Journal of Applied Surface Finishing, **3** (4), 173-177 (2008)

Electronically Monitoring Plating Stress in Real-Time

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Internal stress in plated coatings was defined and measured by Stoney early in the last century, using deformation of a membrane supporting the coating. Mathematics used to calculate stress has been refined for various configurations including rectangular strips, helixes, circular membranes and others. An electronic monitor with fluidic amplification is described. Digital or analog output is processed to provide real-time stress readings in various engineering units. Stress graphs are automatically prepared and process control is simplified. Examples of stress vs. current density and time are shown. Closed loop PID control is described for experimental plating.

Keywords: *residual stress, real-time monitoring, electrodeposit stress, computerized monitor*

Introduction

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 will be 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 here. 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 will be shown.

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 recap 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 imbalance 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 of 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

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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% phosphorus 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 metals 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, 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 a few thousand 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 is commonly 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.

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 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 given by Zhou, *et al*.[2]

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 1950s. 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 described in the early 1980s by Engelhaupt while at Martin Marietta, 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 the same time was the introduction of a patented electronic version of a 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.[3,4] Shortly thereafter, alternate versions appeared but were not commercialized due to the patents. These devices failed to provide reliable results with one patented device being 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. Further, 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 using a low expansion fluid. The membrane area was about 1000 times that of the sensor area, which produced a substantial signal without the need for large deformation of the membrane.[6] This substantially, though not completely, reduced the deformation of the membrane to measure the signal. Thus the elastic properties of the membrane were nearly unobserved in the measurement of stress in a plating environment with this approach. Also, the silicon membrane in the sensor had 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.[5,6] The continuous stripping of the deposit while recording reveals stored stress (energy) as a reverse function of the time of plating.

Calibration of the electronic stress monitor

Calibration of the monitor is accomplished by applying air pressure to the membrane through the use of a special holding fixture. Beyond this initial calibration, it is only required to apply small calibrated weights to the surface of the probe while it is lying on the back to check the output. Adjustments are internally available to correct any errors. A lens tissue is used to protect the membrane and the weight is applied to a small diameter ring centered on the membrane. Additionally it is possible to calibrate the computer readout within the software.

Closed loop PID control

Either digital or analog signals may be used for closed loop control of stress.[3,4] If analog signals from the monitor are used, an A/D converter is used to provide a signal to the computer. Otherwise a digital signal may be provided by a USB interface.[6] The computer software can determine the slope of the stress in real-time and predict the required current change needed for zero stress. Typically in nickel from sulfamate, for example, an increase in stress output slope would signal for a reduction in current. A programmable power supply is connected to the parts of concern and is programmed by the same commercial software[5] to react in parallel to the gauge response thus controlling the plated component stress as well as monitoring and controlling stress on the gauge.

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 of 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 μm deviation from the mandrel.[5]

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, pulsed parameters 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. 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. However if the stress is substantially zero over the range of current density applied, the deformation of the part will also be essentially zero and may be determined for many instances as in Figs. 1, 2 and 3. The effect of pulsed plating parameters on stress is observed in real-time for a low pH NiP process as in Fig.

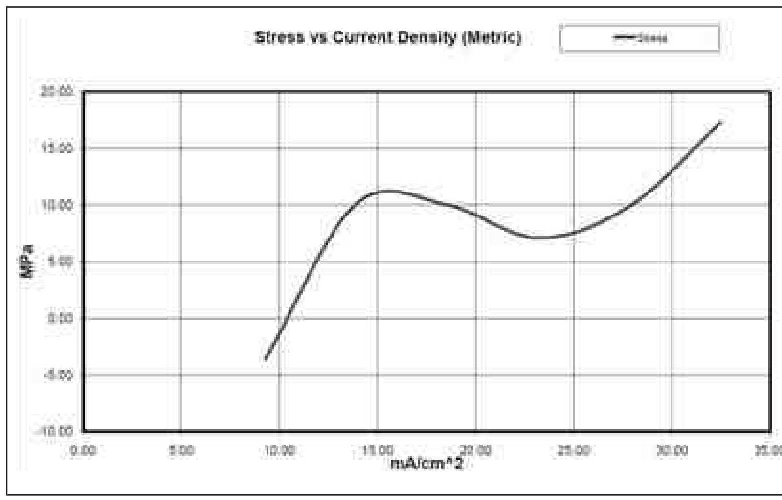

Figure 1—Stress vs. current density in a nickel sulfamate solution.

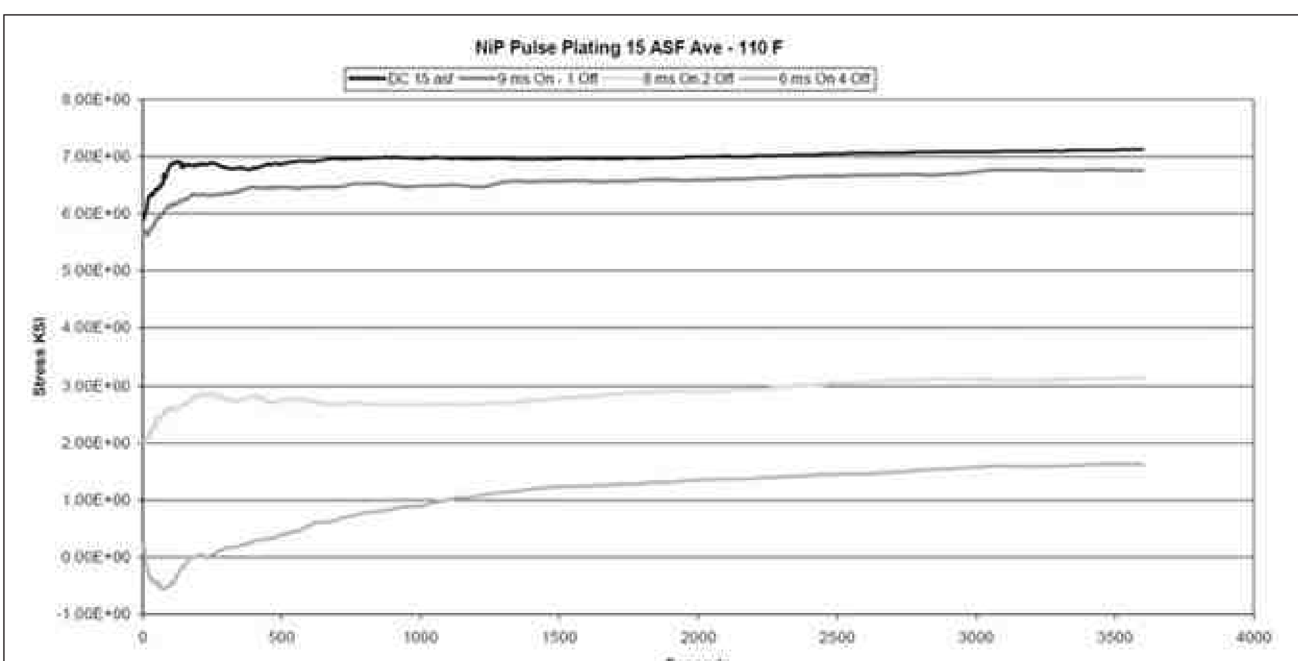

Figure 2—Stress vs. pulsed parameters in low pH Ni-P plating.

4. Many parameters for stress monitoring exist in just about any process of concern.

Another example is the control of stress vs. agitation. With the "ski-jet" agitation device shown in Fig. 5, it is possible to vary the agitation in real-time while simultaneously monitoring stress over a range of interest. In this case the gauge shown in Fig. 6 (and schematically in Fig. 7) and the "ski-jet" shown in Fig. 5, is used first to monitor the agitation by varying the impeller speed and recording the tachometer output, and second to measure the force applied to the membrane by kinetic fluid principles to calibrate, and then measure 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 as in Fig. 3. It becomes possible to determine the agitation at the part or measure a few optical components to determine the level of stress, and 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 rotational 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, conditions can be adjusted to match to nearly zero stress on the instrument and accordingly on the parts as well.

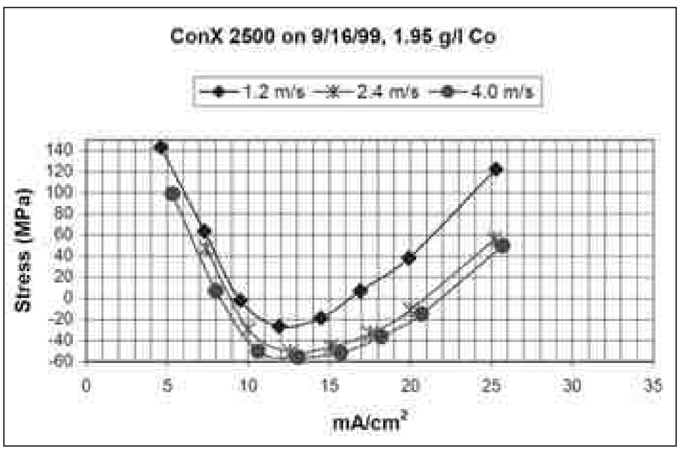

Figure 3—Ski jet stress vs. agitation in nickel-cobalt phosphorus.

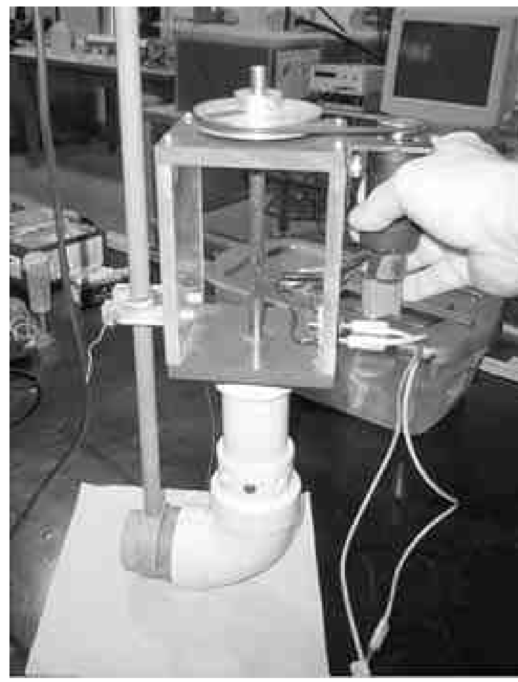

Figure 5—Ski jet stress vs. agitation device.

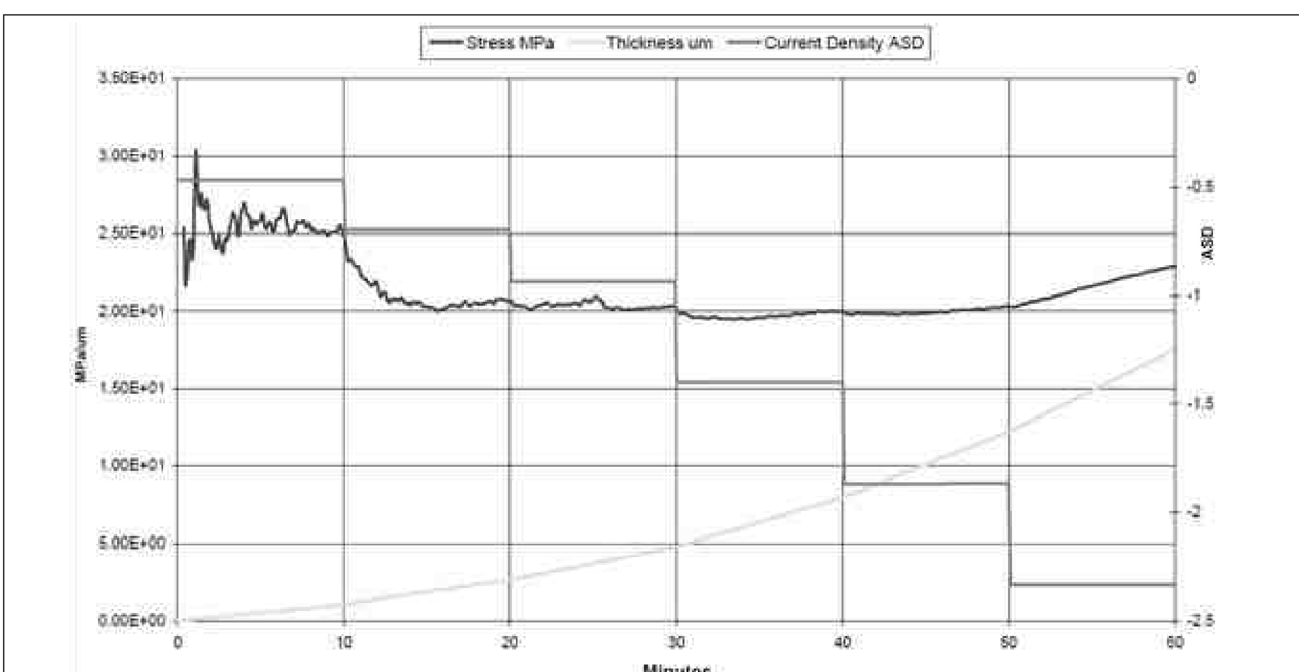

Figure 4—Cumulative stress in real-time for a Ni-P plating solution.

Figure 6—Electronic stress-meter.

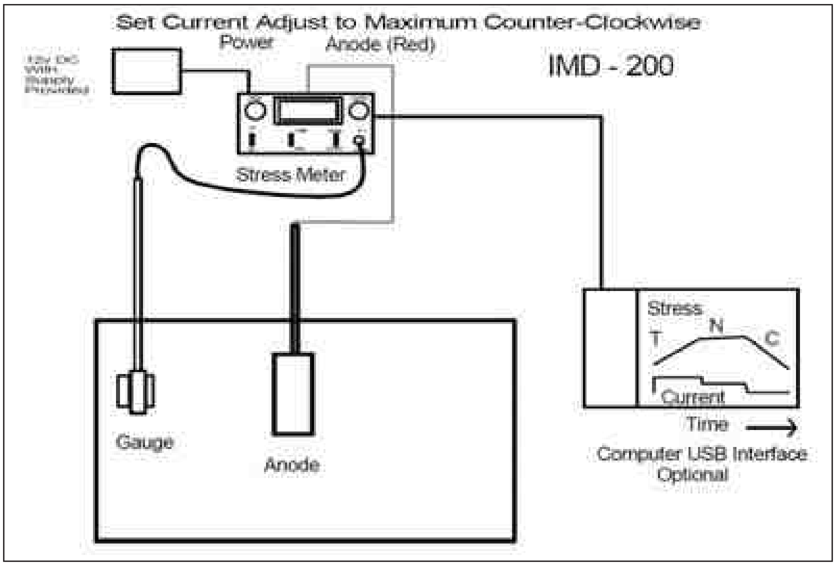

Figure 7—Schematic plating set-up with electronic monitor.

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About the author

Darell Engelhaupt is a Senior Research Scientist at the University of Alabama in Huntsville with more than 30 years experience in fabricating high tolerance components using electroforming processes. He has 15 patents as author or co-author in metal finishing and in instrumentation design. Mr. Engelhaupt is a staff member of the UAH Center for Applied Optics where he is currently developing manufacturing processes for NASA's Space Science x-ray optics programs. His education includes a B.S. in General Engineering/Physics at the University of Missouri - Kansas City (UMKC) and graduate studies in Mechanical Engineering/Physics at the University of Kansas. He is a long-standing member of NASF (AESF), SPIE and ECS.