Acta Cryst. (1955). **8**, 361

The determination of the elastic constants of silicon by diffuse X-ray reflexions.

By S. C. Prasad and W. A. Wooster, *Crystallographic Laboratory, Cavendish Laboratory, Cambridge, England*

(*Received* 27 *January* 1955)

Introduction

A disc of silicon, 18 mm. diameter, 1 mm. thick and cut parallel to the (100) plane, was loaned to the authors by Dr E. Billig, of the Associated Electrical Industries Laboratories, Aldermaston, Berks. The disc had been cut perpendicular to the growth direction from a large monocrystalline ingot which was pulled from a melt of hyperpure silicon by a special technique developed at the A.E.I. Laboratories. This technique consists essentially of an adaptation of the Czochralski method to deal with highly reactive materials at high temperatures, special care being taken to avoid contamination of the melt and straining of the ingot. This specimen was used in conjunction with a Geiger-counter spectrometer in the manner already described (Ramachandran & Wooster, 1951*a*, *b*) to determine the intensity of scattering from small volume elements of reciprocal space along lines (rekhas) passing through the reciprocal point (relp) 400. The rekhas chosen were parallel to [100], [010] and [110]. The intensity of first-order diffuse scattering is proportional to the reciprocals of c_{11} and c_{44} for the rekhas [100] and [010] respectively. The corresponding intensity for the rekha parallel to [110] is proportional to

$$\frac{1}{c_{11}-c_{12}}+\frac{1}{c_{11}+c_{12}+2c_{44}} .$$

Thus from the measurement on the rekha parallel to [110] the third constant c_{12} can be determined.

Experimental results

The results of observations along the three rekhas parallel to [100], [010] and [110] passing through the relp 400 are given in Table 1 (R is expressed in centimetres on a representation of the reciprocal lattice for which unit distance = 50 cm.).

Table 1

R (cm.)	$1/R^2$ (cm.$^{-2}$)	I (counts in 5 min. for direction of rekha) [100]	[010]	[110]
4·5	0·0494	43·3	55·6	57·3
3	0·111	62·2	105·8	110·9
2	0·25	117·5	193·2	204·3
1·5	0·444	177·3	327·9	325·7
1·25	0·64	246·3	—	—
Background		24	24	24
Slope of line I versus $1/R^2$		345	690	712

Corrections were made for divergence, though only the ψ correction exceeds 1%. A correction of 2% was applied on account of the second-order diffuse scattering.

The ratios of the elastic constants found from these results were as follows:

$$c_{12}/c_{11} = 0{\cdot}39_3,\quad c_{44}/c_{11} = 0{\cdot}50 .$$

It is estimated that the accuracy of the elastic ratios is 5%. The compressibility of silicon is given by Bridgman (1948) as

$$\beta = 1{\cdot}001\times10^{-12}\ \text{cm.}^2\ \text{dyne}^{-1},$$

and since $\beta = 3/(c_{11}+2c_{12})$ we may combine this value with the ratios given above to obtain the values

$$c_{11} = 16{\cdot}8,\quad c_{12} = 6{\cdot}6,\quad c_{44} = 8{\cdot}4\times10^{11}\ \text{dyne cm.}^{-2}.$$

McSkimin, Bond, Buehler & Teal (1951) and McSkimin (1953), using an ultrasonic method, have also determined the elastic constants of silicon and the values obtained are

$$c_{11} = 16{\cdot}57,\quad c_{12} = 6{\cdot}39,\quad c_{44} = 7{\cdot}96\times10^{11}\ \text{dyne cm.}^{-2}.$$

The values obtained in the present investigation using a frequency of approximately 10^{11} Hz. do not differ significantly from those obtained by McSkimin at ultrasonic frequencies.

One of the authors (S. C. P.) feels a great pleasure in recording his gratefulness to the Government of Bihar, India, for the grant of a research scholarship and the leave for the period in which this work has been carried out.

References

Bridgman, P. W. (1948). *Proc. Amer. Acad. Arts Sci.* **77**, 189.

McSkimin, H. J. (1953). *J. Appl. Phys.* **24**, 988.

McSkimin, H. J., Bond, W. L., Buehler, E. & Teal, G. K. (1951). *Phys. Rev.* **83**, 1080.

Ramachandran, G. N. & Wooster, W. A. (1951*a*). *Acta Cryst.* **4**, 335.

Ramachandran, G. N. & Wooster, W. A. (1951*b*). *Acta Cryst.* **4**, 431.

Acta Cryst. (1955). **8**, 361

Bragg reflexion of polarized X-rays from a perfect absorbing crystal.

By K. S. Chandrasekharan, *Department of Physics, University of Madras, Madras 25, India*

(*Received* 7 *March* 1955)

The dynamical theory of X-ray reflexion leads to the well known result that in the case of a surface reflexion from a perfect non-absorbing crystal, the integrated reflexions, obtained with X-rays polarized with the vibration direction parallel and perpendicular to the plane of scattering, are in the ratio of $|\cos 2\theta|:1$, where θ is the Bragg angle. This result, however, has to be modified for actual crystals having finite absorption. For mosaic crystals, the ratio is $\cos^2 2\theta : 1$, independent of absorption, and it can be shown that for perfect crystals, the value of the ratio (α) progressively deviates from $|\cos 2\theta|$ towards $\cos^2 2\theta$ with increasing absorption. The value of α depends in a complex way on the structure factor, Bragg angle, linear absorption coefficient and other constants of the crystal, and could be calculated for any particular case from the semi-empirical formulae derived by Hirsch & Ramachandran (1950).

The change of the ratio with the degree of perfection was observed by Ramaseshan & Ramachandran (1953, 1954), using natural and ground surfaces of a crystal of sodium nitrate. Measurements made with polarized X-rays, on a natural cleavage of a perfect crystal of calcite are reported here. The investigation was undertaken to see whether, for a perfect crystal, the theoretically predicted value different from $|\cos 2\theta|$ is actually found. The natural surface used by Ramaseshan & Ramachandran was not perfect and they obtained a value less than that given by theory, even taking absorption into account. Calcite can be obtained in a highly perfect condition and, further, it has a fairly large linear absorption coefficient (190 cm.$^{-1}$, for Cu $K\alpha$ radiation). Consequently, the theoretical values for the perfect crystal in this case are appreciably different according as to whether absorption is taken into account or not.

The second- and third-order reflexions from a cleavage face of calcite were studied. The integrated intensities at various azimuths of polarization were obtained by a Geiger-counter spectrometer, similar to that adopted by Ramaseshan & Ramachandran (1954) for the study of the degree of perfection of $NaNO_3$. However, the following improvements were incorporated: (*a*) The monochromatic polarized beam of Cu $K\alpha$ radiation was obtained by using a 311 reflexion from a single crystal of copper, which occurs at $\theta = 45° 6'$; thus the beam was almost completely polarized. (*b*) For each azimuthal setting the crystal was oscillated at a slow uniform rate through 3° on either side of the Bragg angle, by means of a synchronous motor, and the integrated intensity was found from the total number of counts recorded by the Geiger counter during this period, a correction being made for the background. The measured integrated intensities had an accuracy of 5%.

The results for the two reflexions are given in Table 1. The theoretical values of $r(\varphi)$ were calculated from the formula $r(\varphi) = \cos^2 \varphi + \alpha \sin^2 \varphi$.

It is seen from the table that within the limits of accuracy of the measurements, the data agree with the values calculated from the dynamical theory, taking absorption into account. It is also interesting to note that when the absorption coefficient is large the range of variation of α from a perfect to a mosaic crystal is appreciably less than the difference between $|\cos 2\theta|$ and $\cos^2 2\theta$.

Table 1. *Variation of integrated intensity with the azimuth of polarization for calcite.*

$r(\varphi)$ is the ratio of the value at azimuth φ to that for azimuth zero. The subscripts *PN*, *PA* and *M* respectively stand for perfect non-absorbing, perfect absorbing and ideally mosaic.

Reflection* and Bragg angle	φ (°)	30	45	60	75	90
422 30° 30′	$r_{PN}(\varphi)$	0·871	0·742	0·614	0·519	0·485
	$r_{PA}(\varphi)$	0·844	0·687	0·531	0·416	0·374
	$r(\varphi)$	—	0·676	0·518	0·400	0·352
	$r_M(\varphi)$	0·809	0·618	0·426	0·286	0·235
633 49° 39′	$r_{PN}(\varphi)$	0·790	0·581	0·371	0·218	0·162
	$r_{PA}(\varphi)$	0·767	0·535	0·324	0·132	0·070
	$r(\varphi)$	0·777	0·597	0·324	0·139	0·069
	$r_M(\varphi)$	0·756	0·513	0·280	0·091	0·026

* The indices are referred to the X-ray unit cell with $a = 6{\cdot}361$ Å, $\alpha = 46° 7'$ (Wyckoff, 1948).

The author wishes to thank Prof. G. N. Ramachandran for suggesting the problem and for guidance during the course of the work.

References

HIRSCH, P. B. & RAMACHANDRAN, G. N. (1950). *Acta Cryst.* **3**, 187.

RAMASESHAN, S. & RAMACHANDRAN, G. N. (1953). *Acta Cryst.* **6**, 364.

RAMASESHAN, S. & RAMACHANDRAN, G. N. (1954). *Proc. Indian Acad. Sci.* A, **39**, 20.

WYCKOFF, R. W. G. (1948). *Crystal Structures*, vol. 1, chap. 7, p. 7. New York: Interscience Publishers.

Acta Cryst. (1955). **8**, 362

A simplified computation technique for structure refinement by means of two-dimensional $F_o - F_c$ synthesis.

By E. HARNIK, *Physics Department, Hebrew University, Jerusalem, Israel*

(*Received* 18 *March* 1955)

In the course of a refinement cycle of a centrosymmetric projection by means of two-dimensional $F_o - F_c$ synthesis each atom is shifted along the gradient, at the same atomic coordinates, of the function

$$D(u, w) + D(x/a, z/c) + \Sigma_h \Sigma_l (F_o - F_c)_{hl} \cos 2\pi (hx/a + lz/c).$$

It follows that the only use one makes of the figure field is the determination of the magnitude and direction of these gradients, and, therefore, one may be interested in computing the figure field only in the immediate neighbourhood of the atomic positions. This, however, cannot be done conveniently with the Beevers–Lipson strips when used in the conventional way, but it can be performed quite easily and rapidly if the strips are given a different interpretation.

The Beevers–Lipson strips present the function $A \cos 2\pi hu$ or $A \sin 2\pi hu$; for any given value of A and of h there is a strip on which the values of the function