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STRESS ELASTIC CONSTANTS

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THE USE OF X-RAYS AND NEUTRONS TO MEASURE (RESIDUAL) STRESS ELASTIC CONSTANTS

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I. Introduction

Diffraction is perhaps the most widely used methodology for the measurement of stresses in a body. This is because it is inherently nondestructive, readily reproducible, and permits the estimation of errors in a straight-forward manner [1]. This technique does not, however, truly measure stresses : by measuring the change in the interplanar, or "d", spacing of a sample, strains are actually determined. In order to convert to stresses, elasticity theory and conversion factors are required. These conversion factors are called "diffraction elastic constants" (DELK) and are denoted as $S_1(hkl)$ and $S_2/2(hkl)$. In an isotropic medium, these terms have the values

$$S_1 = -\nu/E \quad S_2/2 = (1 + \nu)/E \quad (1)$$

where ν is Poisson's ratio and E is Young's modulus.

Most materials are anisotropic and, for highly anisotropic materials, mechanically determined values can lead to errors in the stress measurement of as much as eighty percent [2]. Different processing methods have also been shown to affect these values [3-6], perhaps due to grain interactions, texture, and local composition.



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Theoretical equations [7-10] utilize the knowledge of single-crystal elastic constants, which may not exist for a sample of particular interest; these equations do not, however, incorporate the influence of the factors noted above. Also, until recently [11] most literature values have not reported the errors involved in the measurements, thus reducing the practical value of these results. For these reasons, it is felt that the DELC should be measured for a given material and machining operations before any residual stress determinations are attempted.

An automated system for the determination of x-ray elastic constants has been programmed to achieve an operator-specified error in either of these constants [11]; this permits one to ascertain the errors involved in the results. The technique of measuring the elastic constants requires the determination of a number of d vs. $\sin^2 \psi$ plots performed at a range of applied loads. This procedure should be independent of the loading device used, so this system was given the additional option to employ a four-point bending device [12], as well as a uniaxial tensile device previously described [13].

The increasing use of neutron diffraction to measure residual stresses [14-16], as well as elastic constants [17], has led to this study. Neutrons, which have smaller linear absorption coefficients, have the capability to penetrate on the order of mm or even cm into a sample, whereas x-rays are confined to a surface region of 2 - 100 microns (see Table 1). This significantly greater depth penetration allows the use of psi angles which are impossible to achieve with x-rays [15]. It also allows one to examine different depths within the sample.

called "probe regions" [14]. Thus by comparing the DELC measured with x-rays and neutrons, a comparison between surface and volumetric sampling is made.

This comparison is of interest because the depth of penetration is important with respect to the stress gradients in any stress field [18,19]. Thus it seems that by sampling vastly different volumes of the sample one is sampling significantly different distributions of the applied stress field. This would, in turn, affect the measured DELC.

II. Theory

The measurement of DELC is performed in a manner similar to that of residual stresses. The sample axes P_i and the laboratory axes L_i are related to each other by the angles ϕ and ψ , such that L_3 is normal to the diffracting planes (figure 1). In what follows, unprimed stresses and strains represent those in the sample and primed values are in the laboratory system. The strain along L_3 is :

$$\begin{aligned} \epsilon_{33}' = (d - d_0)/d_0 = & \epsilon_{11} \cos^2 \phi \sin^2 \psi + \epsilon_{12} \sin 2\phi \sin^2 \psi + \epsilon_{13} \cos \phi \sin 2\psi \\ & + \epsilon_{22} \sin^2 \phi \sin^2 \psi + \epsilon_{23} \sin \phi \sin 2\psi + \epsilon_{33} \cos^2 \psi \end{aligned} \quad (2)$$

where d_0 is the interplanar spacing in the unstressed lattice.

The d vs. $\sin^2 \psi$ plots are then measured at several applied loads. The data for each plot is fit to a linear least-squares line, and the slope (m') and intercept ($d_{\psi=0}$) are obtained. The slope of m' vs. applied load is proportional to $S_2/2$, while the slope of $d_{\psi=0}$ vs. applied load is proportional to S_1 . This should be true regardless of

the linearity of the initial d vs. $\sin^2\psi$ plots [3,20].

III. Experimental

The x-ray experiments were performed at the X-Ray Diffraction Facility of the Materials Science and Engineering Department of Northwestern University. The neutron experiments were made on the 2XD line of the University of Missouri-Columbia Research Reactor Facility (MURR).

The diffraction elastic constants for two peaks, the 211 and 310, of a high-strength, low alloy (HSLA) steel were examined using x-ray and neutron diffraction. The x-ray experiments used both a uniaxial tensile and four-point bending devices, while the neutron experiments examined full cross-section and probe regions under uniaxial tension loading.

A. Sample

The material selected was a HSLA steel obtained from Inland Steel Co. of East Chicago, Indiana. Three identical samples were cut from the slab and then annealed for one hour at 648 K to relieve most of the machining stresses. One of these samples was then etched to a depth of 175 microns, in 25 micron increments. Residual stress measurements performed on the 211 peak using $\text{Cr K}\alpha$ radiation, six psi tilts, and a value for $S_2/2$ of $5.80 \times 10^{-6} \text{ MPa}^{-1}$ ($4.0 \times 10^{-6} \text{ psi}^{-1}$) indicated a stress of roughly $-28.0 \pm 4.2 \text{ MPa}$ ($-4.0 \pm 0.6 \text{ ksi}$) at each depth: the error shown here is due to the counting statistics [21].

The sample was 95.25 mm long x 12.7 mm wide x 2.54 mm thick, which represented the thickest sample which could be accommodated on the x-ray

tensile device. This was three times thicker than all previous DELC samples used on the x-ray unit, but thinner than most samples for the neutron tensile device [17]. All of the x-ray and neutron measurements were performed on one specimen.

B. Operating Conditions

A comparison of the general experimental operating conditions is shown in Table 2. The "pseudo-Voigt" fit is an optimized average of a Gaussian and a Lorentzian function. The Lorentzian function is required to more accurately fit neutron peaks which are highly collimated [22]. More specific operating conditions are presented in Tables 3 and 4.

The data at MURR was collected using a position sensitive detector (PSD) developed especially for use on the 2XD line [23]. This PSD improved the counting statistics and decreased the length of time involved in the neutron experiments. Previous experiments verified the resolution and reproducibility of this detector in residual stress measurements [24].

C. Errors due to Counting Statistics

It has been shown [13] that the variances in the DELC, $V(S_1)$ and $V(S_2/2)$, due to the fact that diffraction is a statistical phenomenon are proportional to the variance in peak location, $V(2\theta)$. The appropriate equations are :

$$V(S_1) = \frac{1}{d_0^2} \cdot \left[\frac{\sum (\sigma_{app} - \bar{\sigma}_{app})^2}{\sum (\sigma_{app} - \bar{\sigma}_{app})^2} \right]^2 \cdot \left(\frac{\pi}{180} \right)^2 \cdot \left(\frac{\lambda \cos \theta}{2 \sin^2 \theta} \right)^2 \cdot \frac{V(2\theta)}{2} \quad (3)$$

$$V(S_2/2) = \frac{1}{d_0^2} \cdot \left[\frac{\sum (\sigma_{app} - \bar{\sigma}_{app})^2}{\sum (\sigma_{app} - \bar{\sigma}_{app})^2} \right]^2 \cdot \left[\frac{\sum (\sin^2 \psi - \bar{\sin^2 \psi})^2}{\sum (\sin^2 \psi - \bar{\sin^2 \psi})^2} \right]^2 \cdot \left(\frac{\pi}{180} \right)^2 \cdot \left(\frac{\lambda \cos \theta}{2 \sin^2 \theta} \right)^2 \cdot \frac{V(2\theta)}{2} \quad (4)$$

Although it is more desirous to work at high angles (150° 2θ), the available range of the neutron diffractometer was only 120 degrees. Equations 3 and 4 above demonstrate the dependence of the errors on the ψ and load values which are used. Thus, in spite of the lower angles employed at MURR, we were able to maintain statistical errors in the DELC at about ten percent by utilizing larger ψ and load ranges in the neutron experiments (as shown in Table 2).

D. Probe Region

By placing slits on the incident and diffracted neutron beams, a volume called the "probe region" is defined (figure 2). This probe region allows for the determination of strains at varying depths in a sample [14]; this permits the strain/stress profile to be determined. In a similar manner, the DELC were measured at a single depth at the center of the specimen.

The 310 peak (90° two theta) was examined using this probe procedure. The fact that the sample was only 2.54 mm thick imposed a maximum slit width of 1 mm on these experiments. This was necessary to insure that the probe region was entirely within the sample; if this were not the case, large inaccuracies in peak positioning could occur, particularly if the probe region is inside the specimen at some ψ tilts and not in others [14].

IV. Results

An example of the results for one set of runs is shown in Table 5. The table shows the results of the series of ten replicate measurements

of S_1 and $S_2/2$, along with the statistical and instrumental errors of each run. These errors were determined as per equations found in [1]. The standard deviation of the replicate runs were roughly the same as the experimental errors involved in each individual run.

A summary of all of the results from these experiments is shown in Table 6. The top half of this table contains other values for these elastic constants, some of which are from theoretical calculations, in order to demonstrate the range of values which exist in the literature [25]. As can be seen, the results for the DELC using x-rays and neutrons were the same, within experimental error. There was no difference found in the x-ray results due to different loading devices.

The only discrepancy in these results is found in the S_1 values for the probe region. This was due primarily to accidental sample mispositioning which occurred during the first measurement. In this first run, the sample was moved so that part of the probe region was no longer within the sample. This caused a peak shift which was smaller than it should have been, resulting in a smaller S_1 value. The value of $S_2/2$ for this run was also smaller than that for the second probe measurement, but the overall effect was not nearly as considerable as for S_1 .

From this study, one can conclude that, for a sample without a large stress gradient, the diffraction elastic constants should not be dependent upon the radiation source which is used to measure them. Nor are they dependent upon whether uniaxial tension or four-point bending is used. This is, of course, just a beginning; future experiments must

address the effects of a large stress gradient in the sample. This is necessary not merely as a logical next step, but because it is also a more realistic representation of typical residual stress samples.

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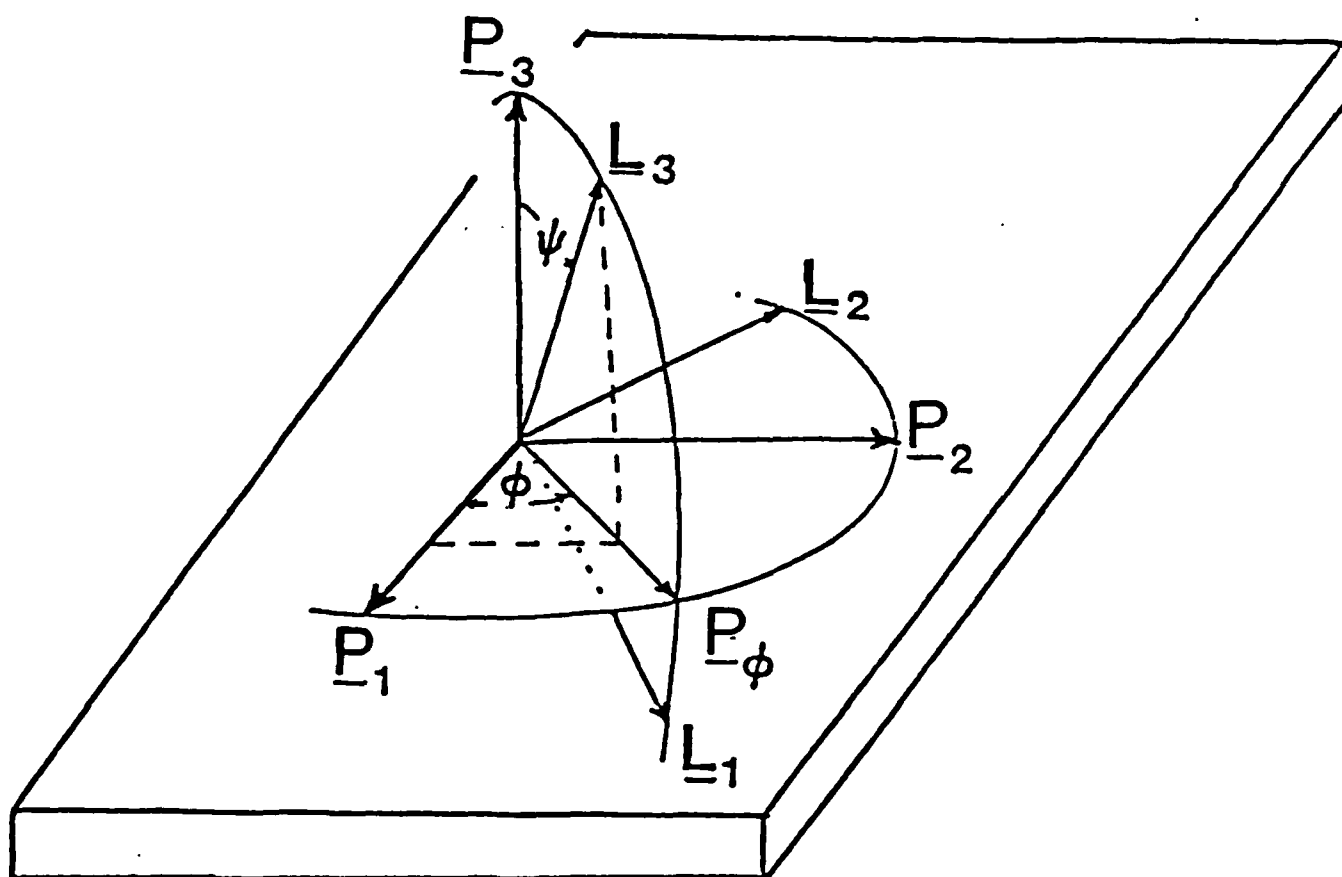


Figure 1 : The axial convention used in these measurements.
 P_1 , L_1 are the sample and laboratory axes, respectively.
 and are related to each other by ϕ and ψ . The planes
 which are used are normal to the L_3 direction.

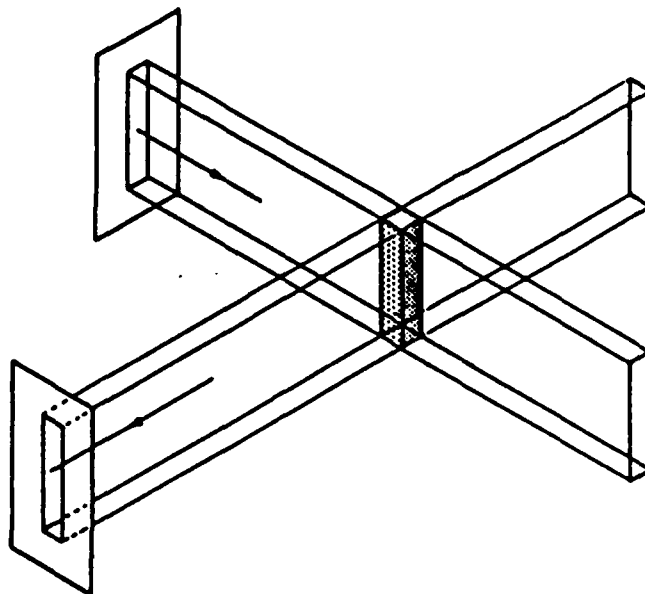


Figure 2 : A probe region (shaded area) is created by placing slits on both the incident and diffracted neutron beams. From (14).

TABLE 1 : NEUTRON and X-RAY SCATTERING CHARACTERISTICS

ELEMENT	NEUTRONS*			X-RAYS**		
	b (10^{-12} cm)	μ (cm^{-1})	50% (cm)	f (10^{-12} cm)	μ (cm^{-1})	50% (cm)
Al	0.39	0.10	7.05	5.69	131	0.53×10^{-2}
Ti	-0.34	0.45	1.55	9.12	938	0.74×10^{-3}
Fe	0.96	1.12	0.62	11.50	2424	0.29×10^{-3}
Ni	1.03	1.86	0.37	12.90	407	0.17×10^{-2}
W	0.47	1.05	0.66	42.30	3311	0.21×10^{-3}

* = $1.29 \text{ \AA}$

** = $1.54 \text{ \AA}$, Cu K x-rays; f values at $(\sin\theta)/\lambda = 0.5 \text{ \AA}^{-1}$

"b" and "f" are the atomic scattering factors for neutrons and x-rays, respectively. μ is the linear absorption coefficient, and "50%" represents the thickness of the material which absorbs 50% of the incident beam intensity.

From : [14]

TABLE 2 : GENERAL CHARACTERISTICS OF THE EXPERIMENTS

	<u>X-RAYS</u>	<u>NEUTRON</u>
TWO THETA	HIGH (150-160)	LOW (67-91)
PSI RANGE	0 - 45	0 - 90
LOAD RANGE	17 - 69 MPa	27 - 207 MPa
NO. OF RUNS	10 REPLICATES	4 REPLICATES
PEAK FIT	7 POINT PARABOLAE	PSEUDO-VOIGT
FIT INTERVAL	TOP 15 %	WHOLE PEAK
INTENSITY	$3 - 8 \times 10^3$ CPS	$2 - 5 \times 10^3$ CTS *
AVG. ERROR $V(2\theta)$		
211	0.0019°	0.0017°
310	0.0023°	0.0028°

* For 200,000 monitor counts (about 22 minutes).

Four loads and six psi tilts were used in all experiments.

TABLE 3 :

X-RAY OPERATING CONDITIONS *

PEAK	211	310
RADIATION	Cr	Co
ANGLE	155.5°	161.6°
TUBE VOLTAGE (V)	38	38
TUBE CURRENT (mA)	12	18
DIVERGENT SLIT	1°	1°
SIZE OF BEAM	0.15 x 0.15 in	0.15 x 0.15 in
SCATTERING SLIT	1°	1°
RECEIVING SLIT	0.11°	0.15°
FILTER	NONE	NONE
APPROX. INT (PEAK)	4000 cps	8000 cps
COUNTING TIME (APPROX)	70 secs/pt	110 secs/pt
ERROR IN PEAK LOCATION	0.0016-0.0022°	0.0018-0.0028°
BACKGROUND SUBTRACTION	YES	YES
PEAKSHIFT CORRECTION	NO	NO
SAMPLE OSCILLATION	NO	NO

* All runs were performed with psi values of 0, 18.43, 26.57, 33.21, 39.23, and 45.0 degrees. Loads were 17.2, 34.5, 51.7, and 69.0 MPa.

TABLE 4 :

NEUTRON OPERATING CONDITIONS *

PEAK	211	310
ANGLE	67.5°	90.5°
APPROX PEAK INT	4500 CTS	2800 CTS
ERROR IN PEAK LOCATION	0.0017° 2θ	0.0028° 2θ
PSI VALUES	0, -5, -10, 60, 69, 90	0, 13, 18, 63, 72, 90

* Wavelength of 1.29042 Å used. These parameters apply to the full cross-section experiments. Divergent slit of 12.7 x 12.7 mm (0.5 x 0.5 in); monitor counts of 200000/peak were used in all cases. Loads were 27.6, 69.0, 137.9, and 206.9 MPa.

TABLE 5 : X-RAY TENSILE DEVICE 211 PEAK (UNITS OF 10^{-6} MPa $^{-1}$)

RUN	S ₁	STAT ERR	INSTR ERR	TOT ERR
1	-1.2487	0.0873	0.1189	0.1475
2	-1.3040	0.0813	0.1182	0.1434
3	-1.3640	0.0783	0.1137	0.1380
4	-1.3713	0.0908	0.1190	0.1496
5	-1.3241	0.0783	0.1154	0.1395
6	-1.1773	0.0856	0.1164	0.1444
7	-1.2105	0.0864	0.1219	0.1495
8	-1.2949	0.1060	0.1206	0.1607
9	-1.2564	0.0821	0.1083	0.1359
10	-1.5530	0.1059	0.1189	0.1592
MEAN	-1.3104	0.0882	0.1171	0.1468
STD DEV	0.1056			

RUN	S ₂ /2	STAT ERR	INST ERR	TOT ERR
1	6.0030	0.2297	0.2842	0.3654
2	6.0770	0.1989	0.2825	0.3455
3	5.0779	0.1944	0.2717	0.3341
4	6.0059	0.2042	0.2845	0.3502
5	5.2026	0.1943	0.2835	0.3437
6	5.2853	0.2037	0.2820	0.3479
7	6.0668	0.1989	0.2916	0.3529
8	5.5999	0.2323	0.2823	0.3655
9	5.3752	0.1926	0.2588	0.3226
10	6.1074	0.2567	0.2843	0.3831
MEAN	5.6800	0.2106	0.2805	0.3511
STD DEV	0.4144			

TABLE 6 : STRESS ELASTIC CONSTANTS VIA VARIOUS TECHNIQUES (10^{-6} MPa $^{-1}$)

	$-\gamma/E$	$^{211} \frac{(1 + \gamma)}{E}$	PEAKS $-\gamma/E$	$^{310} \frac{(1 + \gamma)}{E}$
BULK MECHANICAL MEASUREMENT	-1.36	6.22	-1.36	6.22
X-RAY EXP CALIB	-1.48	6.35	-1.84	7.48
VOIGT (CONSTANT STRAIN)	-1.23	5.63	-1.23	5.63
REUSS (CONSTANT STRESS)	-1.31	5.83	-2.28	8.76
NEERFELD (AVERAGE OF VOIGT AND REUSS)	-1.28	5.73	-1.75	7.19

THESE STUDIES :

X-RAY TENSILE *	-1.31 (0.11)	5.68 (0.41)	-2.22 (0.21)	8.01 (0.47)
X-RAY BENDING *	-1.14 (0.10)	4.97 (0.38)	-1.83 (0.14)	7.06 (0.69)
NEUTRON TENSILE **	-1.56 (0.50)	5.88 (0.57)	-1.78 (0.35)	7.24 (0.14)
NEUTRON PROBE ***			-0.94	7.73

* 10 REPLICATE MEASUREMENTS

** 4 REPLICATE MEASUREMENTS

*** 2 REPLICATE MEASUREMENTS

The top half of this table shows literature values from reference [25], while the bottom half demonstrates the results of these experiments. The values in parentheses are the standard deviations in the replicate measurements.

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