Electronically Monitoring Plating Stress in Real-Time

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Internal residual stress in plated coatings was defined and measured by Stoney using deformation of a membrane supporting the coating early in the last century. Mathematics used to calculate stress have been refined for various configurations including fixed or free standing rectangular strips, helixes, circular membranes, cylindrical rods etc. A brief history of stress measurements for plating is presented.

Electronic monitoring of plating stress has been performed for more than 25 years and includes a large number of methods including attachment of strain gauges to a bending membrane or to monitor length change in a plated rod, optical monitoring of a bending membrane, capacitance coupling to a membrane, mechanically coupling potentiometers to a deforming surface, a mercury switch High-Low sensor and use of an incompressible fluid filled chamber to couple a membrane to a pressure sensor. Also many physical methods for individual measurements of residual stress have been accomplished including x-ray diffraction, neutron diffraction, before and after deflection of a strip and drilled-hole size changes. However the physical measurements have not been extended to real-time monitoring for stress while changing in an electrodeposit during plating.

A real-time electronic monitor with fluidic amplification is described. Analog output is generally processed off-line to provide real-time stress readings in various engineering units. This information is also digitized and used directly in a computer routine. Stress graphs are automatically prepared and process control is simplified. Examples of stress vs. current density, additive concentration, solution agitation and time are shown.

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1.0 Internal residual stress in a depositing metal

While much information is available on the nature and measurements of internal stress in materials, especially in metals, a brief re-cap is in order. See for example the excellent works of P. Withers and H. Bhadeshia.[1] Internal or residual stress is that disordered arrangement of crystalline and atomic structure leading to an equilibrium unbalance in a material resulting in stored energy. It can be the manifestation of disturbances of different spatial resolution. Long order resolution refers to disturbances of the magnitude of the grain size or larger in a material. This may be the result of grain boundary sliding due to external forces or foreign particle or impurity inclusions in a plated material. Also the grain size in an alloy or single element deposit can vary with plating conditions. If this occurs while plating, the stress can be non-uniform with thickness. Intermediate to this are disruptions on the order of crystallite size in a material. This type internal stress in a plated material may be the result of incomplete solution of the metals in an alloy with lattice or unit cells not having the same or similar lattice parameters. Many phases can also exist for a binary (or higher) alloy and when a non-stoichiometric composition exists, intermetallic or multiple phases of the material can be formed and will generally overlap. This results in saturation at a given composition requiring substitution within a range of superlattice alloying, causing "jumps" in the lattice structure with regard to changes in the alloy. This often results in very complex unit cells with many atoms. Short order refers typically to the disordered atomic structure which can be the interjection of extraneous atoms or omission of atoms causing stress within a unit cell. These different manifestations are commonly called type I, II, or III stress and can co-exist in a given material.

Additionally, stresses are manifested in amorphous deposited alloys. While the grain size may be below resolution in a light microscope or the crystalline structure not detected by x-ray diffraction, there exists an atomically ordered lattice structure. Notably deposits of nickel, cobalt and iron alloys with more than 10% phosphorous and certain phases of copper-lead are considered amorphous. In addition to the alloying elements, stress can be manifested in amorphous deposits by interstitial injection of hydrogen, nitrogen, carbon and low concentrations of other cationic and anionic materials forming dilute interstitial alloys with modest to severe internal stress as well as compounding stress in simpler deposits. By controlling the concentration of these diatoms in the material, the stress can often be controlled to low values and may even be tensile or compressive in nature.[2]

Not commonly considered in metallurgical evaluations of induced stress in plated metal is the fact that a deposited material proceeds to generate a structure from a given starting surface with differing lattice parameters and develops the characteristic intrinsic stress over time as the material deposits. If the growth is considered to occur as infinitesimally thick layers (atomic dimensions) then the stress occurs soon after deposition begins. This is true in both electrolytic (including electroless) and vacuum deposited films. Since the substrate is considered rigid in comparison to an atomic layer of deposit, the deposit layer is subjected to strain opposing the manifested stress. If that strain enters into the plastic domain of the deposited metal, then a partial relaxation is invoked. Thus after depositing a thin layer of say a few thousands of angstroms, the stress will appear non-uniform in the film with the highest stress on the outer layers. This in turn is no longer an axial or biaxial stress as in the case of a mechanically strained component, but is a bending stress and will tend to curl if released as a free-standing form. Electroformed shapes such as optical components are very sensitive to residual stress. It is more important in this case to control the stress throughout the electroformed shape. By carefully monitoring the solution and the developing stress it is possible to deposit nearly distortion-free optical forms.

2.0 Monitoring internal stress in a deposited metal electronically

As mentioned, the initial calculation for stress in a coating applied to a strip was developed nearly 100 years ago assuming the stress to be uniform in the coating with thickness.

This is the analysis in Appendix I. and excludes thermal expansion differences and changes in temperature. If the sample is removed from the substrate then the deformation can be seen to be due to bending. A recent analysis of this is found in Ref.[3].

Probably the most commonly used plating stress monitoring principle is the Spiral Contractometer developed by the National Bureau of Standards (now National Institute of Standards and Technology) in conjunction with the development of alloy plating processes by Dr. Abner Brenner in the 1950's. This is a flat strip wound into a spiral, annealed and connected to a dial indicator. The stress in the plated coating either unwinds the spiral for tensile, or contracts the spiral for compressive stress. An electronic variant of this capable of real-time measurements was presented in Las Vegas at the Electrochemical Society in the early 1980's by the author, which consisted of a variable resistor (potentiometer) connected to the shaft in place of the dial indicator. A bias on the potentiometer could be monitored on a recorder or computer input in place of manually reading the dial. At about this time also was the introduction of an electronic version of stress monitor using strain gauges on a flat strip which were monitored by computer and provided an output which could be used to calculate stress in real-time. See patents 4,647,365 and 4,648,944, Martin Marietta Orlando Aerospace, K. Irlesberger, D. Engelhaupt, et al. Shortly thereafter, alternate versions appeared but were not commercialized due to the MMOA patents. These devices failed to provide reliable results with one patented device the better unit. This device incorporated two membranes linked together with the strain gauge completely encapsulated in the aft section. This made for a more reliable unit in which the first (forward) membrane could be removed and stripped in acid without risk of damaging the strain gauge. The modified Spiral Contractometer was not particularly sensitive and required substantial stress to determine a value. Also hysteresis was prevalent due to mechanical linkages connecting the indicator to the spiral and/or the large deformation of the spiral.

In 1989 a new approach was introduced involving the use of a fluidic amplifier in the form of a sealed chamber with a membrane coupled to a silicon pressure sensor with a low expansion fluid. The membrane area is about 1000 times the sensor area which produces a substantial signal without the need for large deformation of the membrane.[4] While not completely, this does substantially reduce the deformation of the membrane to measure the signal. Thus the elastic properties of the membrane are almost not present in the measurement of stress in a plating environment with this approach. Also the silicon membrane in the sensor has virtually no elastic hysteresis, resulting in excellent repeatability. The primary advantage to any electronic device is that a recording device may be used to record the signal and with data collection by a computer the data can be reduced quickly or read and converted in real-time values. By the use of an inert membrane the deposit can be stripped electrochemically by reversing the current in the plating bath or in another solution. A continuous series of tests are run to find the parametric relationship with respect to a dependent variable. Most commonly the current density is varied incrementally and the stress is read at each of a sequence of values. This is then reduced to a chart by calculations from the computer and retained as a record of stress vs. current density. Also of interest is the recording of stress with respect to agitation, pH, additives, time, and solution formulation or operating parameters in general. The continuous stripping of the deposit while recording reveals stored stress (energy) as a reverse function of the time of plating.

3.0 Examples of stress monitoring in real-time

The most common case for monitoring stresses precisely and in real-time is notably in the electroforming of precise components such as optically reflecting devices. An interesting extension to the requirements for precision is seen in the fabrication of shorter wavelength optical, XUV and x-ray imaging devices. By using the real-time methods in conjunction with the use of a computer the National Aeronautics and Space Administration (NASA) has been able to develop plating processes of high strength nickel alloys with extremely low stress. The incorporation of these processes and procedures has enabled large optical quality x-ray mirrors to be produced with approximately one square meter surface and deformation in less than a few microns over the entire electroformed alloy component. Smaller nickel alloy x-ray mirrors have been produced with as little as 0.5 microns deviation from the mandrel.

Typically a set of stress vs. current density curves are recorded at different levels of a second variable. This might be a set of data for stress/current density in an alloy such as nickel cobalt vs. additive concentration or pH. With this data it is possible to tailor the process to provide a nearly flat response to stress with respect to current density over a wide range. This is used to accommodate variant current density over the part while maintaining uniform stress. That is if the current density varies over the part then the thickness will also vary. However if the stress is substantially zero over the range of current density invoked, the deformation of the part will be essentially zero also. See Figures 1-2. Another example is the control of stress vs. agitation.

With the “ski-jet” agitation device in figure 3, it is possible to vary the agitation in real-time while monitoring stress also over a range of interest. In this case the gauge in Figure 4-5, is used to monitor first the agitation by varying the impeller speed and recording the tachometer output and measure the force applied to the membrane by kinetic fluid principles to calibrate, and then measuring the steady state stress at different agitation levels. With this data the profile of stress vs. current density and agitation may be combined to provide a precise control of the plating part. It becomes necessary to determine the agitation at the part or measure a few optical components to determine the level of stress then from the data, increase or decrease agitation as required. Agitation is fixed at the pump and nozzle level but we are able to increase or decrease the rotation velocity of the parts according to fluid dynamics Reynolds and Taylor numbers to provide the same stress in the part as the “ski-jet” indicates. As time introduces variations in the process, we can adjust conditions to zero stress on the instrument and accordingly on the parts as well. Finite element analysis demonstrates that it is required to have near zero and not just uniform stress to avoid deformation of a free-standing electroformed optical component.

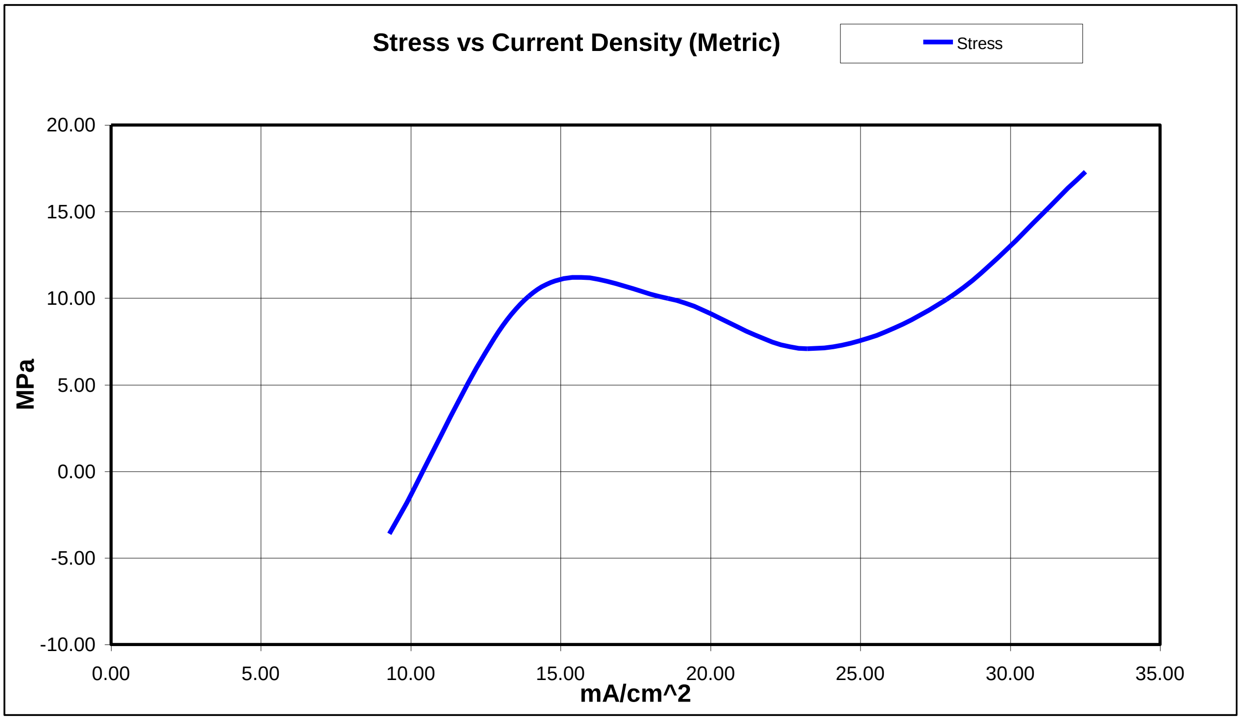

Figure 1 Stress vs. Current Density in Ni Sulfamate solution

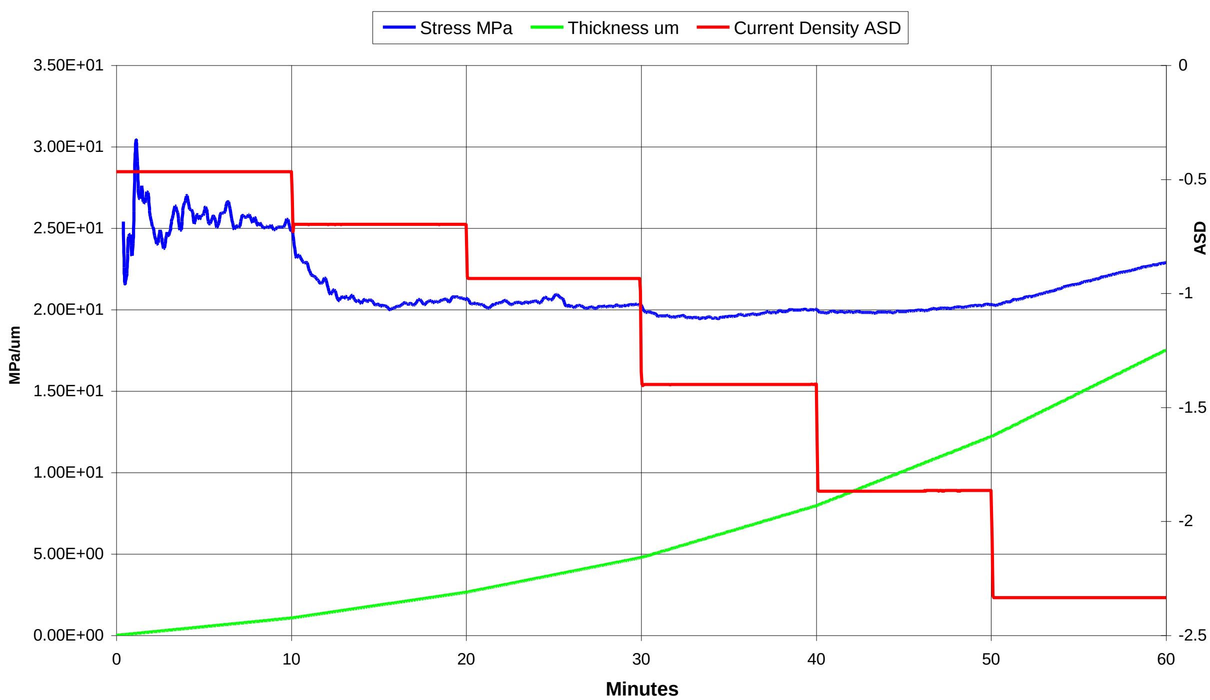

Figure 2 Cumulative Stress Real-Time - NiP Electroplating Solution

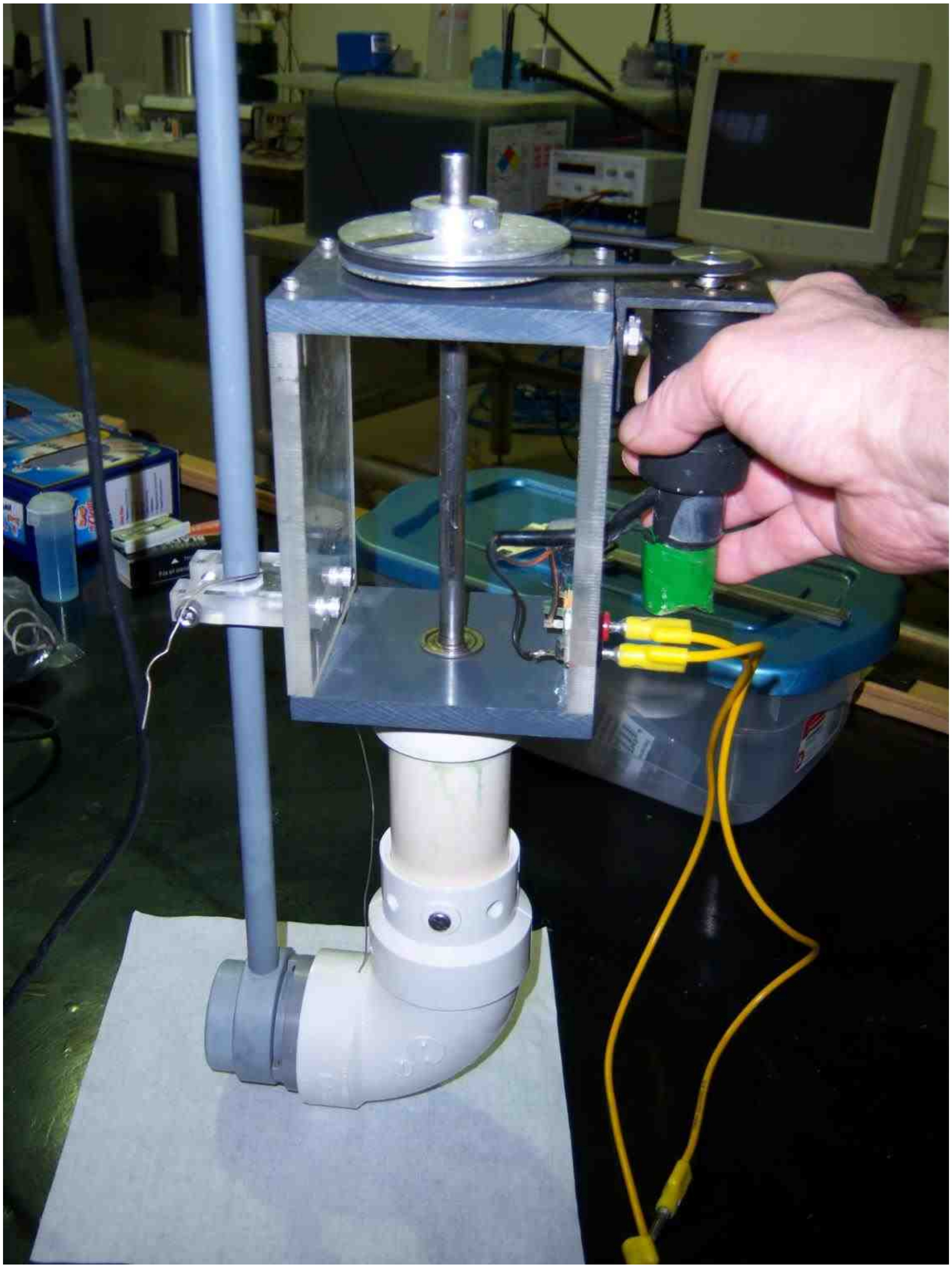

Figure 3 Ski Jet Stress vs. Agitation Device

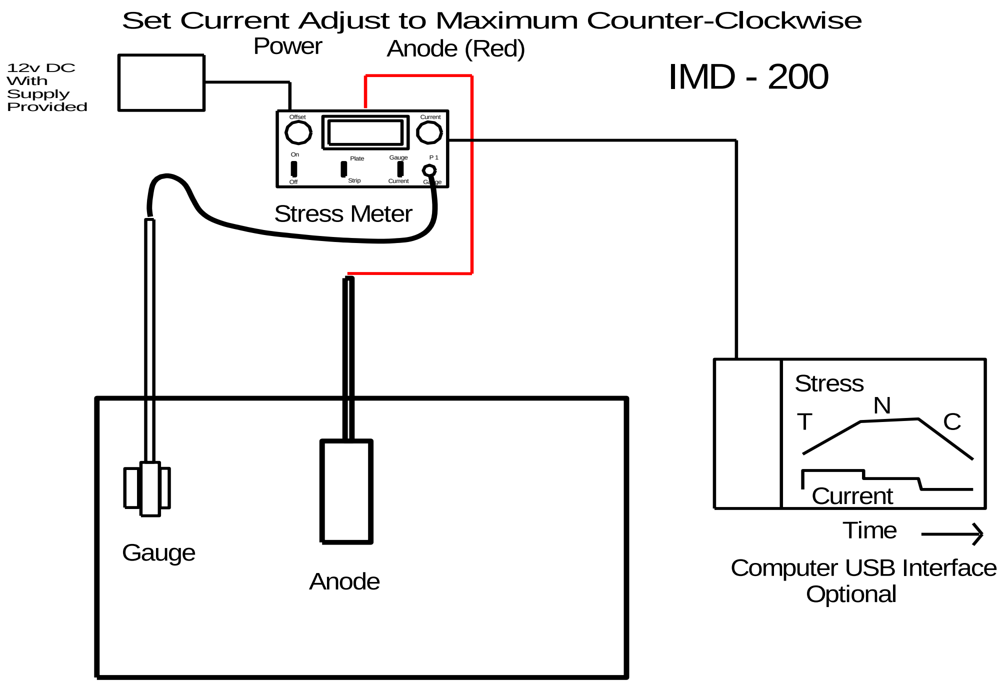

Figure 4 Schematic Plating Set-up with Electronic Monitor

Figure 5 Electronic Stress Meter

Appendix I.

Stress in Bent Strip

	Add Dimension		
Enter:			
L = Length of Strip	Length	6.0000	Inch
Ec = Elastic modulus of coating	Ec Ni	2.70E+07	psi
Eb = Elastic modulus of base steel strip	Eb Steel	2.90E+07	psi
t = thickness of strip	t Strip	0.0100	Inch
d = thickness of coating	t Coating	0.0030	Inch
h = height of bent strip in center of curvature	height	0.0020	Inch

Calculate:		
C= (L-h)/4	**1.4995**	**Inch**
R = Ec/Eb	**0.9310**	**Dimensionless**
r = radius of curvature of plated strip	**140.5323**	**Inch**
Stress = Eb[R(t+d)^4 - (R-1)t^4]/(6rdt(t+d))	**2405.8319**	**psi**

Enter:	**Calculate:**	**Calculate:**	
h	r	Stress	
0.0010	281.0630	1202.9224	psi
0.0020	140.5323	2405.8319	psi
0.0030	93.68901	3608.7158	psi
0.0040	70.26763	4811.5611	psi

5.0 References

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