

# Comparative Study of Three Internal Stress Measurement Methods

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## Introduction

Several new developments have taken place since 1963 when L. C. Borchert published his 3-way comparison of methods for the measurement of internal stress in electrodeposits<sup>1</sup>. In the West, the change of length method became associated with an instrument known as the I. S. Meter<sup>2,3</sup>, the rigid strip technique has been improved with two-legged strips and an in-tank test cell<sup>4</sup>, and a multitude of new, stress-sensitive plating and electroforming processes were pushed to the technological forefront by high-tech industries. Many practical processes today require stress measurement accuracies of less than one MPa rather than tens or hundreds of MPa (or several hundred rather than thousands of psi). While industry needs an accurate real-time technique for stress monitoring and control, the scientific community could apply it to solving one of the long lingering mysteries in electrocrystallization - that of the origins of stress in electrodeposits. The need for a precise, sensitive, fast and affordable stress testing method is now greater than ever before.

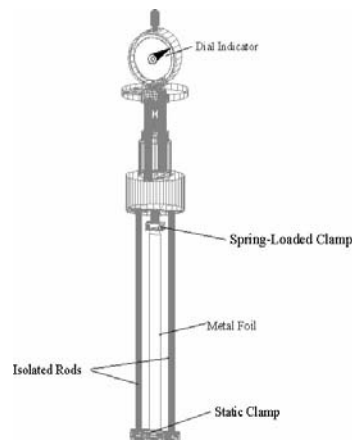
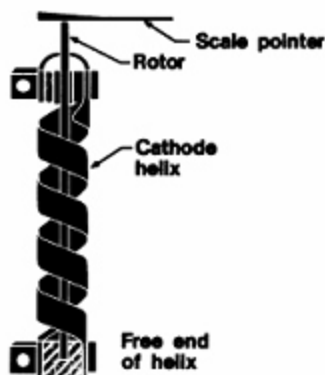


Fig. 1c. IS Meter

Fig. 1a Spiral Contractometer

Fig. 1b Bent Strip on Stand

In this paper we will present a quantitative comparison of three techniques widely used in scientific and industrial stress measurement – the spiral contractometer, the bent strip and the I.S.Meter (see Figure 1), to help readers choose the more appropriate method for their diverse applications. We selected these methods for evaluation based on the following criteria: **quantitative character** - there should exist a theoretically derived formula for calculating internal stress based on the registered physical changes in the sensor; **anticipated high accuracy and resolution**; **ease of use** and, finally, **prevalence of use**.

Our task was made somewhat simpler by the fact that the study was being conducted in an industrial environment with numerous tanks and parts being electroformed while measurements were taken. This often served as a useful 'reality check' for the meaningfulness of generated data. All the described tests were done in sulfamate nickel tanks under normal electroforming operating conditions.

## Spiral Contractometer

Since its development in 1949 by Brenner and Senderoff<sup>3</sup>, the Spiral Contractometer has become the preferred industrial instrument used to measure stresses in electrodeposits. The primary component of the device is a strip of stainless steel formed to the shape of a helix. The internal side of the helix is coated with a lacquer or a layer of *Teflon*<sup>TM</sup> while the external side remains bare, allowing it to plate. During the plating cycle stresses in the electrodeposit will cause the helix, one end of which is fixed, to deform: compressive stresses will result in a tightening of the helix while tensile stresses will cause it to unwrap. The angular deflection of the free end of the coil is transferred to a pointer by a set of gears and can be monitored throughout the testing process on a dial mounted atop the coil. The user is then able to calculate the corresponding deposit stress level based on this information.

Of all the test methods examined, the contractometer was the most difficult and time-consuming to operate. The setup included determining the spring constant of the helix, fastening the helix to the instrument, masking the top and bottom regions of the helix to prevent plating, and centering the pointing device on the scale. If the manufacturer's instructions are to be followed, it is necessary to utilize a beaker and a hot plate in order to operate the test externally to the plating tank. Fortunately, our study provided evidence that this is not an essential part of obtaining good test results. There was found to be good correlation between readings from tests taken directly in the plating tank with those taken in a beaker.

## Bent Strip

Developed by G. C. Stoney in 1909<sup>6</sup>, the bent strip technique is probably the most straightforward and simple means of measuring stresses in electrodeposits. It works in the following manner: a thin strip of metal is plated on one side while a protective lacquer coats the other. Any stresses in the deposit will cause a net curvature to be induced upon the strip, and this curvature can be converted to an equivalent stress level.

The Bent Strip test is extremely simple and easy to carry out, and one of its most attractive features is the fact that the test can be performed directly in a plating tank. The measurement is generally performed with .002 in. thick beryllium copper strip which has two legs, each with one side coated to prevent plating. The strip is placed into a specially designed tubular test cell that is fastened to the edge of the plating tank where it is plated for a given amount of time. The cell's functions are to prevent the strip from solution flow disturbances, to create an electric field conducive to uniform deposit thickness distribution across the strip and to constrain the ends of the strip during testing. Since the bare sides of the strip legs face opposite directions, a stressed deposit will result in a net spread with each of the legs bending either toward (compressive deposit) or away (tensile) from its exposed surface. After plating is completed, one simply measures the resulting spread and determines the deposit stress.

## Change of Length Method

The concept behind this method, first proposed by Popereka in 1958<sup>7</sup>, is fairly straightforward: a thin strip of metal is kept in tension by a given preload while being plated on both sides. Any inherent stress in the deposit will cause a change in the length of the strip that is measured by keeping one end fixed and allowing the free end move with the stress and affect a gauge attached to it. The reading from the gage can once again be converted into an equivalent deposit stress level.

Running a test with the I. S. Meter is fairly straightforward. An appropriate test strip, usually .001” thick by .5” wide, is aligned and fastened into the clamping jaws. The entire assembly is then placed into a plating tank, and after coming to thermal equilibrium the strip is plated to the desired deposit thickness while gage readings are taken at a given interval.

## **Comparison of Readings from Different Instruments**

Providing that stress calculations in each method are based on sound theories, readings taken by one type of instrument should produce close results to those taken under identical plating conditions by another type. Unfortunately, we found this not to be the case. Measurements taken by different instruments often produce substantially disparate numerical stress values while maintaining a general correlation in the sign of internal stress (compressive or tensile) and, sometimes, in the zero points as well. We consider the following factors to be primarily responsible for those differences: the effect of different substrates on the initial instantaneous stress values which in turn affect the resultant average stress levels recorded and the assumptions and simplifications made in each mathematical model used to convert deflections into stress levels.

### ***Influence of Substrate Material***

It is commonly accepted that differences in crystal structure between a substrate and the deposit material result in stresses that are often observed to be very high in the early stages of plating, and this effect will be discussed in more detail later in the paper. Because each instrument is generally operated with a different substrate material, difficulties often arise when comparing readings taken with different instruments. The high stress levels in the early stages of deposition can serve to exaggerate the average stress recorded during a test, and care must be taken to understand the phenomenon and record data which reflects what occurs at steady state. Generally, the easiest way to achieve this is to perform tests to a deposit thickness that will allow the initial effects to be overcome by sufficient plating time after these effects have disappeared. However, it is often desirable to minimize test duration and for this case there are other options available. The simplest alternative is to strike the substrate with a thin deposit of about .0005 in before the test itself is begun. At this point the measuring instrument is reset and data is acquired. If adequate test parameters are decided upon and followed closely, it is possible to obtain very good measurements of the stress level that occurs during steady state.

### ***Mathematical Model Differences***

The assumptions made in deriving the mathematical model for a particular sensor play a large role in the meaningfulness of its readings. For example, the formula most commonly used to convert the curvature in the bent strip to an equivalent deposit stress is based on the simplified assumption that there is a linear relationship between the radius of curvature and the stress level. This assumption is only valid when the measured deflections are on the order of magnitude of the thickness of the strip itself: in reality they are many times greater. This simplification results in the fact that as stress increases, the measured stress values fall short of the actual ones with an ever-increasing error. This phenomenon also occurs with the Spiral Contractometer, but since the helix thickness is typically much greater than the deposit, this error is masked by the low resolution of the instrument.

Together, these and other factors listed above serve to create a difference in the readings obtained from one type of instrument to another, even if all tests are performed under identical circumstances. Generally, as the deflection phenomenon and the model used to calculate stress in the change of length method are each linear, readings from change of length instruments are greater in magnitude than readings from the bent strip method or the spiral contractometer. Fortunately, in practical applications it is possible to choose one instrument with a sufficient resolution and establish a meaningful relationship between its measurements and the state of electroformed objects. Until better understanding of the stress phenomena is developed, it is sometimes helpful to think of stress readings not as measurements of an absolute quantity, but

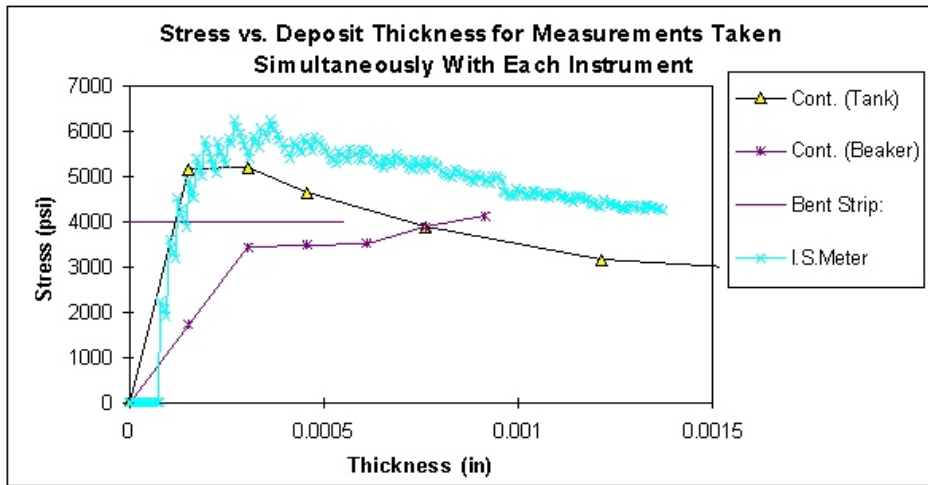


Fig. 2.

as a scaled indicator of the condition of a plating tank.

Figure 2 provides a graphical presentation of the results from a typical test in which each technique was used to measure the stress level in a tank simultaneously. This graph also displays data from runs taken with the spiral contractometer both directly in the plating tank and in the external beaker. Although the two readings are not exactly the same, they are close enough to justify the elimination of the external beaker and this was typical of the tests taken with the contractometer in both configurations. It is recommended that carrying tests out in a beaker, as opposed to directly in the plating tank should be avoided when taking measurements with the spiral contractometer. The bent strip test, which was taken at a deposit thickness of 0.00055 in., yielded a value that was lower than that of the I. S. Meter and the in-tank reading from the contractometer, but slightly higher than the external reading of the contractometer. Such results were found to be typical of the research performed.

## Initial Stress Patterns

During electrodeposition, the internal stresses that occur are not constant as deposit thickness increases. In certain cases, stresses in the very early stages can be many times higher than those occurring at steady state, and they are occasionally even of the opposite sign. These effects seem to be primarily related to the difference in crystal structure between the substrate surface and that of the deposit material. Although the physical nature of this phenomenon does not seem to be well understood, it is possible to use the change

of length method to accurately measure these initial stress levels. The electroplater can then use this information to determine how to better manage current density throughout a specific electroforming process.

The following graph, Figure 3, contains data taken from three different plating systems. It is interesting to note that while all three tests yield essentially the same steady state stress level, their behavior early on was entirely different. When nickel was plated onto a passivated nickel substrate, the first few layers appeared to have been deposited in a highly compressed state. This effect seems to have been rather short in duration, and tensile plating brought the average stress near the steady state level at a thickness of approximately 0.00015 in. For the case of non-passivated nickel plated onto nickel, stress seemed to remain between zero and the steady state level early on. Once again the steady state level was reached by a deposit thickness of about 0.00015 in. Finally, the result of plating nickel onto a non-passivated steel substrate was that highly tensile stresses occurred initially. However, the effects of this phenomenon had been overcome by a deposit thickness of about 0.0001 in, at which point the steady state stress level was apparent.

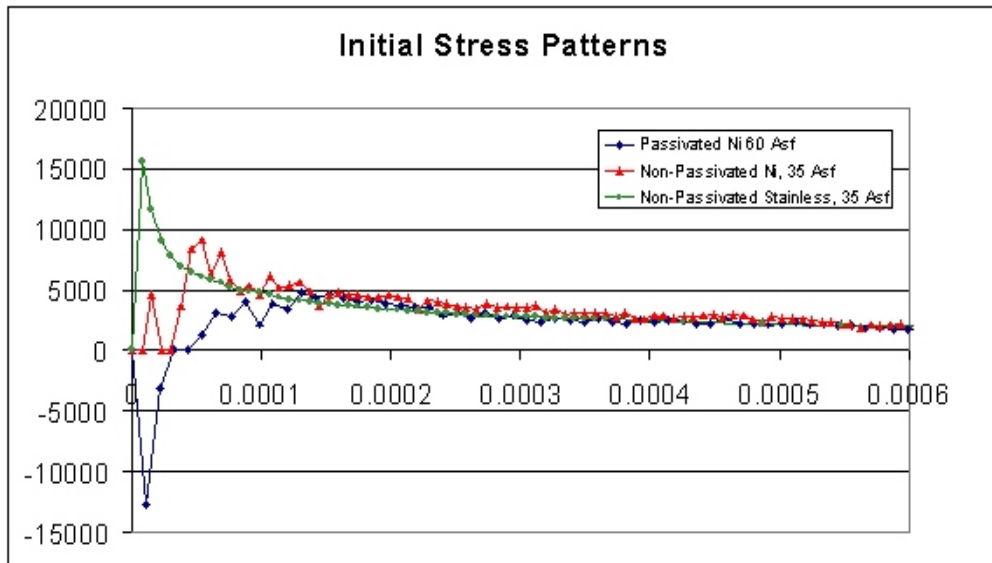


Fig. 3.

In general, changes in the initial stress levels are short lived and their influence is generally gone by a deposit thickness of about 0.0002 in. However, it has been observed that plating onto certain materials yields initial stresses high enough that even the steady state readings are artificially exaggerated. In these situations it is better to pre-plate the substrate for a given time and begin taking readings only when this has been done. With care and practice this technique can lead to acceptable stress measurements in most situations. It should also be noted that this data was provided not to show how nickel behaves when plated onto specific substrates but as an example of the fact that these initial effects are very real and that it is possible to accurately measure them.

It is also worth noting that stress measurements in the very early stages of plating are not easily made with the Spiral Contractometer or the bent strip test and can only be taken with great care using the I.S. Meter. This is due to the fact that these instruments do not ordinarily provide the resolution required for such precise investigations. For this application the authors have developed more accurate instrument modifications

based on the change of length concept. Design improvements have allowed much more precise determination of initial stress levels.

## Repeatability

For a measuring device of any kind, repeatability is a crucial characteristic. In regards to measuring stresses in electrodeposits, in its Standard Test Method for Measurement of Internal Stress of Plated Metallic Coatings with the Spiral Contractometer (B 636 – 84 ) the ASTM has established a standard for determining the repeatability of the Contractometer:

“Consider the procedure repeatable if the results of three consecutive tests on the same solution sample produce internal stress values that do not vary by more than 10% from the mean of the three values. For near zero stress values, -10 to +10 MPa (-1450 to +1450 psi), it is more difficult to detect small changes with the contractometer and an acceptable range for repeatability is +/- 5 MPa.”

The repeatability of the contractometer has been established in several previous publications and no attempt was made to confirm this. Repeatability tests were taken with both the bent strip technique and the change of length method in two different baths, and the results are displayed in the following table:

<i>Test Method</i>	<i>Bent Strip</i>	<i>Change of Length</i>
Test Number 1	1058 psi	3072 psi
Test Number 2	1139 psi	3087 psi
Test Number 3	1221 psi	3455 psi
Mean	1139 psi	3205 psi
Variation from Mean	+/- 7%	+/- 8%

As can be seen from the data, both test methods have easily met the ASTM requirements.

## Resolution

Test characteristics have a profound influence on the resolution that may be obtained from each technique. Properties such as substrate material, substrate thickness, current density, test duration and the measuring instrument used all play important roles in determining the final resolution. For the contractometer and the bent strip tests, substrate properties are fixed as the test specimens are purchased from an outside vendor (although the helixes for the contractometer are available in other materials, they all have approximately the same elastic moduli as the original stainless steel type). With the change of length method, it is possible to use different substrate materials in order to increase resolution. Since a thinner, less rigid material will be deformed more by a given stress than will a thicker, more rigid material it is often beneficial to change the substrate. For example, switching from a carbon steel substrate with a thickness of .002 in to a brass substrate with a thickness of .001 in will result in approximately a fourfold increase in the overall deflection incurred, thereby bettering the resolution by the same factor. Another way to increase resolution is to plate to a greater deposit thickness. This will cause more deflection for a given stress level which will again yield an improved resolution. Unfortunately, as stated previously both the spiral contractometer and the bent strips become less accurate as overall deflection increases, so there is a tradeoff between the maximum obtainable resolution and the integrity of the measurement. Finally, the means by which measurements are taken plays the most important role in determining resolution. In general, readings from the bent strip tests and from the spiral contractometer are taken visually, from a graduated scale. This is obviously a limiting factor because there is a great deal of human error involved and there can be no precise determination of deflection. For the change of length method, a digital dial indicator with a resolution of +/-

.00005 in was used. This allowed for much more accurate measurements and eliminated the human error that hampered the other two approaches. Other studies in the past have used a displacement transducer in place of the dial, which can yield resolutions as fine as .000005 in. on the deflection of the strip. The authors found this level of precision to be unnecessary in an industrial environment as the noise from tank vibrations and fluid flow renders it meaningless.

## **Practical, Real World Comparison**

Having the ability to measure deposit stress holds the key to successful electroforming. In high-precision processes, maintaining the desired stress level assures the dimensional stability of electroforms, the flatness of mirrors and other optical components and the integrity of the mandrel-electroform system during electroforming. Stress in objects that undergo machining after electroforming needs to be maintained within a certain range to assure their machineability. When thick-wall items need to be electroformed, even moderate stresses in the deposit may build up to levels causing an ultimate distortion of the finished product, unless careful stress control is exercised during the entire deposition cycle. Furthermore, a certain level of tensile or compressive stress may be desirable in some electroforms depending on the geometry of electroformed parts to facilitate their separation from the mandrels.

The specifics of stress measurement in an industrial environment are quite different from those in a research facility. The real-world electroplater requires a measurement technique which is simple, quick, cost-effective, sufficiently accurate and, most of all, representative of the chemistry's performance. Each of the methods presented has advantages and disadvantages associated with its use, and the electroplater must decide which best suits his or her specific applications.

The change of length method can be extremely accurate, and can provide a wealth of information about the initial stages as well as the steady-state conditions of the electroplating process. It is also possible to obtain precise readings in a much shorter time period than that required by either of the other two devices. The bent strip method is a remarkably simple and quick means of evaluating deposit stress, and very little time is required to set up a single run. It also requires no expensive instrumentation, as the disposable strips, the in-tank cell and a measuring scale are all that is needed.

Of course, each approach has its drawbacks. With the change of length method it is necessary to use a new substrate for each test that is taken. Replacing the strip can be time consuming, and it is difficult to align the test strip in the I. S. Meter. Using bent strips to measure stress may be easy and takes little time, but the disposable strips are rather expensive and there is a definite loss of accuracy when they are used. Finally, the spiral contractometer probably has the most drawbacks associated with it. As previously discussed, setting up the contractometer is a very tedious process. Furthermore, as it was designed to be used with a special anode in a 4-Liter (1-gal.) beaker, the Contractometer is not well suited to testing directly in an industrial plating tank. Although our research showed correlation between tests taken directly in a plating tank and those taken with the same solution in the beaker, it was rather difficult to mount the Contractometer in a tank. Because of all the problems it introduced, using the contractometer externally was found not to be practical in the least. Some of the difficulties involved are keeping the solution at the same temperature as that in the original tank, the difference in geometry brought about by the cylindrical anode, and the much-reduced volume of the beaker. Additionally, stripping the nickel deposit from the stainless steel coil requires 4-6 hours, which virtually rules out the possibility of running back-to-back stress tests with just one or two available coils.

## Summary

The following table summarizes the most important characteristics of the three studied stress measurement techniques.

	<i>Bent Strip</i>	<i>Change of Length</i>	<i>Spiral Contractometer</i>
Resolution (4 $\mu\text{m}$ /.00015 in deposit)	~1500 psi	~600 psi	~8000 psi
Resolution (8 $\mu\text{m}$ deposit)	~300 psi	~150 psi	~2000 psi
Resolution (16 $\mu\text{m}$ deposit)	- - -	~50 psi	~600 psi
Typical Setup Time	5 min	10 min	25 min
Typical Test Duration	~20 min	~10 min	~60 min
Maximum Test Frequency	2 per hour	2 per hour	1 per day per available helix
Substrate cost	\$3.00 ea.	~\$0.50 ea.	\$75.00 ea. (reusable)
Ability to Use Different Substrates	No	Yes	Yes, with purchase of add-l helix
Cost of Measuring System	<\$250	N/A*	\$1000 (\$2000 w/ ext. Anode)

\*The authors don't know of any commercially manufactured instrument, based on the change of length method, which is currently available.

Several conclusions may be drawn from the preceding table. First, it is apparent that the bent strip technique is the most practical one for industrial applications as it provides decent accuracy and quick readings at a reasonable cost. And although the change of length method is the most accurate of the three, unfortunately the authors are not aware of any commercial manufacturers of this testing equipment. Finally, based on its poor resolution, slow turnaround times and general awkwardness in operation the spiral contractometer may have outlived its practical usefulness but will certainly remain the benchmark against which newer test methods will be gauged for quite some time.

Values in the previous chart reflect the following test properties and conditions:

Resolution values based on the stress equivalent to one unit of deflection on the appropriate scale.

Bent Strip tests taken with a Beryllium copper substrate, .002 in. thick, deflections read manually from a scale.

Change of Length tests taken with a Carbon Steel substrate, .001 in. thick, deflections read with a digital dial indicator.

Contractometer tests taken with a .01 in. thick Stainless Steel substrate, deflections read manually from an angular scale.

All tests performed at 35 Asf unless otherwise noted.

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