9-1977

Recent Advances in the Measurement of Residual Stress by X-Ray Diffraction

M. R. James
Northwestern University

Recent Advances in the Measurement of Residual Stress by X-Ray Diffraction

Abstract
overview talk on the recent work that has been done in the x-ray residual stress area and some of. the reasons why it is not always accepted as a val1d technique. The main problem that we have.to lo?k at is that when anyone measures macroscop1c resldual stresses, by whatever method - mechanical. techniques, x-ray techniques, ultrasonic technlquesone is always-measuring a different property of the material and trying to relate that to the macroscopic residual stress. There is no reason that residual stress must be the same for all these particular techniques. People get upset when the x-ray technique does not coincide with mechanically measured values, but there are definite reasons why it doesn't, and I want to go through some of those reasons. Measurements of residual stress by x-ray is a diffraction technique - it's not a radiography technique. I want to go through the principle~ very quickly and then emphasize some of the recent lnstrumentation advances developed in the last couple of years. Then I want t~ use the remaining time to discuss the situations where the validity of the x-ray technique is sometimes questioned.

Keywords
nondestructive testing, nondestructive evaluation

Disciplines
Materials Science and Engineering | Structures and Materials

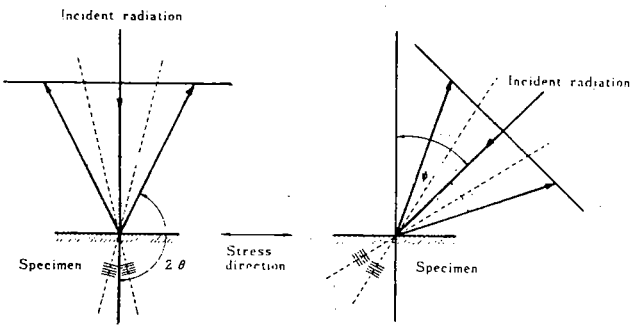
RECENT ADVANCES IN THE MEASUREMENT OF RESIDUAL STRESS BY X-RAY DIFFRACTION

M. R. James
Northwestern University
Evanston, Illinois 60201

I was asked to come here and give an educational overview talk on the recent work that has been done in the x-ray residual stress area and some of the reasons why it is not always accepted as a valid technique. The main problem that we have to look at is that when anyone measures macroscopic residual stresses, by whatever method - mechanical techniques, x-ray techniques, ultrasonic techniques- one is always measuring a different property of the material and trying to relate that to the macroscopic residual stress. There is no reason that residual stress must be the same for all these particular techniques. People get upset when the x-ray technique does not coincide with mechanically measured values, but there are definite reasons why it doesn't, and I want to go through some of those reasons. Measurements of residual stress by x-ray is a diffraction technique - it's not a radiography technique. I want to go through the principles very quickly and then emphasize some of the recent instrumentation advances developed in the last couple of years. Then I want to use the remaining time to discuss the situations where the validity of the x-ray technique is sometimes questioned.

Metallic materials in industrial use are in general aggregates of polycrystalline materials with different orientations. The x-ray diffraction technique obtains information only from those crystallite lattice planes which are oriented to satisfy the Bragg condition of diffraction. The first thing this means is that it is a selective technique. We're only measuring information from certain lattice planes in the crystal, and we're trying to relate the strain from those lattice planes to a bulk residual stress on the surface of the sample.

In Fig. 1 we see that the incident radiation diffracts from more than one set of grains, but the same crystallographic lattice planes, because the grains are oriented differently within the polycrystalline material. Now, what this allows us to do is change the orientation of the incident radiation, as in Fig. 1b, and examine the same hkl planes now oriented differently with respect to the stress direction. Using Bragg's law, given in Fig. 1, we can correlate this angle of diffraction with the interplanar spacing, and because we have a change in the interplanar spacing between the two inclinations of the sample due to the resultant stress component on each of the lattice planes, we have a resultant shift in the diffraction angle.



$$\lambda = 2d\sin\theta$$

$$\frac{\Delta d}{d} = -\cot\theta\,\Delta\theta$$

Figure 1. Principles of x-ray residual stress measurement

To show exactly what is done in the x-ray technique we normally measure the interplanar spacing, usually signified by d, which is parallel to the surface. Then we can either rotate the sample or we can rotate the x-ray beam and measure the planes at an angle, ψ, with respect to the surface. From the change in interplanar spacing we have a strain which can be related to the stress.

Because the x-rays only penetrate the surface of the material to a shallow depth of, say, 25μm or so, we're only going to get a surface residual stress. Also, because of the selective nature of the x-ray diffraction technique, the gauge length we're talking about here is just the interplanar spacing of the crystallographic planes.

In order to get the stress component from the strain, we have to make various assumptions. The first is that on the surface of the specimen we have a biaxial stress, which is reasonable, because we can't have a stress normal to the surface of the material and we're only looking at a shallow depth. The second assumption we make is that it is a homogeneous material, or at least if we're looking at one phase, that it is uniform. The third assumption, and this is the one that gets us into trouble, of course, is that isotropic elasticity applies. It's really not a bad assumption, because we are looking at a lot of grains in the surface of the sample. However, when preferred orientation is present, the anisotropic nature of the grains complicates the situation, but we will deal with this later.

Figure 2 shows the coordinate system we're talking about. ψ is the angle between the normal to the diffraction plane and the normal to the sample surface. We want to determine the stress, σ_ϕ, in the direction given by ϕ . Stress is a tensor and dependent on direction, so we arbitrarily define ϕ as an angle from one of the principal axes. Normally, we don't know what ϕ is; we just say we're looking at the longitudinal stress or the tangential stress or so forth.

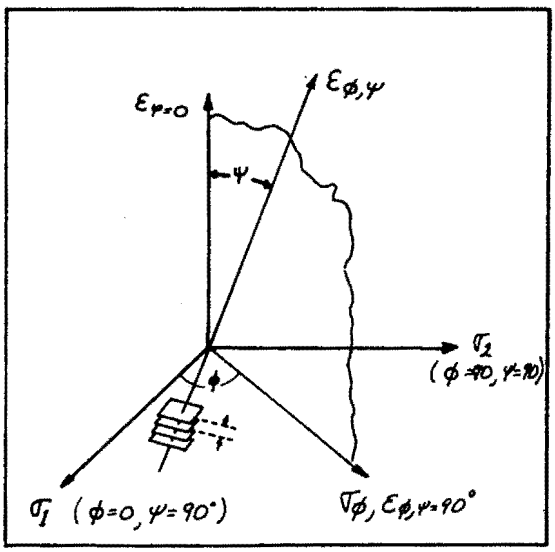


Figure 2. Illustration of symbols used in x-ray stress measurement.

I don't want to spend any time on the derivation of the equations used in residual stress techniques, they are found in numerous textbooks, but I do want to emphasize some of the points the equations imply. The relation between the stress and strain is given in Fig. 3. The first equation forms the basis for the so-called 'sin $^2\psi$' method of x-ray residual stress analysis. The terms $s_2/2$ and s_1 are the x-ray elastic constants. Now, if isotropic theory really applies, then it can be shown that these x-ray elastic constants are functions of the bulk mechanically measured values. This isn't always true because the isotropic theory doesn't always apply, so a lot of times we actually measure these values.

$$1)\quad \varepsilon_{\varphi,\psi} = \frac{s_2}{2}\sigma_\varphi \sin^2\psi + s_1(\sigma_1+\sigma_2) = \frac{d_{\varphi,\psi}-d_o}{d_o}$$

$$2)\quad \frac{s_2}{2} = \frac{1+\nu}{E} \qquad s_1 = -\frac{\nu}{E}$$

$$3)\quad m^* = \frac{\partial \varepsilon_{\varphi,\psi}}{\partial \sin^2\psi} = \frac{1}{d_o}\frac{\partial (d_{\varphi,\psi})}{\partial \sin^2\psi} = \frac{s_2}{2}\sigma_\varphi$$

FROM 3 $\qquad \sigma_\varphi = m^* / \frac{s_2}{2}$

Figure 3. Relations between stress and strain.

The important feature of this equation is that if we plot the strain versus $\sin^2\psi$ at a number of inclinations, then that line will be linear, it will be straight, and we can get the surface stress, σ_ϕ, just from calculating the slope and knowing the elastic constants.

Now, the fact that there is a linear relationship between $\sin^2\psi$ and the strain allows us to use what is normally called a two-tilt technique. We assume a straight line; therefore, we only need to measure the interplanar spacing at two particilar ψ inclinations. We can go even further and make another simple trigonometric substitution and we get the peak shift, the change in the Bragg angle related to the surface stress σ_ϕ, as given in Fig. 4.

TWO TILT METHOD

$$\sigma_\varphi = \frac{E}{1+\nu}\frac{1}{\sin^2\psi}\frac{\cot\theta}{2}(2\theta_o - 2\theta_\psi)$$

$$= K\,\Delta 2\theta$$

$$\text{where } K = \frac{E}{1+\nu}\frac{1}{\sin^2\psi}\frac{\cot\theta}{2}$$

Figure 4. Expression for σ_ϕ

In this case, the cotangent θ is a slowly varying function of θ so we can lump all of this into one term and call it the stress constant, K. This stress constant can be measured for the particular material that one is looking at. The two-tilt method is easy, especially when doing the measurement by hand, but the $\sin^2\psi$ method is more accurate.

Now, how is this measurement accomplished? Years ago it used to be done with film techniques; now it is normally done on a diffractometer in the back reflection region so that instrumental errors and systematic aberrations are minimized. We determine the profile of the diffraction curve and use curve fitting procedures to determine the actual peak location. By hand, this measurement now, normally, takes from a half an hour to an hour and is rather tedious, a very boring type of measurement, so computer applications have come in very handy. At Northwestern we've developed a completely automated package allowing for complete optimization of data collection. The measurements now take anywhere from five minutes to, say, half an hour depending on the statistical accuracy that the operator desires. The program includes sample alignment and either the two-tilt or $\sin^2\psi$ methods. However, the real instrumental improvements in the past few years have tended towards dedicated x-ray stress analysis devices, such as the fast stress system manufactured here in the United States,[1] and the Strain Flex[2] unit manufactured in Japan. These types of units enable stress measurements to be done in 1 to 5 minutes, but only at an accuracy, at best, of plus or minus 3,000 psi, probably more like 6,000 or 7,000 psi. They are reasonably expensive, the initial cost is \$60-\$65,000 and they suffer from the fact that they are not very flexible and are definitely not portable.

At Northwestern, we are completing work on the feasibility of applying a position sensitive detector to the measurement to improve the speed many times over[3]. The detector simultaneously records the entire diffraction profile without any movement. Figure 5 depicts a typical profile obtained from a mild steel sample in 60 seconds. The spatial resolution, that is, the resolution along the length of the detector, is about 180μm.

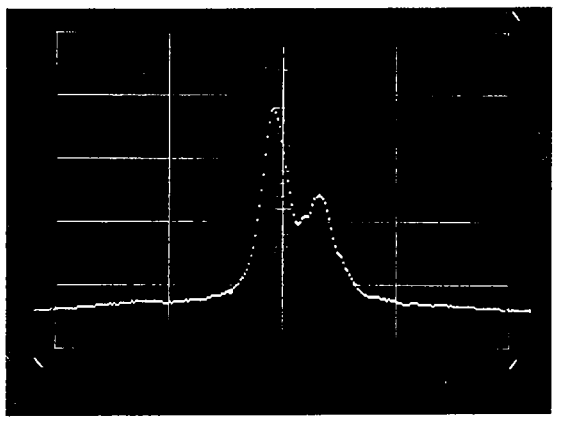


Figure 5. 211 diffraction peak from SAE 1045 sample.

We simultaneously collect data across the entire diffraction profile, therefore not wasting time collecting data point by point over the profile. The data is stored in a multichannel analyzer and then the profile is dumped to a computer. Curve fitting techniques are used to define the peak position, and while the computer is typing a report, we're accumulating data from the next ψ tilt, and so on. This is a commercial detector, by the way, and can be bought on the market right now. The packaging arrangement isn't very good and we're trying to get the commercial manufacturer to repackage it in a different form so it's about the size of a pocket dictionary so that the pre-amplifiers are actually in back of the detector. We're combining this detector with a new light-weight air cooled portable x-ray tube. The unit is 6 inches long, 2 inches in diameter and only weighs about 5 pounds. With the PSD and such an x-ray source, one can build a nice small device that one man can carry with a remote package for the power supply and detector electronics which is about the size of an attache case and weighs about 50 pounds. The actual detector assembly will be about 20 pounds.

What kind of times are we talking about? Well, on mild steel samples we can determine the residual stress to an accuracy of plus or minus 5,000 psi in 10 seconds - no problem. Hardened steel samples have a very broad peak profile, usually about 15° to 20° from background to background. To do the analysis on such a sample to plus or minus 10,000 psi takes about thirty seconds. So, this is considerably faster than existing techniques, and we hope to demonstrate such a unit to manufacturers in the next month and a half or so, and maybe someone will actually get one of these things out on the market within a reasonable amount of time.

For the remainder of the talk, I would like to discuss some of the problems with the x-ray residual stress technique. We're talking about measuring a residual macrostress, a stress that is long range, and it would be nice to be able to correlate this with mechanical measured values, but what is more important is to correlate it with fatigue life predictions, quality control, and so forth, and thereby know when the technique is useful.

There are problems which arise from the selective nature of x-ray diffraction in the peak shift measurement. Residual stresses are associated with both macrostrains and microstrains. Elastic deformation gives rise to uniform macrostrains which cause a shift in the position of the x-ray peak; this peak shift is then related to a macroscopic stress system. When a metal is plastically deformed, microstrains or variations in the interplanar spacing of the order of the subgrain size are introduced. They arise due to the energy or strain field produced by faulting, segregation of solute atoms, or dislocations. On the subgrain level this produces a distribution in the average interplanar spacing which gives rise to broadening of the diffraction profile and unfortunately a peak shift. It is this dependence of the peak shift on microstrains that is considered to be a problem in the residual macrostress analysis, because instead of measuring a pure macrostress, the quantity determined by the x-ray technique may be both a macrostress and a microstress superimposed upon each other.

The first evidence for the contribution of microstrain came from the existence of oscillations in d vs $\sin^2\psi$. Classical theory of x-ray residual stress analysis predicts that the relation between the interplanar spacing or strain and $\sin^2\psi$ should be linear. Experimental evidence has shown that this is not always the case. As shown in Fig. 6, oscillations can exist which not only prohibit the use of the 'two-tilt' method, but lead one to question the basic formulae in the x-ray technique.

Recently, Marion and Cohen[4] have derived a model to account for this effect based on the theory of Weidemann[5] which describes an orientation dependent relief of the elastic stresses present in a polycrystalline aggregate. During deformation texturing develops because some crystallite regions will have a tendency to rotate to a more energetically favorable position so that multiple slip or some dynamic recovery process takes place. The elastic stresses are relieved by the local plastic deformation. A microscopic distribution of strains will be present which is directly related to the texture developed during deformation. The distribution of the microstrains is non-random and produces oscillations in d vs $\sin^2\psi$. Marion and Cohen showed that the oscillations do indeed correspond to the developed texture, as shown in Fig. 6.

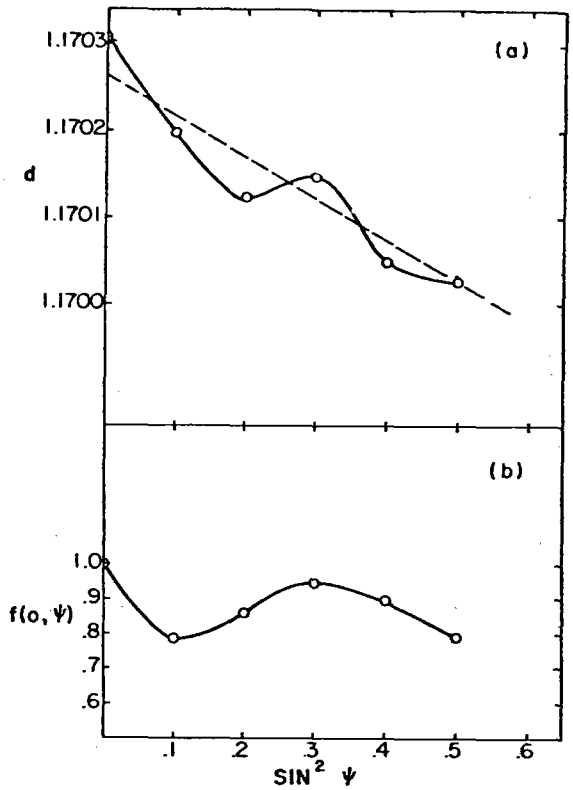


Figure 6 (a) d vs $\sin^2\psi$ for ARMCO iron specimen deformed in tension to a true strain of 18.4 pct. 211 peak with $Cr_{K\alpha}$. σ_φ = -10,148 psi.

(b) Texture distribution function for sample described in (a).

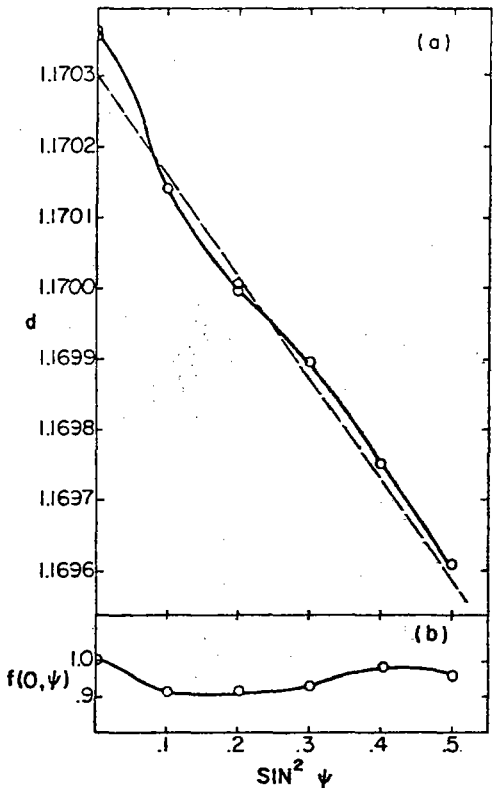


Figure 7 (a) d vs $\sin^2\psi$ for SAE 1045 steel deformed in tension to a true strain of 13 pct. 211 peak with $Cr_{K\alpha}$. σ_φ = -30,745 psi.

(b) Texture for sample described in (a).

They showed that a simple texture function can be obtained from the intensity of the diffraction peak and developed formulae to include the distribution of microstrains. Figure 6 depicts the oscillations found in an Armco iron sample. This has about .01 weight % C and is a reasonably homogeneous alloy. Figure 7, a 1010 steel, shows that the oscillations still exist but are very much diminished. Marion and Cohen showed that the effect is much more pronounced in homogeneous metals and their technique enables one to obtain the macrostress in the presence of oscillations in d vs $\sin^2\psi$. This technique is also automated in our residual stress package at Northwestern and adds maybe 10 minutes when using a diffractometer.

Another problem occurring when plastic deformation has taken place is that the microstrains may show a linear dependence of d vs $\sin^2\psi$, not just the oscillations accounted for by the Marion-Cohen method. As I said before, a linear dependence of d on $\sin^2\psi$ is usually associated with a macrostress. If it is possible to have microstresses existing which also yield a linear dependence, then the measured quantity is ambiguous because it is the superposition of the two types of stresses. This effect has been termed a 'fictitious' or 'baseline' stress by the Japanese and a 'pseudo-macrostress' by the Americans. It is an anomally in the x-ray technique which leads to an error in the measured macrostress.

An experimental technique to distinguish between residual macrostress and microstress concerns measuring the residual lattice strain on new surfaces as the specimen is progressively thinned. One expects that the microstress will remain sufficiently constant throughout the cross section while macrostresses must change to obey equilibrium. Characteristic results[5] on cylindrical specimens undergoing tensile plastic deformation are shown in Fig. 8. The stress measured in the direction of deformation through the cross section of the specimen shows that the stresses are in equilibrium in

the copper sample indicating a true macrostress. In the high carbon steel on the right, however, equilibrium is not obtained indicating that an anomalous stress is being measured and superimposed on a real macrostress. From these types of experiments it has been shown that this anomalous macrostress is measured only when axisymmetric plastic deformation takes place, that is, only when plastic deformation in one direction occurs such as in uniaxial tension or when rolling takes place.

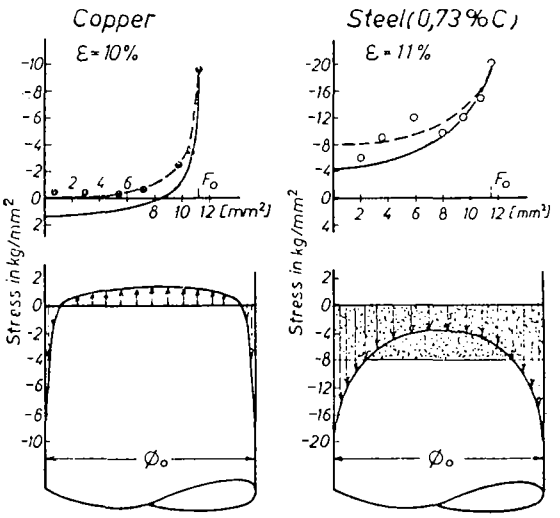


Figure 8. Distribution of residual stress in deformed specimens of Cu and plain steel from Ref. 5.

There are a number of reasons why this occurs. The most obvious is that the residual stress is being determined in only one phase of the material whereas other phases may have balancing residual stresses. Also back stress and work hardening occur differently between phases setting up the microstrain system which we are selectively sampling. Other mechanisms have been proposed (a good summary is given in Refs. 6 and 7) but only one method has been proposed to accurately obtain the true macrostress in the presence of this phenomena. Taira et al[8] have shown that in steel, the 'baseline' stress is dependent on the carbon content and have proposed a method, valid only for steel, whereby one can determine the 'fictitious' component of the peak shift and thereby obtain the macrostress. Their procedure is purely empirical and does not shed much light on the mechanism or mechanisms responsible for the anomally.

What is clear from experimental results is where one might expect the two anomalies discussed (the oscillations in d vs $\sin^2\psi$ and the pseudo-macrostress) to become important. Oscillations in d vs $\sin^2\psi$ are found most often in homogeneous materials whereas the pseudo-macrostress problem is found in heterogeneous alloys. In both cases, the anomally takes place only after axisymmetric plastic deformation such as that caused by uniaxial tension or rolling. Residual stress caused by heat treating, shot peening or grinding yields corresponding values when measured by x-ray diffraction and mechanical methods[9].

Thank you.

REFERENCES

1. E. W. Weiman, J.E. Hunter, and D.D. McCormack, Meta Progress, 96, No. 1, 88 (1969).
2. Y. Sakamoto, Adv. in X-Ray Analysis, Vol. 20, in press.
3. M.R. James and J.B. Cohen, Adv. in X-Ray Analysis, Vol. 18, 466 (1974).
4. R.H. Marion and J.B. Cohen, Adv. in X-Ray Analysis, Vol. 18, 466 (1974).
5. W. Widemann, Ph. D. Thesis, Techische Hochsule, Aachen, Germany (1966).
6. E. Macherauch, Exp. Mech., 6. 140 (1966).
7. R. I. Garrod and G. A. Harohes, Brit. Jrl. Apply. Phys., 14, 433 (1963).
8. S. Taira, K. Hayashi, and S. Ozawa, Mechanical Behavior of Materials, The Society of Materials Science, Japan, 287 (1974).
9. H. R. Letner, Trans. ASME, 77, 1089 (1955).

DISCUSSION

PROF. JOHN TIEN (Columbia University): Are there any questions?

DR. ROY SHARPE (Nondestructive Testing Centre, Harwell Labs): Is this an exercise in scientific stimulation or is there a real need for this? It seems that every ten years or so there is an interest in stress measurements, and then it dies away and people sort of learn to live with stress.

DR. JAMES: I think that's one of the problems with the field. It hasn't gotten out of the laboratory stage and to the field to be used for a fatigue life prediction, or for quality control. But a country like Japan, for instance, which is much smaller than us, smaller population and so forth, they have five times the number of residual stress devices out in industry and they're using it as a real in-field use tool. Apparently they feel it is a worthwhile tool.

DR. BOB ERWIN (Northrop Aircraft Div.): How do you introduce your material stress constant in this new equipment in such a rapid manner?

DR. JAMES: I beg your pardon? I didn't understand.

DR. ERWIN: How do you introduce your material stress constant in such a rapid manner? You said ten seconds in this new equipment.

DR. JAMES: Oh, well, that assumes you already know what your x-ray elastic constants are. You are determining the stresses only to plus or minus 10,000 psi, so the use of an average stress constant is satisfactory. For bulk mechanical values you are within 20 percent of the actual x-ray elastic constants, unless heavy plastic deformation has occurred. What is important is that the sample is in a compressive mode and not tensile mode or it's 10,000 and not 50,000 psi. It's those kinds of gross changes that are used as life prediction type of changes and not just changes on the order of 1 or 2 psi.

DR. DWAYNE JOHNSON (Failure Analysis Associates): What total weight is the package?

DR. JAMES: Well, I think we can get the part that is actually carried by hand to be about 20 pounds. You hold it by two hands and you do the measurement at one angle and then you actually move the tube and detector to the 45° tilt and take the data at the other angle. I suppose one could put on two of these detectors and two x-ray tubes, but you tend to make it very heavy, although you are saving half the time. Then you have the power supply and a micro-computer to analyze the data which can be 10 or 20 feet away. With an air cooled x-ray tube and solid state power supply, you don't have to worry about cooling water, etc. and everything plugs into a 110 line.

DR. SEYMOUR FRIEDMAN (Naval Ship Systems): In the use of a position detector, do you give the actual position of the diffraction peak----

DR. JAMES: What we do first of all is to calibrate the detector using a known standard so we can obtain the peak 2θ value along the relative position of the detector. All data is transformed into $^{\circ}2\theta$ because in residual stress measurement you have to do Lorentz-polarization and absorption corrections to the intensity with subsequent curve fitting procedures to determine the peak position.

DR. FRIEDMAN: The actual determination of where the value of 2θ is, that's not that drastic?

DR. JAMES: We're using a least square parabolic curve fitted to the upper region of the peak profile. We're using as many of the data points as we can and still remain in the parabolic region to improve the statistics and so forth. We determine the actual peak position in degrees two theta ($^{\circ}2\theta$) for each ψ tilt.

DR. FRIEDMAN: Can you get enough counts in 30 seconds to get a reading?

DR. JAMES: Oh, yes, when you're talking about a hardened steel sample and separating the detector into, say, 256 channels, I get usable information over about 120 of those channels. Using the theory of random counting statistics and propagating it through the curve fitting and stress formulae give us what I'm calling the precision of plus or minus 10,000 psi. That is the counting statistical precision, and we have shown that it indeed corresponds to the observed precision over many repeated runs.

DR. FRIEDMAN: I see. If you waited longer you would get a better sample?

DR. JAMES: Yes. If you wait longer, say the order of 200 seconds on a hardened steel sample, you can get statistical counting errors of plus or minus 2,000 psi.

PROF. TIEN: You don't happen to have one of these on you, do you?

DR. JAMES: No, but as I said, we hope to demonstrate the hand held unit within two months.

PROF. TIEN: One more question.

DR. ERWIN: Will this equipment handle titanium?

DR. JAMES: There's no reason why not, except that the proper characteristic radiation should be $Co_{K\alpha}$ or, preferrably $Cu_{K\alpha}$ which would mean changing x-ray tubes. One has a large flourescence problem when using $Co_{K\alpha}$, a lot of $Ti_{K\alpha}$ is produced, but this doesn't prohibit the use of the position sensitive detector.

PROF. TIEN: Thank you.