A METHOD OF DETERMINING THE ELASTIC PROPERTIES OF ALLOYS IN SELECTED CRYSTALLOGRAPHIC DIRECTIONS FOR X-RAY DIFFRACTION RESIDUAL STRESS MEASUREMENT

Paul S. Prevéy

ABSTRACT

A technique and apparatus are described for obtaining the elastic constant E/(1 + ν) in selected crystallographic directions for the purpose of calibrating x-ray diffraction residual stress measurement methods. The preparation of a simple rectangular beam specimen with two active electrical resistance strain gages applied to the test surface is described. Samples are clamped in a diffractometer fixture designed to minimize displacement errors, and loaded in four-point bending to several stress levels below the proportional limit. A method is described for calculating E/(1 + ν) and an estimate of the experimental error.

Values of E/(1 + ν) obtained for several alloy-(hkl) combinations are presented. The results indicate that several alloys of current commercial interest exhibit significant elastic anisotropy.

INTRODUCTION

The strain in the direction defined by the angles ϕ and ψ in a sample of homogeneous material when under conditions of plane stress may be expressed in terms of the stress in the surface of the sample as:

$$\in_{\phi\psi} = \left(\frac{1+\nu}{E}\right)\sigma_\phi \sin^2\psi - \frac{\nu}{E}(\sigma_1 + \sigma_2) \tag{1}$$

In this expression, the quantities σ_1 and σ_2 are the principal stresses, σ_ϕ is the stress in the plane of the surface of the sample in the direction defined by the angle, ϕ, as shown in Figure 1; and ν and E are Poisson's ratio, and the elastic modulus of the material.

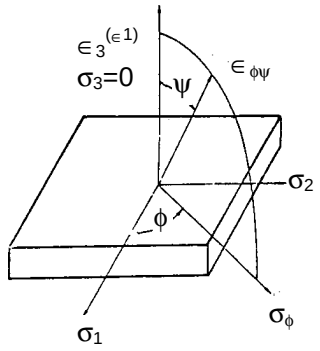

Fig. 1 - Surface Under Plain Stress

Several x-ray diffraction techniques may be employed to solve equation (1) for the stress, o_ϕ, which may be either applied or residual. If the strain, $\varepsilon_{\phi\psi}$, is determined experimentally as the strain in the crystal lattice for at least two values of the angle ψ, σ_ϕ may be expressed in terms of the strain in the crystal lattice, as,

$$\sigma_\phi = \left(\frac{d_\psi - d_\perp}{d_\perp}\right)\left(\frac{E}{1+\nu}\right)\frac{1}{\sin^2\psi} \tag{2}$$

Equation (2) is the working equation for the "two-angle" technique, in which the lattice spacing is measured at ψ = 0 and ψ = ψ to determine $d_\perp$ and d_ψ, respectively. The quantity E/(1 + ν) is the elastic constant required to calculate the macroscopic stress, σ_ϕ, from the strain measured in a specific crystallographic direction. For an accurate calculation of σ_ϕ, E/(1 + ν) must be determined in the direction normal to the lattice planes employed for stress measurement.

In the measurement of residual stresses, it is not uncommon to find mechanical values of E and ν being employed to reduce x-ray diffraction data. For

Advances in X-Ray Analysis, Vol. 20, Plenum Press, New York, NY, (1977) pp. 345-354.

isotropic materials, this procedure will provide results sufficiently accurate for most applications. However, many of the alloys of current interest in the aerospace and nuclear industries are highly anisotropic. The use of a mechanically determined value of $E/(1 + \nu)$ to reduce x-ray diffraction residual stress measurement data for these alloys can lead to errors as high as 80%.

It is the purpose of this paper to describe a technique for determining $E/(1 + \nu)$ in four point bending which is used routinely in the author's laboratory. Specific elastic constant data obtained for several alloys is also presented to emphasize the importance of eliminating one of the major sources of systematic experimental error encountered in the measurement of residual stresses by x-ray diffraction techniques.

TECHNIQUE

Samples were prepared in the form of simple rectangular beams with nominal dimensions of 4.0 x 0.750 x 0.060 inches as shown in Figure 2. All surfaces of the samples were finish ground holding the thickness and width of the beam to a tolerance of ±0.001 inch. Unless a specific heat treatment of the alloy was to be investigated, test coupons were annealed prior to grinding to the final dimensions. Fully annealed samples will generally provide diffraction peaks in the high 2θ range which are sufficiently sharp to allow separation of the $K\alpha_1$-$K\alpha_2$ doublet. When possible, diffraction data was taken using the $K\alpha_1$ peak. After final grinding, a region 1 in. long in the center of one face of the sample was electropolished to a depth of approximately 0.010 in. to remove the plastically deformed layer produced by grinding.

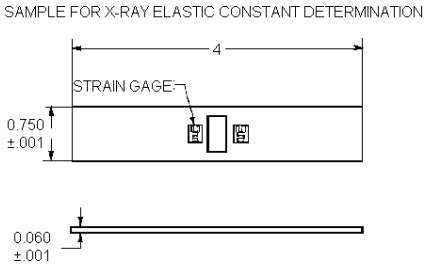

Fig. 2. - Four-Point Bending Sample

Two electrical resistance strain gages were applied to one face on either side of the center of the sample in the electropolished region. The gages were aligned to measure the outer fiber strain in the direction parallel to the longitudinal axis of the sample. The two gages were positioned on either side of the center of the sample leaving a bare region approximately 1/2 in. wide to be irradiated during the determination of the elastic constants. The strain gages were bonded to the electropolished surface using a furnace curing epoxy cement to provide maximum stability of the strain gage bond. Room temperature curing contact epoxies were found to be less stable, and appeared to creep under sustained load.

The two active gages on the sample beam were wired with two identical gages attached to a temperature compensating block of the same alloy to form a full bridge circuit as shown in Figure 3. The circuit was arranged so that the voltage across the bridge was proportional to the sum of the strains measured by the two active gages. The strain in the diffracting area between the two gages was assumed to be equal to the average strain measured by the two active gages. In this manner, any linear strain gradient along the length of the beam under four-point loading was eliminated. A protective coating was applied to the strain gages, and the entire assembly was allowed to cure at room temperature for at least 48 hours prior to loading. After curing, the bridge circuit was attached to a strain indicator, and the sample was flexed to approximately 80% of the yield strength several times until the gages would return to a reading of zero strain without hysteresis.

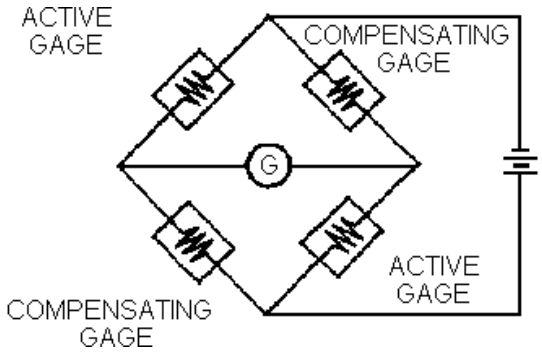

Fig. 3. - Strain Measurement Circuit

The strain gages were calibrated by placing the instrumented sample in a four-point bending fixture, and dead weight loading the sample to a known stress level. Knowing the applied load, the linear dimensions of the sample, and the moment arm of the four-point bending fixture, the actual stress in the outer fiber of the sample in four-point bending was calculated from the relation,

$$\sigma = 3Pa / bh^2 \qquad (3)$$

where P is the applied load, a is the moment arm of the bending fixture, b is the width of the sample and h is the sample thickness. An effective gage factor for the active gages on the sample was calculated from

the ratio of the actual stress to the stress indicated using the strain gage manufacturer's supplied gage factor. Systematic errors due to misalignment of the strain gages and variations in the glue bond thickness were eliminated by using the effective gage factor to calculate the applied stress when the sample was placed on the diffractometer.

The samples were placed on the diffractometer in a four-point bending fixture designed to load the sample surface in tension while holding the diffracting volume rigidly over the center of the goniometer. The apparatus, shown in Figure 4, consists of a four-point bending fixture and a clamp which is bolted to the ψ table and shimmed to position the diffracting volume of the sample over the ψ table-goniometer axis of rotation. The sample is held in the spring loaded clamp so that the bending fixture moves outward radially from the center of the ψ table as the sample surface is loaded in tension. The clamp minimizes displacement of the sample from the center of rotation of the goniometer as loads are applied.

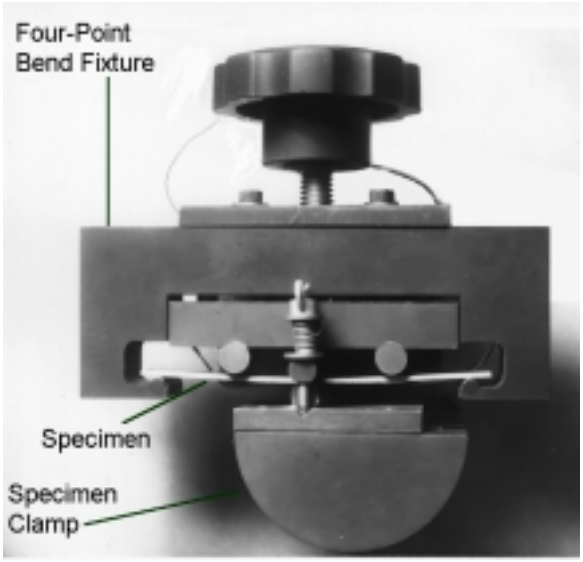

Fig. 4. - Four-Point Bending Apparatus

When positioned in the four-point bending apparatus, the samples were stressed to approximately 5%, 40%, and 75% of the yield strength of the alloy. At the highest and lowest levels of applied stress, calculated from the effective gage factor previously described, the lattice spacing of the selected set of planes was measured five or six times at ψ angles of 0.0 and lattice strain measured was linearly dependent upon 45.0 degrees. Two or three measurements were made at the intermediate stress level to assure that the the applied stress. A deviation from linearity indicates the failure of a strain gage bond or excessive loading beyond the yield strength of the material. The data were concentrated at the highest and lowest loads to minimize the uncertainty in the slope of a plot of applied stress versus the change in lattice spacing, Δd, between ψ = 0.0 and ψ = 45.0 degrees.

The lattice spacing at each load and ψ angle was determined using a parafocusing technique. The diffraction peak vertex was found using a least-squares parabolic regression procedure employing five points chosen in the top 15% of the diffraction peak after correction for a linearly sloping background intensity and for the Lorentz polarization and adsorption factors. The inverse intensity at each of the five points chosen was determined by measuring the time required to obtain 100,000 counts. Calculation[2] of the systematic error due to the curvature of the samples under maximum load (9.0 in. minimum radius of curvature) at the diffraction angle, 2θ, indicates a maximum error in 2θ of 0.015 degrees. As this error is on the order of the random error due to counting statistics, no correction was made for sample curvature. The shift in the lattice spacing, Δd, was then calculated between ψ = 0.00 and ψ = 45.00 degrees for each repeat measurement and plotted against the applied stress. Two sets of data taken for the (220) planes of Inconel 718 are shown in Figure 5.

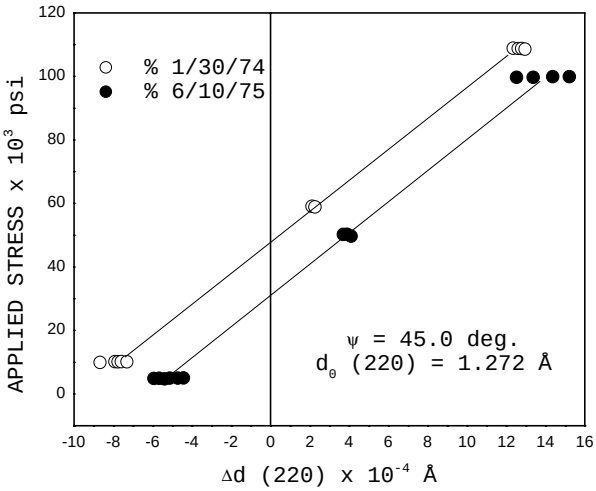

Fig. 5. - Change in d (220) Versus Applied Stress, Inconel 718, Annealed

TABLE 1

X-Ray and Mechanical Elastic Properties

Material	(hkl)	λ, Kα	2θ (deg.)	E/(1+v) x 10^6 psi (hkl)	(Mech.)	Ref.	Δ %	K_{45} (ksi/deg.
Iron Base, BCT								
4340, 50 R_c	(211)	Cr	156.0	24.5 ± 0.4	22.7	3,4	+7.9	89.3
410 SS, 22 R_c	(211)	Cr	155.1	25.6 ± 0.1	22.6	3,4	+13.2	98.4
410 SS, 42 R_c	(211)	Cr	155.1	25.1 ± 0.2	22.6		+11.0	96.7
422 SS, 34 R_c	(211)	Cr	154.8	26.4 ± 0.2	22.7	3,4	+16.3	103.2
422 SS, 39 R_c	(211)	Cr	154.8	26.1 ± 0.2	22.7		+14.9	103.4
Iron Base, FCC								
304 SS	(220)	Cr	129.0	20.2 ± 0.6	21.9	5,6	-7.7	170.0
Incoloy 903	(220)	Cr	128.0	31.2 ± 0.4	17.3	3,4	+80.3	264.0
Incoloy 800	(220)	Cr	129.0	23.4 ± 0.6	21.4	4,6	+9.3	196.0
	(420)	Cu	147.0	21.5 ± 0.4	21.4		+0.5	110.0
Nickel Base, FCC								
Inconel 718	(220)	Cr	128.0	31.2 ± 0.3	22.7	3,4	+37.4	263.0
	(220)	Cr	128.0	31.4 ± 0.7	22.7		+38.3	265.0
	(331)	Cu	145.0	19.7 ± 1.0	22.7		-13.2	109.0
	(331)	Cu	145.0	20.3 ± 0.3	22.7		-10.6	112.0
Inconel X750	(220)	Cr	131.0	36.8 ± 1.2	23.9	3	+53.9	301.0
Inconel 600	(220)	Cr	131.0	21.1 ± 0.5	24.0	3,4	-12.1	174.0
	(420)	Cu	151.0	23.1 ± 0.1	24.0		-3.7	105.0
Monel K500	(420)	Cu	150.0	21.0 ± 0.3	19.7	3,4	+6.6	98.4
Copper Base, FCC								
85 Cu-15Ni	(420)	Cu	146.0	18.6 ± 0.3	13.6	3	+36.8	98.8
Aluminum Base, FCC								
7075	(311)	Cr	139.0	8.83 ± 0.07	7.82	3	+12.9	56.9
Titanium Base, HCP								
Ti-6Al-4V	(213)	Cu	142.0	12.2 ± 0.07	12.3	7	-0.8	74.0
Ti-6Al-2Sn-4Zr-2Mo	(213)	Cu	140.7	14.8 ± 0.2	12.5	3,5	+18.4	92.3

A set of data was reduced by determining the slope of the applied stress as a function of the measured change in the lattice spacing, Δd, by linear least-squares regression. The uncertainty in the slope was obtained from the least-squares fit assuming all random error occurs in the determination of the change in the lattice spacing, Δd. The elastic constant is then calculated from the relation:

$$\frac{E}{(1+\nu)_{(hkl)}} = m^* \sin^2\psi \; d_{o(hkl)} \qquad (4)$$

where m* is the partial derivative of applied stress with respect to the change in lattice spacing, i.e., the slope of the data plot; d_o is the unstressed lattice spacing, taken to be the value for ψ = 0; and ψ is the range of the angle ψ, 45.0 degrees.

RESULTS AND DISCUSSION

The results obtained to date for iron, nickel, copper, aluminum, and titanium alloys are presented in Table I. The data have been grouped by base alloy and crystal structure. The commercial alloy name, (hkl), wave length, and approximate diffraction angle, 2θ,

are given. The elastic constant E/(1 + ν), determined in the direction normal to the (hkl) planes, is listed with an uncertainty equal to ± one standard deviation. The published mechanically determined value of E/(1 + ν) is included for comparison, along with the percent difference between the (hkl) and mechanical values, Δ%. The quantity K_{45} giving the approximate stress required to produce a one degree, 2θ, shift in the diffraction peak position for a ψ rotation of 45.00 degrees is calculated from the (hkl) value of E/(1 + ν) as:

$$K = \frac{E}{(1+\nu)} \frac{1}{\sin^2{}_\psi} \left(\frac{\cot \theta_o}{2} \right) \frac{\pi}{180} \tag{5}$$

The data in Table I are presented as empirical results, and no attempt shall be made to explain the origins of the anisotropy observed. Several observations can, however, be made concerning the data and technique. First, the most complex alloys, such as Inconels and Incoloys appear to be most elastically anisotropic. The greatest variation between mechanical and (hkl) values of E/(1 + ν) occurs for Incoloy 903 and Inconel X750 which differ by 80.3 and 53.9%, respectively in the (220) direction. The large deviation for Incoloy 903 occurs because the elastic constant in the (220) the Inconel or Incoloy series. Second, the degree of anisotropy appears to be highest for the lower order planes. Data obtained for Incoloy 800, Inconel 718, and Inconel 600 indicates a greater degree of anisotropy for the (220) direction than in the (331) or (420) directions.

Regarding the repeatability of the technique itself, repeat data taken on the same sample of Inconel 718 in both the (220) and (331) directions at intervals of approximately 18 months show agreement within the estimated experimental error. These results are shown graphically in Figure 5, and are presented as separate entries in Table I. Measurements on two samples of 422 stainless steel with slight differences in hardness also agree within the estimated error. These repeat measurements on the same sample, and on separate but nearly identical samples appear to indicate that the random error in the determination of E/(1 + ν) is approximated well by the uncertainty in the calculation of m*.

The major source of systematic error is in the calculation of the applied stress. Care must be taken to determine the effective gage factor using a four-point bending apparatus which does not produce a tensile component. Ball bearing pivoted four-point bending grips of the type used for fatigue testing were found to give excellent repeatable results. Fixtures employing pins in sliding contact with the sample are generally not suitable. The determination of Δd is less susceptible to systematic error. The presence of a residual stress in the sample, or displacement of the sample from the center of rotation of the goniometer results in a shift in the intercept of the plot of applied stress versus Δd. The slope of plot as shown in Figure 5, and therefore E/(1 + ν), is not effected by either sample displacement or the presence of a residual stress.

CONCLUSIONS

The results indicate severe elastic anisotropy in several of the alloys of current interest in the aerospace and nuclear industries. Failure to determine the elastic constant E/(1 + ν) in the direction normal to the lattice planes employed for stress measurement can result in systematic errors in the measurement of residual stresses in these materials as large as 80%.

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