The Measurement of Internal Stress of Electroplated Deposits

by David Crotty*

Introduction

Electroplated metal deposits develop internal stress during and after the electroplating process. This property has been discussed in a number of standard references[1,2] and Weil[3] discussed this property in detail for a range of electroplated deposits. The internal stress of a deposit can in some cases be the deciding factor to determine the suitability of a deposit for a particular application. Sulfamate nickel[4,5] and electroless nickel[6] deposits are sometimes chosen based on this property. In addition, the measurement of internal stress can be an important troubleshooting tool for a number of processes, the most important of which are bright electroplated nickel, sulfamate nickel, electroless nickel and zinc.

A number of mechanical methods have been used to measure the internal stress of electroplated deposits. X-ray diffraction has also been used for this measurement.[7] One example of a study using x-ray diffraction to study the internal stress of zinc-nickel deposits is provided recently by Garcia.[8]

Perhaps the most commonly used mechanical method in the industry is the spiral contractometer as described by ASTM B 636.[9] The spiral contractometer has been demonstrated to be reliable but usually uses a stainless steel helix that is unsuitable for a wide range of deposits. Zinc and zinc alloys, for example require a nickel strike to obtain adhesion. One study of zinc deposits[10] used a mild steel helix that was manufactured especially for that study. However, the manufacturer reported technical difficulties in making these helixes and declined to continue to make this item. Crotty and Greene[11] studied zinc deposits using the stainless steel helix with some success but there was some concern that the nickel-zinc interface could be affecting the magnitude, if not the direction, of the measured internal stress.

Nuts & Bolts: What This Paper Means to You

Measurement of internal stress has been a complex matter since someone found that it was critically important to measure it. At last, a simple method shows promise, and the author describes the design of the instrument and gives a few examples of measurements are provided to illustrate the technique and the results that can be expected.

Two methods have been described that use mild steel strips to study the internal stress of deposits. The first method, presented independently by Soderberg and Phillips,[12,13] calculates the internal stress of a deposit plated on one side of a mild steel strip by measuring the extent of the bend from the center of the strip. General Motors subsequently adopted that method as GM 4453P[14] and designed an instrument set. The set included a pair of plastic blocks to hold the strip so that only the exposed area is plated, as well as a holder for the strip that allows the bend distance to be measured with a micrometer. This method was used by Crotty[15] to determine the internal stress of zinc-nickel deposits as part of a larger study of the properties of this deposit. During that study it was found that the method required that two sets of clamps had to mate exactly to avoid actually bending the strip. For this reason, the direction of the stress was reported in that study, but not the magnitude.

The second method was proposed by Bartlett[16] in which the strip is also plated between a pair of plastic blocks so that only one side of the strip is plated. However, the extent of the bend is measured at one end of the plated area rather the center. Thus, the strip is fastened to the measuring instrument only at one end and the danger of artificially bending the strip during measurement is reduced.

This paper describes an instrument set that uses the Bartlett method to measure internal stress. The design of the instrument is provided and a few examples of measurements are provided to illustrate the technique and the results that can be expected from the method.

Method

The method uses a 15.3 × 1.5 cm (6 × 5/8 in.) strip of 0.254 mm (0.010 in.) thick steel shim stock. This strip is cut using a metal cutter or large paper cutter from commercial 15.3 × 45.7 cm (6 × 18 in.) flat shim stock (Fig. 1). The strip is placed in a plastic block, (Figs. 2 & 3), which has a 10 cm (4 in.) window that allows electroplating on a 10 × 1.3 cm (3.9 × 0.5 in.) part of the steel strip.

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Figure 1—Shim stock used for measurements.

The internal stress in the electrodeposit causes the strip to bend. Compressive stress causes the strip to bend toward the uncoated side of the strip while tensile stress bends the strip toward the coated side. Compressive stress is traditionally expressed as a negative number and tensile stress is expressed as a positive number.

It is important that the electroplated coatings being compared are approximately the same thickness. In addition it is important that a significant thickness is plated so that the measurement of bend is large enough to be significant. It has been found that about 25 µm (1 mil) of thickness generally provides an adequate thickness. The required plating time can be predicted using Equation 1.

$$\text{Plating time (min)} = \frac{\text{[Plate thickness][Plating constant][60 min/hr]}}{\text{[Current density][Bath efficiency]}} \quad (1)$$

Where:

Plating constant	=	A-hr/[Area × Unit Thickness]
Current density	=	A / Unit Area
Bath efficiency	=	Fraction of current used to plate metal
Plate thickness	=	Desired thickness

The plating constant is a factor calculated from Faraday's law for 100% efficient plating for a specific metal and can be found in standard references.[17] For zinc the constant is 1.54 A-h/dm^2 (14.3 A-h/ft^2) to plate 25 µm (1 mil).

The bath efficiency of a particular plating process must be determined experimentally and usually varies with current density. The strip must be plated for a period of time and the deposit thickness determined with a properly calibrated x-ray fluorescence instrument (XRF). The percent efficiency can be calculated using Equation 2. Acid chloride zinc processes are usually about 95% efficient. Alkaline zinc processes vary from about 40% at 3 A/dm^2 (30 A/ft^2) to about 70% at 0.5 A/dm^2 (5 A/ft^2). Nickel plating processes are usually about 100% efficient.

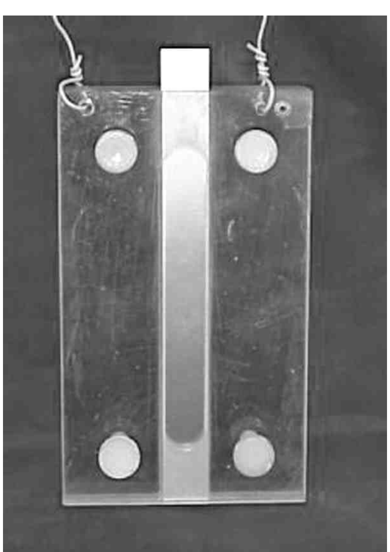

Figure 2—Plating block and strip.

Plating efficiency = [Measured thickness][100]/[Expected thickness] (2)

During the plating operation it is especially important that an accurate ammeter is used. The small ammeters on rectifiers are seldom precise enough for this. However, inexpensive commercial volt-ohmmeters available at most tool stores are adequate for this work.

The internal stress of the deposit is measured by placing the plated strip into the measurement block (Fig. 4). It is important to measure the natural bend of the specific strip before and after plating. The measurement is accomplished by moving the steel ball of the micrometer until is just touches the strip but does not bend it. The exact point can be obtained with the assistance of lined paper under the measurement block.

The stress is calculated using Equation 3:

$$\text{Stress} = \frac{4Ed^2\Delta}{3L^2t} \quad (3)$$

where:

E = Young's Modulus for basis metal [193 to 214 GPa (28 to 31×10^6 lb/in.^{2}) for steel]
d = Thickness of basis metal strip
Δ = Half the difference in the measurements of the strip
L = Length of plated strip area
t = Plate thickness

Figure 5 shows the measurement of Δ, known as the Sagitta.

Equipment

The measurement blocks shown in Figs. 2, 3 and 5 were custom made at a machine shop. A single set can be very expensive, but a larger number can reduce the price. The shim stock, micrometer and micrometer ball are available commercially.** The plastic used for the plating blocks can tolerate up to about 50°C (122°F), so it is not suitable for processes like electroless nickel that operate at about 90°C (194°F). However the plating blocks could be made of polypropylene which would work well for that purpose.

** Micrometer Head, McMaster-Carr #858A68, McMaster-Carr Supply Co., Dayton, NJ 08810.
Micrometer Ball, McMaster-~~Call~~ Carr #2313A11, McMaster-Carr Supply Co., Dayton, NJ 08810.
Steel Shim Flat Sheets, Precision Brand #16870 (0.010 in.; 6 × 8 in.), Precision Brand Products, Inc., Downers Grove, IL 60515.

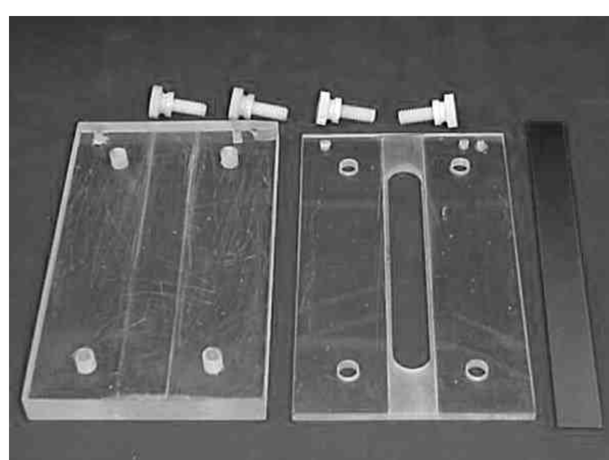

Figure 3—Plating block disassembled.

Stress of Zinc Electroplated Deposits

A number of zinc deposits plated from alkaline and acid chloride processes were tested. A few such tests are shown here to illustrate the type of results that can be obtained. In most cases the strip was plated and the stress measured both immediately after plating and then after five days. The strips were inspected to notice any possible blistering or other defects.

An alkaline zinc process was made up with various amounts of the two components, Part A, the carrier, and Part B, the brightener. Table 1 shows the results from the measurements of this series both as plated and after ten days.

In general our experience with this process is that if the amount of Part B, the brightener, is kept below about 2% by volume, the process is safe from delayed blistering. Experience also shows that, even with low brightener, if the work is not cleaned properly, the part might blister anyway.[11] The data in Table 1 show that on standing the deposit becomes more compressively stressed if the brightener level is in the correct range. However, if excess brightener is added, then the stress is not very compressive and sometimes it will blister.

The test in which excessive brightener was added was repeated and watched for four days, with measurements taken four times during each working each day. Table 2 shows the results of that measurement series.

After four days the deposit had not blistered but the stress was becoming less compressive. The stress did not change much after the fourth day and blisters were observed after about 10 days.

A test series was also performed using an acid chloride process. With this process the two components, Part W, the wetter, and Part B, the brightener, were used in varying amounts. Table 3 shows the results of the internal stress measurements immediately after plating and after ten days.

The results in Table 3 show that, within the range of additions used in this series, the final stress result does not change significantly with the addition of more of the Part B brightener.

A second acid chloride process was also tested. This one was related to the process shown in Table 3 but a different brightener, Part C was used. Table 4 provides the data from that test series.

The Part C brightener changed the nature of the deposit considerably. The deposits were tensile from the start after 0.09% by volume of the Part C brightener was used.

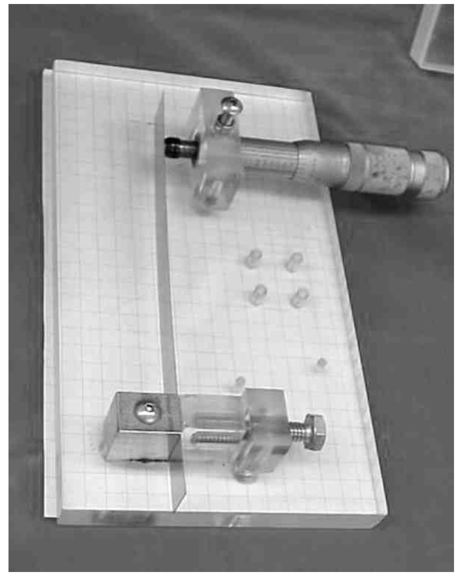

Figure 4—Measurement block.

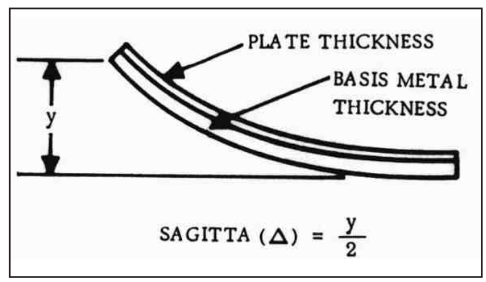

Figure 5—Sagitta measurement.

Table 1
Alkaline Zinc Process: Varying Amounts of Carrier and Brightener

Part A Carrier	Part B Brightener	As Plated Stress, MPa (lb/in.^{2})	Final Stress (10 Days) MPa (lb/in.^{2})
2.0%	0.0%	-91.0 (-13,200)	-170.0 (-24,700)
2.5%	0.0%	-94.5 (-13,700)	-161.0 (-23,400)
3.0%	0.0%	-97.9 (-14,200)	-169.0 (-24,500)
2.5%	0.5%	-104.0 (-15,100)	-159.0 (-23,000)
2.5%	1.0%	-110.0 (-15,900)	-143.0 (-20,700)
2.5%	3.0%	-53.1 (-7,700)	-48.3 (-7,000) Blister

Table 2
Alkaline Zinc Process: 2.5% Part A and 3% Part B, Measured Four Times Per day for Four Days

Day	Stress, MPa (lb/in.^{2})
1A	-42.1 (-6,100)
1B	-61.4 (-8,900)
1C	-70.3 (-10,200)
1D	-68.9 (-10,000)
2A	-71.7 (-10,400)
2B	-74.5 (-10,800)
2C	-72.4 (-10,500)
2D	-68.3 (-9,900)
3A	-56.5 (-8,200)
3B	-59.3 (-8,600)
3C	-60.7 (-8,800)
3D	-61.4 (-8,900)
4A	-52.4 (-7,600)
4B	-53.1 (-7,700)
4C	-51.0 (-7,400)
4D	-48.3 (-7,000)

Table 3
Acid Chloride Process 1: Varying Amounts of Wetter and Brightener

Part W Wetter	Part B Brightener	As Plated Stress, MPa (lb/in.^{2})	Final Stress (10 Days) MPa (lb/in.^{2})
4.5%	0.00%	+24.8 (+3,600)	-23.4 (-3,400)
4.5%	0.04%	-20.7 (-3,000)	-24.1 (-3,500)
4.5%	0.08%	+5.5 (+800)	-15.9 (-2,300)
4.5%	0.13%	-6.9 (-1000)	-30.3 (-4,400)
4.5%	0.17%	+0.7 (+100)	-24.1 (-3,500)

Continued tests of this nature illustrated that various processes performed differently.

Conclusions

1. A method has been demonstrated that measures the internal stress of electroplated deposits.
2. The test method is simple to use and the instruments can be made from commercially available materials and instruments.
3. The method readily shows the differences of various plating processes in terms of internal stress generated inside the deposits.
4. The materials are suitable for highly corrosive plating baths like alkaline zinc.
5. The plating block can be made of polypropylene so that high temperature operation is possible.
6. The test specimens can be made of mild steel which is unavailable for the spiral contractometer. Shim stock is also available in brass and stainless steel.

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About the Author

Dr. David Crotty obtained B.S. and M.S. degrees in chemistry at Xavier University in Cincinnati and a Ph.D. in chemistry at Wayne State University in Detroit. He has worked in the metal finishing industry since 1980 as a research and development chemist. His research interests have included electroless nickel plating, zinc plating, zinc alloy plating, and associated processes.

Table 4
Acid Chloride Process 2: Varying Amounts of Wetter and Brightener

Part W Wetter	Part C Brightener	As Plated Stress, MPa (lb/in.^{2})	Final Stress (10 Days) MPa (lb/in.^{2})
4.5%	0.04%	-6.2 (-900)	-15.9 (-2,300)
4.5%	0.09%	+28.3 (+4,100)	+29.0 (+4,200)
4.5%	0.13%	+44.1 (+6,400)	+45.5 (+6,600)
4.5%	0.18%	+53.8 (+7,800)	+50.3 (+7,300)