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X-RAY MEASUREMENTS OF STRESSES AND DEFECTS IN EFG AND LARGE GRAINED POLYCRYSTALLINE SILICON RIBBONS

First Quarterly Report

By
C. N. J. Wagner

January 1978

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University of California
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SUMMARY

The first model of a modified Bond goniometer has been built and tested for the precision measurement of interplanar spacings in Si-single crystals. A change in interplanar spacing $\Delta d/d \simeq \pm 10^{-5}$ can be detected which corresponds to surface stresses of the order of ±1000 psi. A second version of the goniometer is being assembled incorporating a removable microscope for precision alignment of the Si-strip into the primary X-ray beam.

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I. Introduction

Residual stresses are produced in EFG Si ribbons as a consequence of the growth and cooling process. Such stresses might be responsible for cracking of the ribbons during handling, processing and service of the solar cells. Thus, the knowledge of the sign, magnitude and distribution of these stresses would be valuable in ascertaining the severity and causes of the problem.

To this effect a modified Bond goniometer has been built for the precision measurements of interplanar spacing d in Si-single crystals. This goniometer is capable of measuring $\Delta d/d \sim 10^{-5}$. We will briefly describe the principle and the construction of the goniometer. Then a brief outline is given about the results of the initial work on the determination of the residual stresses.

II. Technical Discussion

A. Experimental Techniques

A goniometer was constructed using a PIC 360:1 worm and worm gear with a precision of 0.002° over the 360° of rotation. On this gear of the goniometer, a GE goniostat was mounted precisely over the axis of rotation of the goniometer whose angle could be read in steps of 0.01° degree , as shown in Fig. 1. The goniostat permits rotations about two mutually perpendicular axes at the precise location where the X-ray beam hits the single crystal. The X-ray beam emanating from a Jarrell-Ash microfocus unit was collimated with a 0.05 x 0.3 or 0.3 x 0.3mm^2 slit located 200 mm from the X-ray target, yielding an irradiated area of 0.1 x 0.4 or 0.4 x 0.4 mm^2 on the sample.

The diffracted X-ray beam was measured at high angles on either side of the primary beam as shown in Fig. 1. To this effect, two special holders were constructed for a large area proportional counter (Reuter-Stokes, 2 at. Xe, 25 mm diameter with a 25 mm area Be window) which could be alternately moved from one holder to the other. The detector holders could also be moved on a limited arc about 200 mm from the center of the goniometer.

This system was tested with a thin slice of a Si single crystal cut with a (110) plane parallel to the surface. First, a (440) reflection was measured with a Co-Kα, radiation ($\lambda K\alpha_1$ = 1.78892 Å). In the Bond technique, the angular position $2\omega_1$, of the peak maximum is read off the odometer of the goniometer, then the detector is moved to its second position, and the crystal is rotated until the maximum is again observed at the angular position $2\omega_2$. The difference in angle $2\omega_1$-$2\omega_2$ corresponds to 180-2θ, i.e.,

$$2\omega_2 - 2\omega_1 = 180° - 2\theta \tag{1}$$

In order to locate the peak position more accurately, the intensity was measured at different values of 2ω about $2\omega_1$ and $2\omega_2$ in steps of 0.01°, and then plotted as a function of 2ω. The peak maximum was determined by measuring the mid-point of the cords at different heights of the peak and extrapolating the loci of these mid-points to the top of the peak. In this way, the peak maximum could be determined to within ±0.001°. Thus the accuracy of the angle 2θ is ±0.002°.

It is obvious from Fig. 1, that the reciprocal vector $\vec{H}$ must be parallel to the diffraction vector $(\vec{S}_1-\vec{S}_o)/\lambda$ and $(\vec{S}_2-\vec{S}_o)/\lambda$ which lie in the horizontal plane. The goniostat enables us to rotate the crystal in such a way that $H \,||\, (\vec{S} - \vec{S}_o)/\lambda$. Otherwise, if the reciprocal vector $\vec{H}$ is tilted by the amount δ away from the horizontal plane, the true Bragg angle θ is related to the measured angle θ' by:

$$\sin\theta = \cos\delta \, \sin\theta'$$

As long as the angle of tilt δ were to remain the same in all measurements, no error would be introduced since we are only interested in relative changes in the d-spacing.

In order to minimize δ, a horizontal, removable receiving slit has been installed in front of each detector. The width of the slit was chosen to be 1.2 mm, but can be made smaller. Since the half-width of the crystalline reflections are of the order of 0.15°, we should use a slit with an opening of twice that size, which corresponds to 1 mm (with a crystal to detector distance of 200 mm).

In order to mount the silicon strips, 3 feet long and 3 inches wide, on the goniostat, a special holder had to be made, consisting of a vertical dove-tail slide and a horizontal translation to locate a desired spot on the strip into the center of the goniostat, which could be accurately determined with the help of a microscope. Unfortunately, the position and dimension of the microscope limits the rotation of the Si-strip. Provisions have been made to rotate the microscope out of the alignment position, but still being able to reposition it accurately for repeated measurements. In this way, the single crystal of Si could be mounted precisely in the center of the goniostat positioned accurately in the goniometer axis.

B. Results

By plotting the peak profiles of the (440) reflections measured with Co-radiation, the difference $2(\omega_1-\omega_2)$ was found to be 42.599° yielding a value of 2θ = 137.401° and a lattice constant a = 5.43078 A using λ = 1.78892 A.

Since the Si-strips were neither of (111) nor (110) orientation, it was felt advantageous to look for a crystal orientation lying in the center of the standard stereographic triangle, i.e., a (321) plane. Using CuK_β(λ = 1.39217 A), the (110) Si single crystal was reoriented in such a way that $\vec{H}_{642}$ become parallel to the diffraction vector. The difference in angular position $2(\omega_1-\omega_2)$ was found to be $2(\omega_1-\omega_2)$ = 32.860° yielding 2θ = 147.140° and $\underline{a}$ = 5.43078 A in excellent agreement with the value $\underline{a}$ obtained with Co-Kα radiation.

Having established that the experimental set-up will yield reproducible values of the lattice parameter, the Si-strip attached to an Aℓ-sample holder (3 ft long, 3 in wide) was mounted on the special holder, and its position was adjusted in such a way that the surface of the Si-strip is in the focus of the microscope, which had been rotated back into the observation position.

In order to speedily orient the Si-strip into reflecting position for the (642) planes, i.e., $\vec{H} \,||\, (\vec{S}-\vec{S}_0)/\lambda$, Laue patterns were made along and across the strip using a Mo X-ray tube and Polaroid film. From these patterns, the angles of tilt χ and ϕ for the (321) orientation could be readily determined.

Six precision measurements of the angle 180°-2θ were made across the width of the strip roughly in its center location. The results of the measurements are given in Table 1. As can be seen, the values of 180°-2θ are very close to that observed in the set-up crystal and vary only slightly over the width of the strip indicating that the residual strains are relatively small.

C. Discussion

It is readily seen that

$$\Delta(180° - 2\theta) = -\Delta 2\theta = +\frac{\Delta d}{d}\, 2 \tan\theta \qquad (2)$$

$$\varepsilon = \frac{\Delta d}{d} = \frac{\Delta(180°-2\theta)}{2 \cdot 57.3 \tan\theta} = \frac{\Delta 2\omega° \tan\omega}{114.6} \qquad (3)$$

Assuming an error of ± 0.002° in 2ω, or ± 0.004° in Δ2ω the error in ε is of the order of 1×10^{-5} using a (642) reflection and CuK_β radiation (tanω/114.6 = 0.00257). If we take the value of Young's modulus as 3×10^7 psi, the error in the residual stress determination will be of the order of 300 psi. It follows from Eq. (3) that the precision in the measurement of the strain ε decreases with decreasing Bragg angle θ or increasing ω.

However, the correct expression which relates stress and strain is given by

$$\varepsilon_\psi = \sigma_\chi(S_1 + \tfrac{1}{2}S_2 \sin^2\psi) + \sigma_\phi S_1 \quad (4)$$

where σ_χ and σ_ϕ are the surface stresses, $S_1 = -\nu/E$, $(\frac{1}{2})S_2 = (1+\nu)/E$ and ψ is the angle of tilt of the specimen, i.e., the angle between the normal of the reflecting plane and the normal of the surface of the sample (Cullity, 1956; Fig. 17-6).

For the (642) reflection, the angle $\psi < 6°$ and $\sin^2\psi$ might be neglected. Thus

$$\varepsilon = S_1(\sigma_\chi + \sigma_\phi) = \frac{d - d_o}{d_o} \quad (5)$$

Using the elastic constant of silicon: $C_{11} = 1.66$, $C_{12} = 0.64$ and $C_{44} = 0.79$ (all in 10^{12} dyn/cm^2), and the relation

$$S_1 = S_2 + \Gamma(S_1 - S_{12} - \tfrac{1}{2}S_{44}) \quad (6)$$

where

$$\Gamma = \frac{h^2k^2 + k^2\ell^2 + \ell^2h^2}{(h^2 + k^2 + \ell^2)^2}$$

and $S_{11} = (C_{11} + C_{12})/C_o$; $S_{12} = -C_{12}/C_o$; $S_{44} = 1/C_{44}$; and $C_o = (C_{11} - C_{12}) \times (C_{11} + 2C_{12})$ we obtain a value of $1/S_1 = -1.1 \times 10^8$ psi $= 7.9 \times 10^{11}$ N/m^2. Thus the error in $\sigma_\chi + \sigma_\phi$ would correspond to ±1000 psi.

As shown in Table 1, the mean value of $2\omega = 180°-2\theta°$ for the crystal with orientation I is 32.8525. Thus the variation is 2ω between positions 1, 2 and 3 is within the accuracy of our measurements, i.e., ±0.002°. Again the mean value of 2ω for orientation II is 32.852. However, this time, the variation in 2ω between positions 4, 5 and 6 is ±0.005 which is believed to be outside our error limits of ±0.002° in 2ω. Thus, we can conclude that the maximum variation in stress across the surface is caused by a strain $\Delta\epsilon$ corresponding to $\Delta\omega = 0.01°$ yielding a stress of $\Delta\sigma = 3000$ psi using Eq. (5).

In this initial study it has been shown that the modified Bond technique is capable of detecting changes in the interplanar spacing $\Delta d/d$ of the order of $\pm 10^{-5}$, yielding relative changes in the surface stresses $(\sigma_1 + \sigma_2)$ of the order of 1000 psi.

III. Conclusions

The most important conclusions to be reached from the initial work is that stresses of the order of ±1000 psi can be determined in Si-single crystals using the non-destructive X-ray Bond technique.

IV. Plans

A second Bond goniometer is being constructed using a high precision 560:1 worm and worm gear with a slow continuous drive. This goniometer will incorporate variable detector holders for easy selection of high angle Si-reflections. A microscope mounted on a rotatable holder is being installed for precision alignment of the Si-ribbons into the primary X-ray beam.

In order to test for imperfection in the Si-ribbons before and after annealing treatment for removal of residual stresses, a

Berg-Barrett topographic camera is being assembled. Specimen and film holder, as well as primary beam collimator, have been obtained from Blake Industries, New Jersey. The camera is being mounted on a track of a GE X-ray tube stand.

References:

Bond, W.L., 1960, Acta Crystallographica 13, 814.

Cullity, B.D. 1956, Elements of X-ray Diffraction, Addison-Wesley, Reading, Massachusetts.

TABLE 1

Values of the angle $2\omega°$ = 180-$2\theta°$, the strain E, and the stress $\sigma_\chi + \sigma_\phi$ across the width of the silicon strip. ($\varepsilon = \Delta d/d = (d-d_0)/d_0$ = 0.00257 $\Delta 2\omega°$; $\sigma_\chi + \sigma_\phi = -1.1 \times 10^6 \cdot \varepsilon$ psi).

	Orientation	$2\omega°$	$2\theta°$	$\varepsilon \times 10^6$	$\sigma_\chi + \sigma_\phi$ psi
I.	$\chi = -6.3°$	32.852	147.148	0	0
	$\phi = -6°$	32.855	147.145	+ 7.7	- 850
		32.850	147.150	- 5.1	+ 560
	Average	32.852_5	147.147_5		
II.	$\chi = +4.4°$	32.852	147.148	0	0
	$\phi = +6°$	32.847	147.153	- 12.9	+ 1420
		32.857	147.143	+ 12.9	- 1420
	Average	32.852	147.148		

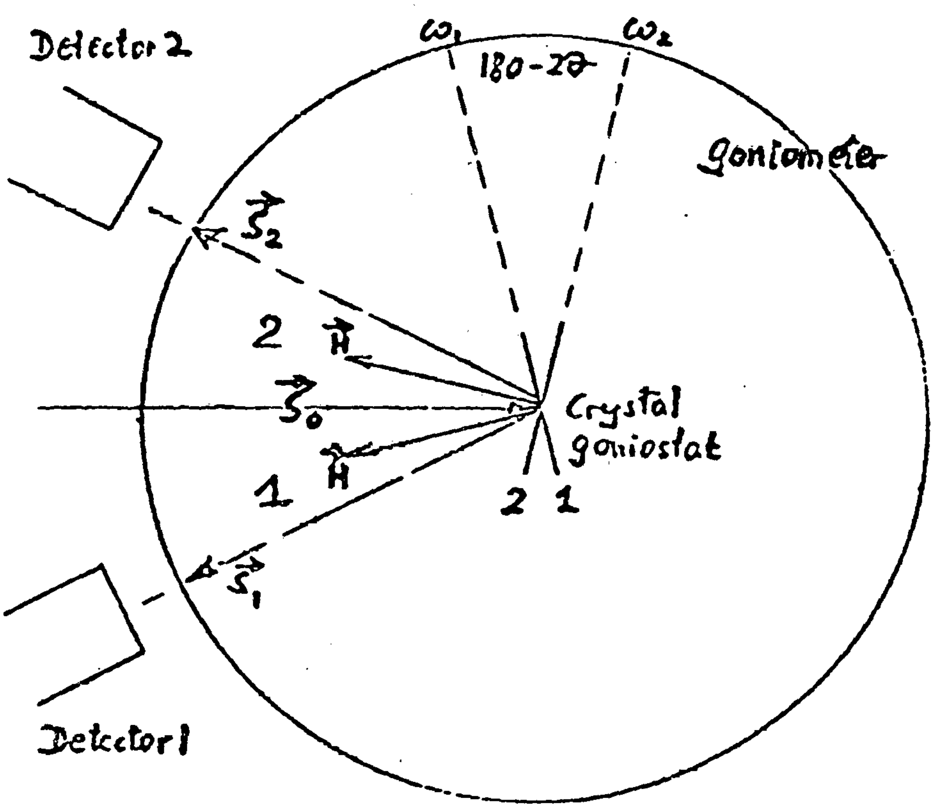

FIG. 1. SCHEMATIC DIAGRAM OF THE BOND TECHNIQUE.

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