral contractometer<sup>2</sup>, and the two disk membrane devices – the stress-meter<sup>3</sup> and its electronic modification. The spiral contractometer (Fig. 2), measures deposit stress levels up to 140 MPa (or 20,000 lb/in<sup>2</sup>) tensile or compressive, but is not applicable in the low stress value range. The spiral basis material is 0.033 cm (0.013 in.) thick. It is made from stainless steel and has an electroplating surface area of about 77 cm<sup>2</sup> (12 in<sup>2</sup>). The manufacturer recommends a deposit thickness of 16 μm (approximately 0.6 mil) for deposit evaluations. A given spiral will contract or expand with use depending upon whether the deposit stress is tensile or compressive in nature. One end of the spiral being utilized is held in a fixed position while the other end is linked to a measuring dial that measures the degrees of spiral movement. The spirals must be calibrated prior to use. This test method is somewhat bulky to use and calculation of the results is time consuming. But the spirals are strippable and can be used for many test determinations as long as the deposit can be removed without destruction of the spiral material.

The stress-meter and its electronic modification (Fig. 3) are rather complicated to use, but this method also permits periodic stripping of the membrane disk. These methods of deposit stress measurement are not applicable for low stress values approaching zero.<sup>4</sup>



**Figure 2. Spiral Contractometer**

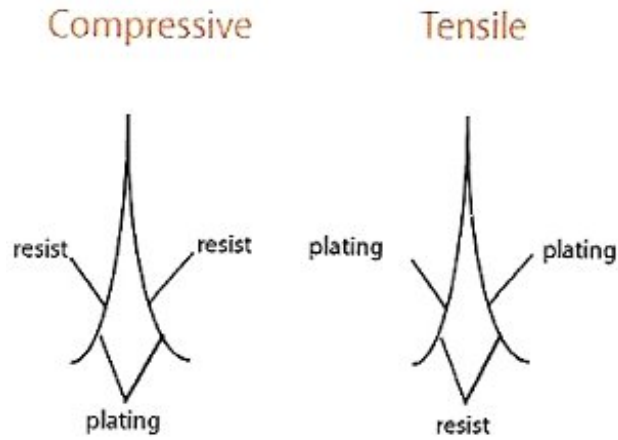


**Figure 3. Stress-Meter**

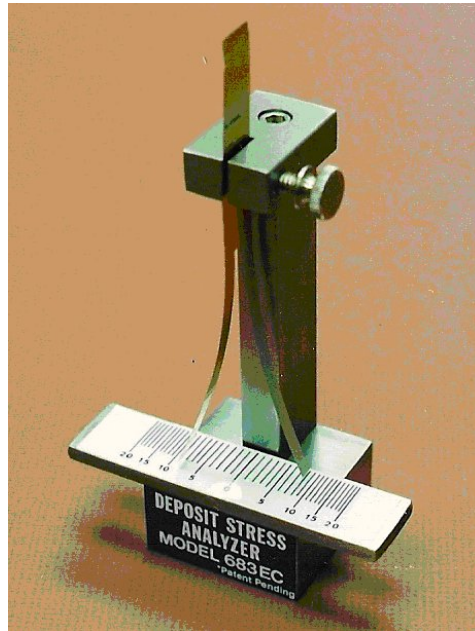
### **Deposit stress analyzer**

A more recent method of determining internal deposit stress values is the Deposit Stress Analyzer. This method utilizes discardable, economic test strips that offer super sensitivity with a capability of measuring a wide range of values from almost zero to 276 MPa (40,000 lb/in<sup>2</sup>). A small surface area of 7.1cm<sup>2</sup> (1.1 in<sup>2</sup>) allows for minimal use of plated deposits, making it a desirable choice for precious metal evaluations. The test strips are calibrated during manufacture, so calibration is not required at the point of use. They offer an easy read of the deflection caused by an applied coating.

The test strips are similar to a tuning fork configuration, having two legs that are placed in a working bath or in a small laboratory set up for use. A solution volume as small as 600 mL is sufficient for test purposes. Since one leg is resist coated on one side and the other leg is coated with resist on the opposite side, deflection of these legs during the deposition of a given coating occurs in opposite directions. If the stress is tensile in nature, the legs spread outward on the coated side of the test strip legs. For compressive stress, the legs spread outward on the resist coated side of the test strip legs (Fig. 4). A measuring stand supports coated test pieces over a scale from which the total increments spread can be determined (Fig. 5).



**Figure 4. Compressive and Tensile Stress**



**Figure 5. The Deposit Stress Analyzer Measuring Stand**

Test strips for the Deposit Stress Analyzer are available as a copper alloy 0.00508 cm (0.002 in.) thick and a nickel iron alloy 0.0038 cm (0.0015 in.) thick. The latter yields the greatest degree of sensitivity and is the best choice for evaluation of low deposit stress values. For a given test result, the total increments spread between the test strip leg tips is designated as the value  $U$ . The formula for calculating the deposit stress of an applied coating is given as follows:

$$S = \frac{U}{3T} K$$

Where: S = Deposit stress (lbs/in<sup>2</sup>)

U = Number of increments spread

T = Coating thickness (in.)

K = Strip calibration constant

The deposit stress in MPa = lb/in<sup>2</sup> divided by 145

The mean coating thickness can be determined by direct measurement of the deposit at the center of a test strip leg, by calculation of the test strip weight gain, or by use of the data given in Table 2. The formula for calculating deposit thickness by weight gain is given as follows:

$$T = \frac{W}{D A}$$

Where: T = Deposit thickness (cm)

W = Deposit weight (g)

D = Deposited metal density (grams/cm<sup>3</sup>)

A = Area of sample (7.74 cm<sup>2</sup>)

The density in g/cm<sup>3</sup> for some of the common metal deposits is listed in Table 1, and the rate of metal deposition is given in Table 2 (1μ-in. = 2.54 x 10<sup>6</sup> cm).

**Table 1. Metal Density Values (Grams / Cubic Centimeter)**

Deposited Metal	Density
Cadmium	8.65
Chromium	7.19
Copper	8.96
Gold (Soft)	19.45
Gold (Hard)	17.60
Nickel	8.90
Palladium	12.00
Platinum	21.45
Rhodium	12.44
Ruthenium	12.20
Silver	10.49
Tin	7.30
Zinc	7.13

**Table 2. Approximate Plating Rates for Common Electrolytes  
(Amps/ft<sup>2</sup> x .108 = Amps/dm<sup>2</sup>)**

Electrolyte	% Efficiency	Amps	ASF	Microinches/Minute
Cadmium	100	.33	40	27.5
Chromium	20	2.90	350	10.0
Copper (ous)	100	.16	20	38.5
Copper (ic)	100	.33	40	37.0
Gold (Soft)	100	.08	10	28.0
Gold (Hard)	40	.33	40	44.5
Nickel	100	.83	100	83.5
Palladium	90	.25	30	35.5
Silver	100	.16	20	60.0
Tin (ous)	100	.42	50	110.0

When the modulus of elasticity of the substrate material is known, equations applicable for simple beam theory (bent strip) such as the Stoney equation<sup>5</sup> can also be utilized.

Several different techniques are available to apply coatings to the Deposit Stress Analyzer test strips. A plating cell with open bottom ports can be placed in a working bath or in a laboratory set up. This cell is equipped with two anodes equally spaced from the test strip that is positioned in its center. The cell contains anode and cathode leads and a place for rectifier engagement.

Another means of plating test strips is by use of the In-Site Plating Cell (Figure 6) invented by Beryl Stein, President of NiCo Form, Rochester, NY. This cell consists of a tube-like structure with side openings and bottom designed to even out the current flow over the exposed test strip legs. The test strip is placed inside the In-Site Cell for deposition of the coating to occur. A 600 mL beaker is sufficient volume for use with the In-Site Cell and said cell assembly can be hung in a working bath such that the anodes are equally spaced from the test strip.



**Figure 6. In – Site Plating Cell.**

With all of the above mentioned methods for the determination of internal deposit stress in applied coatings, numerous false assumptions have masked an understanding of what the most accurate deposit stress value really is for a given set of conditions. This needs clarification and correction. The basic questions to ask are, “What is the actual degree of the deposit stress and how can the correct value be obtained?”

For example, in Table 3 and in Figs. 7-9, the deposit stress values for a given metal coating vary widely with length of deposition time as the only variable. It is recognized that increased deposition time causes greater deposit thickness values. But why are the initial values low, then reach a peak, then fall to lower values again? This phenomenon is observed when determining deposit stress values by all of the methods referenced in the body of this paper. The thinner deposit values appear to be considerably lower than the actual value, and the thicker deposit values appear to be much lower than the actual deposit stress values also.

The reasonable conclusion is that only one of the corresponding deposit stress values for a given coating is close to the actual value. Logically, it follows that the deposit stress actually does not change with plating time as the only variable, but remains constant.

**Table 3. Deposit Stress Test Results**

Nickel Sulfamate Bath, 55°C (130°F), 2.16 amps/dm<sup>2</sup> (20 A/ft<sup>2</sup>)  
(Pounds per square inch divided by 145 = Megapascals)

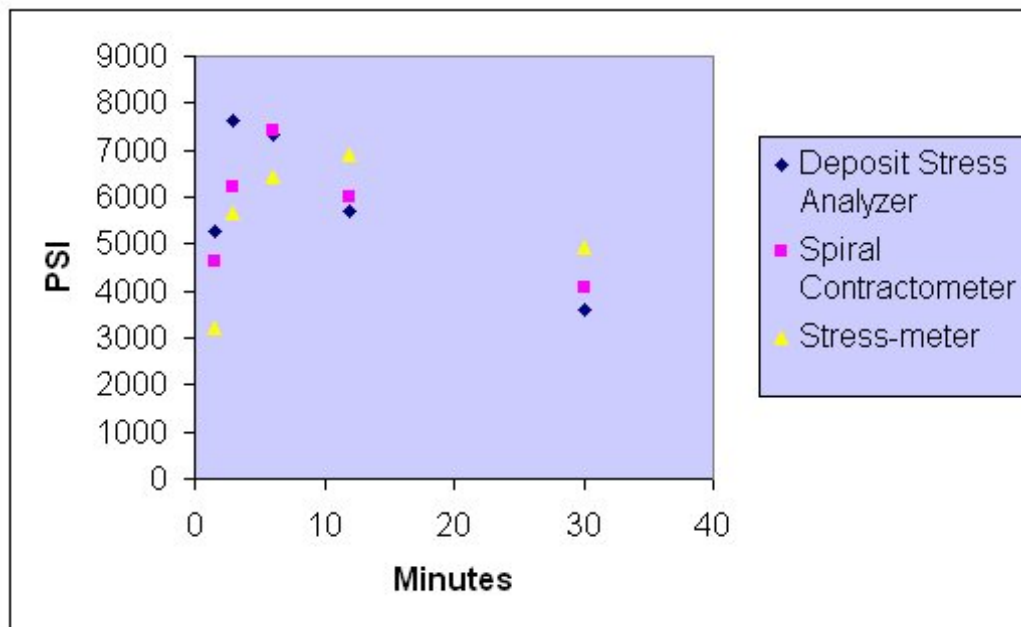
Deposit Thickness, μ"	Time	DSA Test Strip	Units Spread	DSA PSI	Spiral PSI	Stress-meter PSI
25	1.5 min.	steel	1.7	5290	4635	3214
50	3 min.	steel	4.9	7624	6220	5660
100	6 min.	steel	9.4	7313	7415	6410
200	12 min.	steel	14.7	5718	6018	6907
500	30 min.	copper	6.1	3582	4055	4910

Silver Selenium Brightened Bath, 25°C (77°F), 5.4 amps/dm<sup>2</sup> (50 A/ft<sup>2</sup>)

100	0.82 min.	steel	0.5	389	210	
200	1.63 min.	steel	1.9	739	562	
500	4.08 min.	steel	6.7	1043	996	
1000	8.16 min.	steel	11.3	879	909	

Acid Cobalt Hardened Gold Bath, 35°C (95°F), 1.08 amps/dm<sup>2</sup> (10 A/ft<sup>2</sup>)

4	1 min.	steel	0.1	1945		
8	2 min.	steel	0.8	2917		
16	4 min.	steel	1.9	5835		
32	8 min.	steel	4.0	9725		
64	16 min.	steel	7.4	8995		



**Figure 7. Nickel Deposit Stress Values**

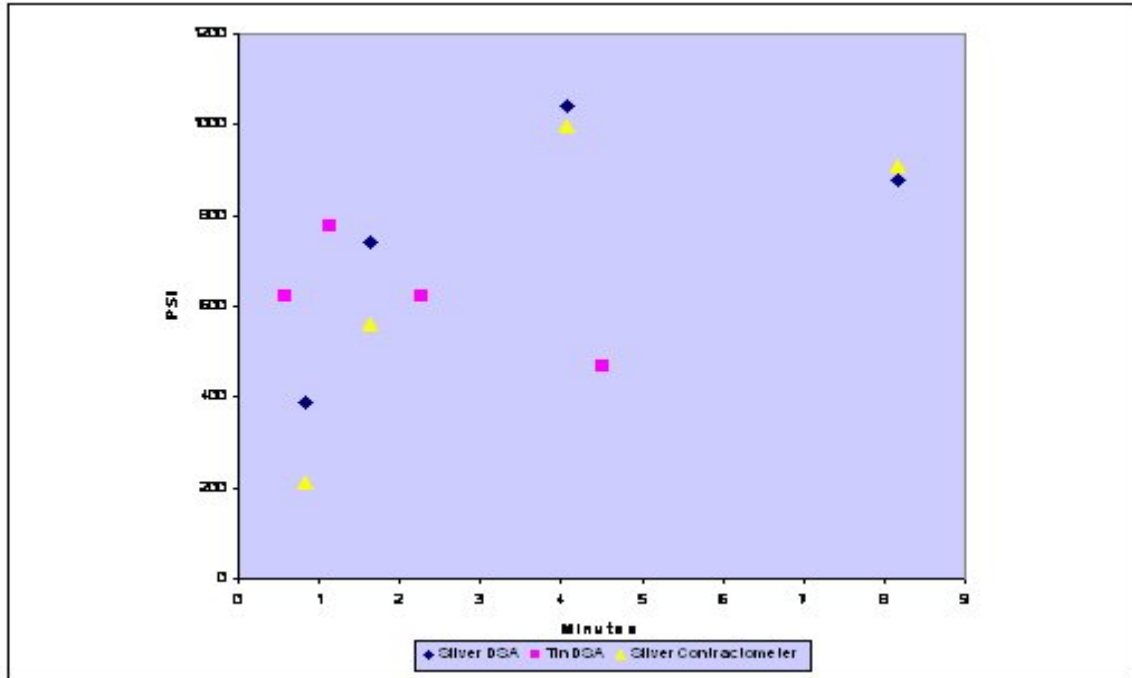


Figure 8. Silver and Tin Deposit Stress Values

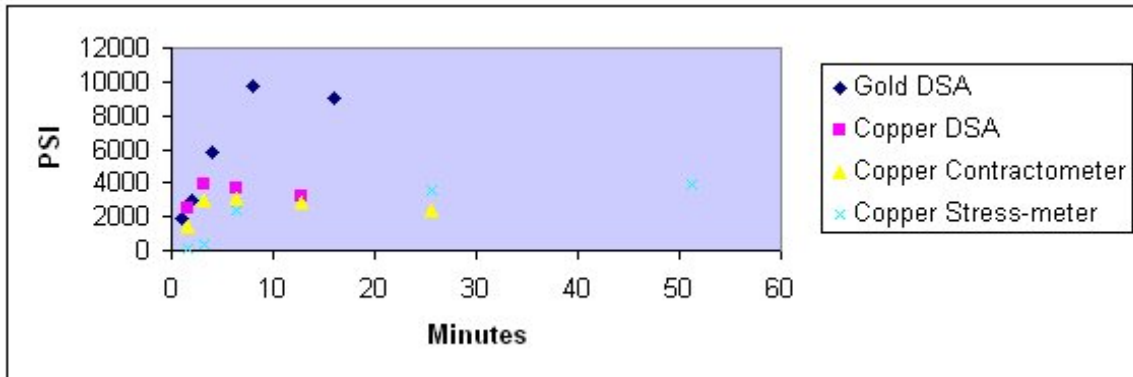


Figure 9. Acid Gold Cobalt and Acid Copper Deposit Values

To answer the questions asked, the change that occurs in the modulus of elasticity as increasing amounts of the plated material are added must be considered.

Increased deposit thickness causes the overall test piece to become more and more resistant to deflection, so the movement of a bent strip or a spiral or a membrane or disk is reduced with increased deposition time. Thus, the modulus of elasticity of the original substrate no longer applies, and the bent strip equations for determining internal deposit stress values based upon the substrate modulus of elasticity reflect increasing error as greater coating thickness is added.

To explain why very thin metallic coatings applied to test pieces using the above mentioned stress evaluation methods seem undervalued, it appears reasonable to assume that initially the deposits lack sufficient energy to overcome the resistance of the test pieces to movement. Said test pieces retain a state of rest or inertia that must be overcome for deflection to occur. Another cause for this phenomenon may be the degree of edge curvature or roughness of the test piece sample.

It is proposed in the context of this paper that the internal deposit stress value, as determined by the test methods discussed here and the equations that apply, is in every case, the test that produces the highest coating stress result. For any meaningful evaluation or test control standard, a plot of the deposit stress should be completed and the conditions that yield the highest value should be noted. Find the parameters and conditions necessary to lower this value to an acceptable range, then standardize these as the test parameters. The true value of internal deposit stress is the highest attainable value observed, which in reality, is the real value for the entire deposit matrix regardless of the coating thickness applied.

Great errors have been reported in deposit stress determinations and gross false assumptions have been believed to be valid to the detriment of the quality of parts being manufactured. For instance, many research and production employees involved in the study or the control of deposit stress levels in the production of electroforms conclude that the deposit stress approaches zero with heavy deposits so “there is little need to be concerned because the internal stress cancels itself out as the deposit thickens.”

The conditions for deposit stress determinations are most frequently selected at random. A test condition is selected that resembles a set of conditions that approximate those for work that is being processed, or a test is chosen because it is easier or more rapid to perform than other test parameters would allow, or testing is not done at all. Deposit stress evaluations are not for the lazy and the ignorant. Do your homework. One must discover and implement the process controls for deposit stress levels that will assure product quality and longevity. Quality parts make happy customers.

## **Conclusion**

The Deposit Stress Analyzer incorporates bent strip technology and affords the most rapid, cost effective technique available for determining deposit stress. A recent improvement in this method has increased measurement sensitivity three fold for accurate determinations at very low deposit stress levels. This method uses pre-calibrated test strips that become deflected by the internal stress exhibited in a wide range of applied coatings. The degree of deflection is easily determined by measurement, and the respective coating stress values can quickly be obtained by using a simple formula that is presented for this purpose. It is no longer necessary to pile up rejected parts through failure to monitor internal stress levels for applied coatings. Regardless of the method selected for control of deposit stress, tests should be made with sufficient frequency to enable the desired quality assurance.

## References

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