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THE MEASUREMENT OF ELASTIC CONSTANTS FOR THE DETERMINATION OF STRESSES BY X-RAYS

BY

K. Perry, I. C. Noyan, P. J. Rudnik and J. B. Cohen

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THE MEASUREMENT OF ELASTIC CONSTANTS FOR THE DETERMINATION OF STRESSES BY X-RAYS

K.Perry, I.C.Noyan, P.J.Rudnik, and J.B.Cohen

Department of Materials Science, The Technological Institute, Northwestern University, Evanston, Illinois,60201

INTRODUCTION

Residual and applied stresses (σ_{ij}) are often measured via X-ray diffraction, by calculating the resultant elastic strains (ϵ_{ij}) from the measured change in interplanar spacing ("d"). This method is non-destructive, reasonably reproducible (typically ±14MPa), can be carried out in the field[1], and is readily automated to give values to an operator-specified precision[2]. Let L_i represent the axes of the measuring system with L_3 normal to the diffracting planes, and P_i represent the sample axes. These axes are illustrated in Figure 1. In what follows, primed stresses and strains are in the laboratory system, while unprimed values are in the sample system. The strains in the direction L_3 are referenced with the angles ϕ and ψ in Figure 1, and can be written in terms of the stresses in the sample[3]:

$$(\epsilon'_{33})_{\phi\psi} = \frac{d_{\phi\psi}-d_o}{d_o} = \tfrac{1}{2}S_2\{\sigma_{11}\cos^2\phi+\sigma_{12}\sin2\phi+\sigma_{22}\sin^2\phi-\sigma_{33}\}\sin^2\psi + \tfrac{1}{2}S_2\sigma_{33}-S_1(\sigma_{11}+\sigma_{22}+\sigma_{33})+\tfrac{1}{2}S_2(\sigma_{13}\cos\phi+\sigma_{23}\sin\phi)\sin2\psi \qquad (1)$$

Here d_o is the d-spacing in a stress-free material, S_1 and $\frac{1}{2}S_2$ are the so-called X-ray elastic constants and the first term in parentheses on the right hand side of Equation 1 will be called

$\sigma_{\phi\psi} - \sigma_{33}$.

For an isotropic material the X-ray elastic constants can be written in terms of Poisson's ratio (ν) and Young's modulus (E):

$$S_1 = -\nu/E \quad (2a)$$

$$\tfrac{1}{2}S_2 = (1+\nu)/E \quad (2b)$$

Other estimates for S_1 and $\frac{1}{2}S_2$, such as those by Neerfield[6] and Kroner[7], are also available. For an anisotropic material these values depend on texture and method of processing and must be uniquely measured.

The normal components σ_{33}, σ_{13}, and σ_{23} are zero at the surface, but the X-ray beam penetrates a sufficient depth so that their contribution can be detected[4,5]. Their presence leads to curvature in $d_{\phi\psi}$ vs. $\sin^2\psi$, which for the shear terms is opposite in sense for $+\psi$ and $-\psi$. The presence of texture and/or the variation in stress from point to point under the X-ray beam can lead to large oscillations in this relationship[8,9]. If both effects are absent, $d_{\phi\psi}$ vs. $\sin^2\psi$ is linear and from the slope $\sigma_{\phi\psi}$ is obtained. This is the common practice, and in such a case measurements at only one ϕ and two ψ tilts are sometimes employed. However, the absence of these effects must be verified before such a simple procedure is applied. Other procedures are available for more complex situations[5,9]. In any case, the measured X-ray elastic constants are required.

The simplest way to measure the X-ray elastic constants is to apply a uniaxial elastic load, say σ_{11}^{APP}, to a sample of the same material under the same conditions as the piece for which the strains to be measured will be used. The total stress is then $\sigma^{APP} + \sigma^{RES}$ and :

$$\sigma^{RES} + \sigma^{APP} = \left[\frac{\partial(\epsilon'_{33})_{\phi\psi}}{\partial \sin^2\psi}\right] \Big/ (S_2/2) \quad (3)$$

When $\phi = 0$: $\sigma^{RES} = \sigma_{11}^{RES} - \sigma_{33}^{RES}$

$$\sigma^{RES} + \sigma^{APP} = \frac{\partial(\epsilon'_{33})_{\phi\psi}}{\partial \sin^2\psi} / (\tfrac{1}{2}S_2)$$

$$= \frac{\partial d_{\phi\psi}}{\partial \sin^2\psi} / d_o \cdot (\tfrac{1}{2}S_2) \equiv m'/d_o \cdot (\tfrac{1}{2}S_2) \quad (4)$$

Thus $\frac{1}{2}S_2$ can be obtained from the slopes (m'') of several plots of "d" vs. $\sin^2\psi$ at different σ_{11}^{APP}:

$$\tfrac{1}{2}S_2 = m''/d_o \quad (5a)$$

where:

$$m'' = \frac{\partial m'}{\partial \sigma_{11}^{APP}} \quad (5b)$$

Similiarly :

$$S_1 = m'''/d_o \quad (6a)$$

where :

$$m''' = \frac{\partial d_{\phi,\psi} = 0}{\partial \sigma_{11}^{APP}} \quad (6b)$$

Errors in the results result from both counting statistics and geometric errors. Consider first the statistical errors. James and Cohen[2] have derived an equation for the variance (V) of m' (which is in terms of the variance of the peak location 2θ).

Assume that one has a straight line: $m' = m''\sigma_{11}^{APP} + b$. Then using the equation[10]:

$$m'' = \frac{\sum_i (\sigma_i^{APP} - \overline{\sigma^{APP}})(m_i' - \overline{m}')}{\sum_i (\sigma_i^{APP} - \overline{\sigma^{APP}})^2} \quad (7)$$

If $X = f(X_1, X_2, X_3, \ldots)$, V is given by[10] :

$$V(X) = \left(\frac{dX}{dx_1}\right)^2 V(x_1) + \left(\frac{dX}{dx_2}\right)^2 V(x_2) + \cdots \quad (8)$$

Applying this to Equation (7) :

$$V(m'') = \frac{\sum_i \left(\sigma_i^{APP} - \overline{\sigma^{APP}}\right)^2 V(m')}{\left[\sum_i \left(\sigma_i^{APP} - \overline{\sigma^{APP}}\right)^2\right]^2} \quad (9)$$

Therefore, combining this with Equation (5a) yields :

$$V(\tfrac{1}{2}S_2) = V(m'')/d_o^2 \quad (10)$$

Following the same procedure for S_1 :

$$V(S_1) = V(m''')/d_o^2 \quad (11)$$

The principal instrumental errors are those due to sample displacement, ψ axis missetting, and horizontal X-ray beam divergence[11]. Formulae for the variance in 2θ due to these effects can be found in this reference, and the error propagated into S_1 and $\frac{1}{2}S_2$ using the above equations. The two variances can then be added. (It can be shown that for S_1, the instrumental factors for the stationary slit method are zero).

To apply these equations requires a nearly linear "d" vs. $\sin^2\psi$ plot. It is unclear from a survey of the literature on X-ray elastic constants[12,13] that this has always been the case. Also, errors have usually been estimated after repeating the measurement only once. Proper evaluation of the errors by the methods described here has never been done. There are reports of large effects of plastic deformation on the elastic constants[14,15]. These may be valid, or could arise from large curvature or oscillations in "d" vs. $\sin^2\psi$. There are also reports of different stresses obtained from different peaks[15]. A new systematic determination of constants for the various reflections of practical interest is sorely needed. In this paper we describe an automated system for this purpose, by which the constants can be obtained to an operator specified precision.

The paucity of carefully determined X-ray elastic constants is not surprising. If six different ψ values and five stress levels are employed, the thirty measurements can take 18 to 24 hours with a normal detector. Automation is needed; also the use of a position sensitive detector can reduce the time by an order of magnitude[16].

HARDWARE

Our miniature tensile device is shown in Figure 2, mounted on a diffractometer. The specimen (A) is held in place by two grips (B), which have been precisely machined to minimize bending. One of the grips is attached to a gear assembly (C) to which a high torque Slo-Syn stepping motor is attached (D). There are 200 steps per revolution and movement is directed by a Motorola 6080 type microprocessor so that the specimen can be loaded and unloaded automatically.

The other grip is attached to a load cell (E), Model 41, manufactured by Sensotec Inc. of Columbus, Ohio. The load cell is bolted to a 0.5 inch thick circular metal plate which is attached to the body of the load cell. The force on the sample is transmitted through the grip via a threaded screw which runs through the center of the cell. The Model 41 senses the deflection between the outer rim bolt holes and a threaded inner hub. The cell used was designed for loads up to 5000 pounds.

The output of this cell is read with a 450-D Single Channel Amplifier, also manufactured by Sensotec, which provides a signal conditioner and digital indicator. The mechanical strain on the sample can be measured by either cementing a thin foil strain gauge to the back of the sample, or attaching a clip-on extensometer. This strain is read by a Model 4412 Voltmeter manufactured by Data Technology Corp. The output of both the 450-D and the 4412 were modified so that they could be interfaced with the microprocessor.

The tensile device is mounted onto a sample holder (F), designed so that the tensile device can be moved horizontally, vertically, and rotated normal to the specimen surface. This holder is mounted onto a track (G) and can be moved along the track by means of an attached micrometer (H), allowing for accurate specimen positioning. All 2θ and ψ movements were made by the Slo-Syn motors, via computer control, while the counting was recorded by the microcomputer.

SOFTWARE

The computer package is written in XYBASIC, a computer language copyrighted by the Mark Williams Chemical Company of Chicago, Illinois and designed especially for process control, data acquisition, and real time applications with 8080-based computers. Our package for elastic constant determination contains the following features :

a. A separate alignment program for determination of sample displacement. (This is determined from the slope of the lattice parameter a_o vs. the Nelson-Riley function for three or more peaks).
b. On-line peak location using a least-squares parabolic fit to the top of a peak.
c. Determination of elastic constants to an operator specified accuracy, or using a preset number of counts.

d. Operator specification of stress values to be used in measurement.
e. Operator choice of psi tilts to be used in measurement.
f. Operator choice of number of data points to be used for parabolic fit to a peak.
g. Option of scattering factor correction.
h. Operator choice of preliminary scan steps and counts.
i. Optional background subtraction.
j. Optional sample oscillation.
k. Optional peakshift correction. (This is due to the effect of K_{α_2} on the K_{α_1} position, which varies with the peak shape[5]).
l. Calculation of statistical error with the optional calculation of geometric error, due to divergence and effects of sample and/or psi axis displacement.
m. Calculation of Young's modulus using an attached mechanical strain gauge, and the corresponding stress-strain plot.
n. Plots of d vs. $\sin^2\psi$ for all stress values; also, plots for m' vs. stress and for $d_{\psi=0}$ vs. stress.
o. Use of any detector.
p. Storage of data on a separate flexible disk for use with a separate data manipulation program, if changes in various terms are desired.

A multiple scan procedure is employed for peak location and to make an estimate of the time required to achieve a desired precision. This is described in reference 2. It is accomplished by multiplying the time needed for a single peak by the number of ψ and σ^{app} values to be employed. This allows the operator an opportunity to choose a larger error if the time is excessive. A sample dialogue with the operator is shown in Figure 3. Tests of the device are described below.

EXPERIMENTAL DETAILS

The materials examined and their preparation are described in Table I. Flat tensile specimens were cut to dimensions of 2.75 inches long by 0.4 inch wide and had reduced sections which were 1.75 inches long by 0.25 inch wide. Typical operation conditions are given in Table II. It is to be emphasized that oscillations of the sample on the diffractometer can considerably reduce oscillations in d vs. $\sin^2\psi$. Although it was not done here, it is also sometimes helpful to shot peen or grit blast a sample. This minimizes texture in the surface and can also reduce oscillations.

RESULTS

Replicate measurements with nickel are given in Table III. The columns labelled "stat" give errors which are estimated from Equations 10 and 11. It can be seen that these are somewhat less than the actual variation. A similar set of data for a brass sample with a preset error of about 20 percent of the S_1 value (rather than the 5 percent error used with the nickel specimen) gave good agreement with the calculated error. Therefore, unless the error is set very low, Equation 10 does give an estimate of the error in $\frac{1}{2}S_2$ with only a single measurement. Errors in S_1 are often larger than the statistical estimates. This is probably due to the fact that any oscillations or curvature in d vs. $\sin^2\psi$ violates the initial assumption of linearity.

An attempt was made to see if any other factors might affect the results. A dial gauge placed on the sample indicated that some displacement occurred during and after loading. For the most part, the displacement was 2×10^{-3} inch or less. Occasionally displacements as large as 5×10^{-3} inch were found. Calculations indicated that the largest change in $\frac{1}{2}S_2$ due to this effect would be 3 percent. The constant S_1 is unaffected by this when the stationary slit method is used.

Some stress relaxation occurred during measurements at a given load. For aluminum, this could change $\frac{1}{2}S_2$ by as much as 6 percent for a 400 reflection, and 1.5 percent for the 422. For softer materials the change was much less (0.1 percent for nickel).

A comparision of the nickel results with other data is given in Table IV. Results for other materials tested are shown in Tables V and VI. Included are some h00 and hhh reflections; ignoring grain interaction stresses, theory indicates that oscillations in d vs. $\sin^2\psi$ due to texture should be eliminated. In practice, this is not always the case. For α-brass and nickel, there was some reduction in oscillations for the peaks shown in Table V. In both cases the hhh reflection is at the same or higher

2θ value as the hkl reflection; thus any oscillations should be equally clear since the peakshift $\Delta 2\theta$ is proportional to tan θ.

If the oscillations are not large, two ψ tilts are sufficient. Recalculating the elastic constants in Table III for $\psi = 0°$ and $45°$ changes $\frac{1}{2}S_2$ by only 3 percent.

In summary, software and hardware for the fully automated determination of X-ray elastic constants have been demonstrated with several materials. Equations have been developed and tested to allow estimates of the error in these constants without repeating the measurement, regardless of whether or not automation was used. It is hoped that future reports on these constants will include such error estimates.

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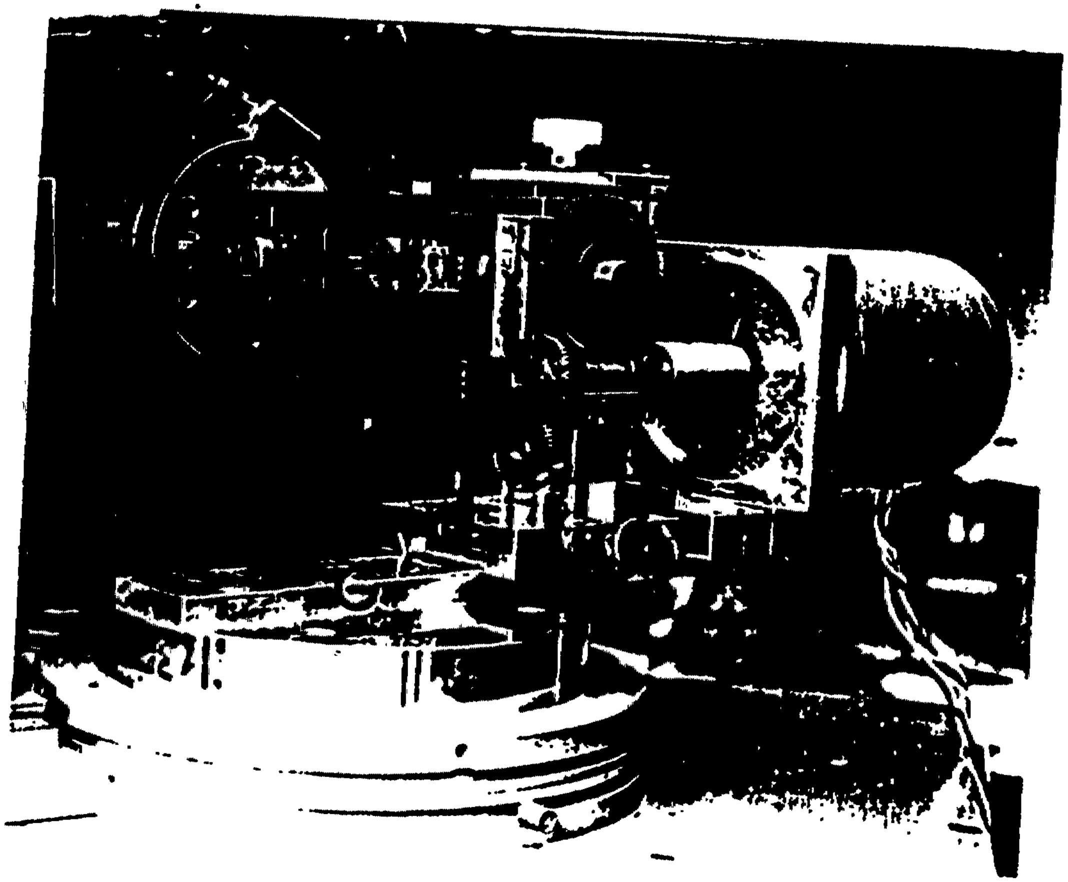

Fig. 2: Tensile device mounted on X-ray unit for measurement of elastic constants. A - tensile specimen; B - grips; C - gears; D - stepping motor; E - load cell; F - device holder; G - track; H - micrometer adjustment.

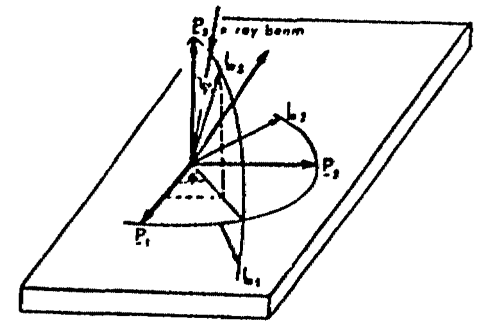

Fig. 1: P_i: Sample Axial System.
L_i: Measuring System.

```
RUN
EXPERIMENTAL DETERMINATION OF X-RAY ELASTIC CONSTANTS
NAME? KATHLEEN
DATE AND TIME STARTED? 2/8/1230AM
SAMPLE? NICKEL 313  RUN 15
TUBE TARGET AND SERIAL? CU
NAME OF DATA FILE? NIC313.015
USING TENSILE DEVICE (1) OR BENDING DEVICE (2)?? 1
USING SOLID STATE DETECTOR(1) OR NO(2)? 2
STANDARD LIMITS OF 2THETA(1) OR NO(2)? 1
2THETA MAX... 150    2THETA MIN... 0
STANDARD LIMITS OF PSI(1) OR NO(2)? 1
PSI MAX... 120    PSI MIN...-10
SPECIMEN CROSS-SECTIONAL AREA (IN SQUARE INCHES)? .008
LOAD CELL LIMIT OF 4000 LBS.(1) OR NO(2)? 1
MAXIMUM LOAD IS 500000 PSI
USE OF MECHANICAL STRAIN GAUGE(1) OR NO(2)? 2
CURRENT SETTING OF 2THETA? 144.42
CURRENT SETTING OF PSI? 105.42
RADIUS OF GONIOMETER? 8.125
WAVELENGTH?? 1.5405
NUMBER OF LOAD READINGS?? 5
LOAD (IN PSI)? 0
LOAD (IN PSI)? 4000
LOAD (IN PSI)? 8000
LOAD (IN PSI)? 12000
LOAD (IN PSI)? 16000
WISH TO USE STANDARD PSI TILTS(1) OR NO(2)? 1
PSI TILT       SIN(PSI)^2
 0              0
 18.43          .1
 26.57          .2
 33.21          .3
 39.23          .4
 45             .5
NUMBER OF DATA POINTS TO BE USED IN FINAL SCAN?? 7
SCATTERING FACTOR CORRECTION(1) OR NO(2)? 2
 PRELIMINARY SCAN DATA
APPROXIMATE 2THETA PEAK?? 144.34
INITIAL 2THETA PEAK?? 144
FIRST INCREMENT?? .1
PRESET COUNTS FOR FIRST SCAN? 1000
SECOND INCREMENT?? .02
PRESET COUNTS FOR SECOND SCAN? 2000
BACKGROUND FEATURE(1) OR NO(2)? 1
2THETA WHERE BACKGROUND IS DETERMINED?? 141
OSCILLATE FEATURE(1) OR NO(2)? 1
ROCKING WIDTH(DEGREES 2THETA)?? 1
PEAKSHIFT CORRECTION FEATURE(1) OR NO(2)? 2
INSTRUMENTAL ERROR(1) OR NO(2)? 1
DIVERGENT SLIT? 1
SAMPLE DISPLACEMENT? 2E-4
PSI-AXIS MISSETTING? 0
PRESET COUNTS(1) OR PRESET ERROR(2)? 1
PRESET NUMBER OF COUNTS? 15000
*****  CHECK LIMIT SWITCHES AND SHUTTER  *****
DEVICE SHOULD BE IN HORIZONTAL POSITION!!!
ARE YOU READY TO BEGIN MEASUREMENT(1) OR NO(2)? 1
```

Fig. 3: Dialogue for elastic constant determination program.

TABLE I

SAMPLE PREPARATION

Specimen	Starting Thickness	Treatment	Final Thickness
1100 Al	.45"	cold rolled to a 90% reduction	.045"
70-30 α-brass	.247"	cold rolled to a 90% reduction	.024"
304 stainless steel	.059"	cold rolled as received	.059"
1075 steel	.032"	cold rolled as received	.032"
Ni	.031"	cold rolled as received	.031"

TABLE II

OPERATING CONDITIONS

Beam Size on Sample	.15" x .45"	
Divergent Slit	1°	
Receiving Slit	.18°	
Tube Voltage	Cu - 40 kV	Fe - 40 kV
Tube Current	Cu - 20 mA	Fe - 15 mA
Goniometer Radius	8.125"	

Six ψ Tilts
Seven Point Parabolic Fit
2° Oscillation to Reduce Oscillations in d vs $\sin^2\psi$
10,000 - 15,000 Cts/Point
Background Subtraction
No Scattering Factor Correction
No Peak Shift Correction

TABLE III

RESULTS OF 10 REPLICATE MEASUREMENTS OF ELASTIC CONSTANTS USING THE 313 REFLECTION OF NICKEL

Run #	XREC* $S_2/2$	Stat.	Error Instr.	Total	XREC* S_1	Stat.
1	4.740	.216	.028	.218	-.757	.102
2	3.655	.227	.030	.229	-.411	.064
3	4.116	.195	.029	.197	-.739	.059
4	4.004	.221	.030	.223	-.587	.069
5	4.000	.197	.029	.199	-.606	.063
6	4.210	.210	.029	.212	-.742	.062
7	4.128	.199	.029	.201	-.776	.063
8	3.593	.185	.028	.187	-.635	.050
9	3.763	.211	.029	.213	-.518	.082
10	4.330	.187	.029	.189	-.611	.057
Mean	4.054	.204	.029	.206	-.638	.067
St. Dev.	.340				.117	

*Units of 10^{-8} psi^{-1}.

TABLE IV

ELASTIC CONSTANTS OF Ni: 313 REFLECTION: IN 10^{-8} PSI^{-1}

Method	$S_2/2$	S_1
This work	4.05 ± .34	-.64 ± .11
Mechanical measurement**	4.49	-1.06
X-Ray Experimental Calibration*	3.83 ± .14	-.83 ± .04
Voight (Constant Strain)*	3.81	-.84
Reuss (Constant Stress)*	3.66	-.79
Neerfield (Average of Voight and Reuss)*	3.73	-.82
Kroner***	3.58	-.77
Calculated from Handbook****	4.37	-1.03

* Reference 13
** E. Macherauch Experimental Mechanics 6 (1966) pp. 140-153.
*** Calculated from single crystal data.
****Metals Handbook ASM, Metals Park, Ohio.

TABLE V

EXPERIMENTAL AND THEORETICALLY CALCULATED VALUES OF $S_2/2$ IN 10^{-8} PSI^{-1}

Material	λ	hkl	$S_2/2$	Total Error	Voight	Reuss	Neerfield(6)	Kröner(7)
Al	Cu	422	12.19	.27	13.13	12.84	12.99	13.01
	Fe	400	10.49	.28	13.13	14.97	14.05	13.96
			11.28	.25				
α-Brass	Cu	331	6.94	1.22	6.85	7.23	7.04	6.98
	Fe	222	4.22	.82	6.85	4.83	5.84	6.14
			4.36	.83				
304 stainless steel	Cu	331	4.48	.20	4.01	3.82	3.92	3.93
	Fe	222	3.75	.35	4.01	3.09	3.55	3.63
			3.51	.38				
1075 steel	Fe	220	4.17	.17	4.01	4.12	4.07	4.06
	Cu	222	3.05	.24	4.01	3.09	3.55	3.63
			2.41	.25				
Ni	Fe	311	4.04	.35	3.64	4.98	4.31	4.19
	Fe	222	3.12	.25	3.64	2.76	3.20	3.28
			3.57	.24				

TABLE VI

EXPERIMENTAL AND THEORETICALLY CALCULATED VALUES OF S_1 IN 10^{-8} PSI^{-1}

Material	λ	hkl	S_1	Total Error	Voight	Reuss	Neerfield	Kröner
Al	Cu	422	-3.81	.08	-3.38	-3.29	-3.34	-3.33
	Fe	400	-3.03	.11	-3.39	-4.00	-3.70	-3.67
			-3.20	.09				
α-Brass	Cu	331	-1.31	.39	-1.64	-1.77	-1.71	-1.69
304 stainless steel	Cu	331	-.94	.05	-.81	-.74	-.78	-.78
1075 steel	Fe	220	-1.05	.06	-.81	-.85	-.83	-.83
	Cu	222	-.77	.06	-.81	-.50	-.66	-.68
			-.76	.06				
Ni	Fe	311	-.61	.10	-.78	-1.23	-1.01	-.97
	Fe	222	-.28	.08	-.79	-.50	-.73	-.67
			-.21	.08				

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The equations for errors in the measurement of x-ray elastic constants are derived. An automated device for measuring these has been developed and tested.

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