

single peaks, indicating the strength of the multiple regular vectors. The correct peak (Fig. 2) was the sharpest of the three and, after subtraction of mean background, was 25% larger than the other two. The exact rotational parameters were determined with a  $1^\circ$  grid and these were then used to calculate the relative fractional coordinates of the fragment in the unknown ( $P2_1$ ) cell.

The absolute position of the correctly orientated rigid group was found using the  $Q(X_0Z_0)$  function (Tollin, 1966) with the 350 largest  $|E_p|^2$  (Fig. 3). This function uses a modified sum function (Buerger, 1959) to express the correlation of the cross vectors between symmetry related molecular fragments with the observed Patterson. For a structure containing a  $2_1$  axis, a spurious peak will occur if there is a pair of atoms in the known group whose  $y$  coordinates differ by  $\frac{1}{2}$ . Seven such false peaks were expected and are marked with an 'X' in Fig. 3. The correct peak, which fixes the position of the fragment relative to the crystallographic  $2_1$  axis, is marked with a '+'.

The resulting atomic position were compared with those obtained from least-squares refinement and found to be in error by from 0.03 to 0.23 Å. Calculation of an  $|F_{obs}|$  synthesis using the trial coordinates clearly revealed the positions of C(29), O(30), C(31), Cl(32), C(21), C(22), and C(23), virtually completing the structure solution.

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**Weak extra peak in rocking curves of X-ray reflexion for incident beams obtained by successive asymmetric reflexions.** By SEISHI KIKUTA, KATSUHIRO KAWASHIMA\* and KAZUTAKE KOHRA, *Department of Applied Physics, Faculty of Engineering, University of Tokyo, Hongo, Bunkyo-ku, Tokyo, Japan*

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A numerical calculation is made on the intensity distribution of the beam obtained from the X-ray collimator system of monolithic silicon crystal, in which double 422 asymmetric Bragg-case diffractions of  $\text{Cu } K\alpha_1$  take place successively. It is concluded that a weak subsidiary peak found in the rocking curve obtained with a double-crystal diffractometer of parallel setting using a collimator of the above type is caused by the relative angular shift of the range of total reflexion for each crystal component due to the refraction effect.

In a previous paper (Kohra & Kikuta, 1968), we reported a monolithic collimator system, by which a substantially parallel X-ray beam of an angular spread of  $0.1$  or  $0.01''$  can be obtained. This collimator consisted of three crystal com-

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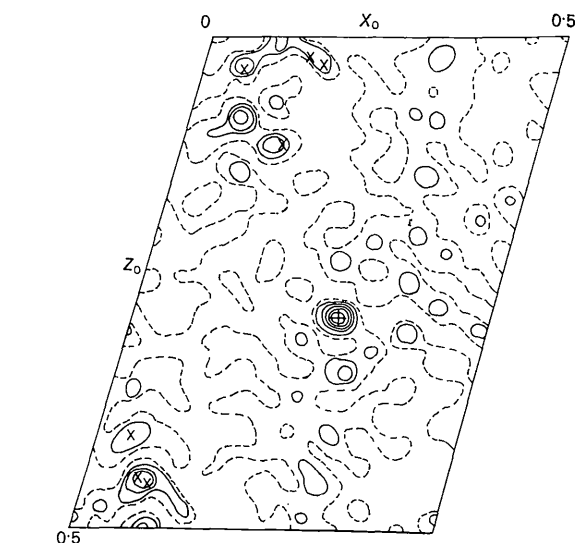


Fig. 3.  $Q(X_0Z_0)$  for *i*-cholesteryl chloroacetate. × indicates the false peaks. + indicates the correct peak.

ponents prepared from a single block of silicon crystal, in which double or triple 422 asymmetric Bragg-case diffractions of  $\text{Cu } K\alpha_1$  radiation take place successively. As its application, we also reported the measurement of rocking curves for the 422 reflexion from silicon single crystals with various asymmetry factors, using this collimator in place of the first crystal in a double-crystal diffractometer of parallel

setting. It was there noticed that the diffraction peak was accompanied by a weak subsidiary peak in general, which was conspicuous particularly for the curve of narrow half-value width. In the present paper, it is pointed out that this extra peak is due to a characteristic of the angular distribution of the exploring beam obtained by the collimator of the above type.

As in the previous paper, the asymmetry factor is defined by

$$b = \frac{\sin(\theta_B - \alpha)}{\sin(\theta_B + \alpha)}, \quad (1)$$

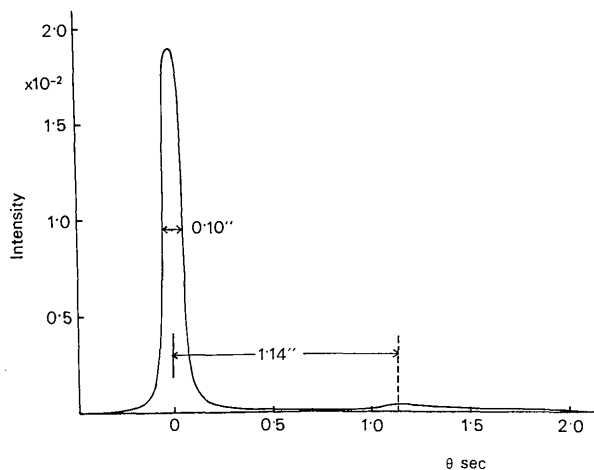


Fig. 1. Calculated angular intensity distribution of the beam ( $\text{Cu } K\alpha_1$ ) obtained from the collimator system of monolithic silicon crystal, in which double 422 asymmetric reflexions with  $b=0.106$  take place. The  $\theta$  scale (sec of arc) is used for convenience, instead of the  $W$  scale.

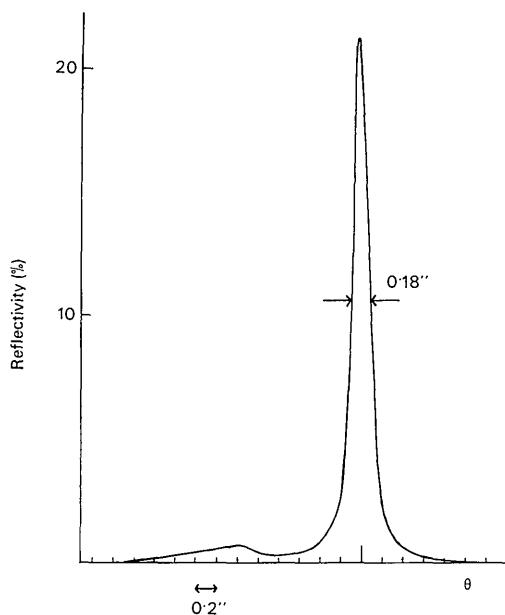


Fig. 2. Convolved curve of the curve given in Fig. 1 and the intrinsic curve for 422 asymmetric reflexion of silicon with  $b=573$ . The  $\theta$  scale is used instead of the  $W$  scale.

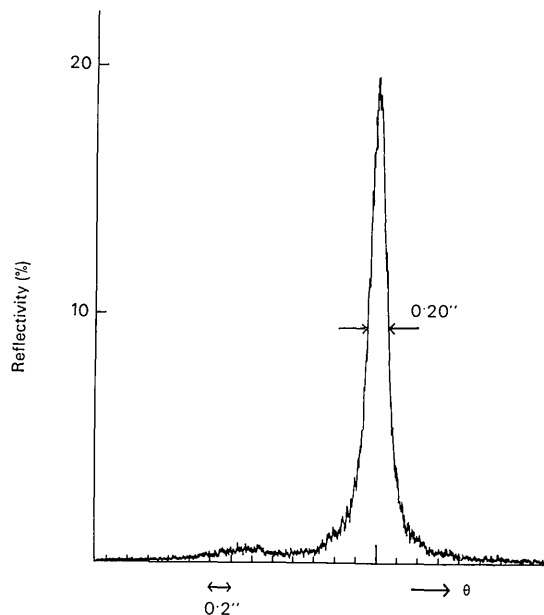


Fig. 3. Rocking curve corresponding to Fig. 2 measured with a double-crystal diffractometer of parallel setting for 422 reflexion of silicon with  $\text{Cu } K\alpha_1$ .

where  $\alpha$  is the angle between the crystal surface and the diffracting plane concerned and  $\theta_B$  is the Bragg angle. If we assume that the asymmetry factors of the first and the second crystal components are the same and the diffracting planes in these components are perfectly parallel to each other, the angular distribution of the X-ray beam diffracted successively by the two crystal components is given as

$$I(W) = R(W)R \left( bW - \frac{1-b^2}{2b^{1/2}} \frac{|\psi'_0|}{|\psi'_h|} \right), \quad (2)$$

where  $R(W)$  is the intrinsic reflexion profile for the first crystal component,  $W$  a parameter representing the angular deviation from the Bragg condition (Hirsch & Ramachandran, 1950), and  $\psi'_0$  and  $\psi'_h$  the real parts of the zero and  $h$ th Fourier components of  $4\pi$  times the polarizability, respectively. In equation (2), we use the  $W$  scale referred to the first crystal component. The result of the numerical calculation of  $I(W)$  for the collimator used in practice is shown in Fig. 1. An extra peak is seen on the high-angle side of the main peak.

The diffraction curve from a specimen crystal obtained with the above exploring beam should be the convolution of  $I(W)$  and the relevant intrinsic diffraction curve. Fig. 2 shows the numerical calculation of the convoluted function assuming the theoretical intrinsic diffraction curve of 422 for silicon crystal with  $b=573$ .\* It is seen that the extra peak at the high-angle side of the main peak moved to the low-angle side by convolution. Fig. 3 is an experimentally observed curve corresponding to Fig. 2. The agreement between these curves is quite satisfactory, including the extra peak.

The extra peak is caused by the circumstance that the di-

\* In the previous paper, the curve for  $b=344$  was measured. The essential feature is not different.

rection of the beam diffracted from the first crystal component of the collimator deviates slightly from the diffraction condition for the second crystal component owing to the refraction effect. This deviation can be avoided by inclining the second crystal component relatively to the first one by an appropriate angle (about  $15^\circ$  in the present case). Such an adjustment has successfully been tried and it was proved that the intensity of the exploring beam obtained from the collimator can be increased to a value about 35 times that without the inclination, and the extra peak vanishes in the observed rocking curve (Kikuta & Kohra, 1968; Matsushita, 1969; Matsushita, Kikuta & Kohra, 1969).

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## Notes and News

*Announcements and other items of crystallographic interest will be published under this heading at the discretion of the Editorial Board. The notes (in duplicate) should be sent to the Executive Secretary of the International Union of Crystallography (J. N. King, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England).*

### Bertram Eugene Warren Diffraction Physics Award

The first Warren Award of the American Crystallographic Association was given to Dr Ulrich Bonse and Dr Michael Hart in recognition of their joint work on the X-ray interferometer and its application to the study of the physics of solids. Formal presentation of the Award was made on 20 August, 1970, at the Ottawa Meeting of the A.C.A.

The Award was established by students and friends of Professor Warren on the occasion of his retirement in 1967 from the Massachusetts Institute of Technology. It is given for an important recent contribution to the physics of solids or liquids using X-ray, neutron, or electron diffraction techniques, published within a six-year period ending 30 June of the year preceding the year in which the award is made. The award consists of a certificate and \$1000 and is to be given every three years.

### International Council of Scientific Unions Abstracting Board (ICSU AB)

The ICSU AB has announced its plans to go ahead with the first stage of a world system for abstracting and indexing services for science and technology.

The member services of the Board are *Astronomy and Astrophysics Abstracts*, *Bibliographie des Sciences de la Terre*, *Bibliography and Index of Geology*, BioSciences Information Service of *Biological Abstracts*, *Bulletin Signalé-*

*tique*, Chemical Abstracts Service, Chemischer Informationsdienst, *Science Abstracts*, *Physikalische Berichte*, *Referativnyi Zhurnal*, *Zentralblatt für Mathematik*. Major international scientific unions, including the IUCr, are also members of the Board. Under the new plan member services from each discipline will be responsible for abstracting certain journals and contributing the abstracts to a pool. This will greatly reduce the duplication of work and the number of articles processed by each abstracting service.

The proceedings of the meeting of the ICSU AB in July 1970, which include the description of the first stage of the plan, are available from the ICSU Abstracting Board Secretariat, 17 rue Mirabeau, Paris 16e, France (U.S.\$15.00, plus mailing charges).

### International Union of Crystallography

The Editors of Volume I of *International Tables for X-ray Crystallography* regret that there is an omission in Table 3.4.1 (page 30) of the 1969 edition. In the cubic system the Laue Class of highest symmetry has been omitted. This should be inserted below the Laue Class  $m\bar{3}$  as follows:

432
$4\bar{3}m$
$m\bar{3}m$

## International Union of Crystallography

### Commission on Crystallographic Apparatus Phase II of the I. U. Cr. Single Crystal Intensity Measurement Project

An International Project which will be phase II of the Single Crystal Intensity Measurement Project, SCIMP (Abrahams, Hamilton & Mathieson, 1970), is proposed by the Commission on Crystallographic Apparatus of the International Union of Crystallography. The plan of the phase II project is for each participant (individual or group) to derive for a specific material,  $\alpha$ -glycine, a set of experimental structure factors,  $F_o$ , as independent of the individual crystal as pos-

sible. To this end, corrections for absorption and extinction will be obligatory. The aim of the project is to determine the measure of accord achieved by the participants over a range of crystals, diffractometers and techniques and also to obtain some guide as to the efficacy of experimental procedures for the diagnosis and correction of extinction effects.

The reasons for initiating phase II of SCIMP derive from the results of phase I, the report on which clearly indicated that, for the material used, D(+)-tartaric acid, the particular characteristics of the individual crystal constituted the major source of error in the derivation of accurate  $F_o$  values. The functional trend of the differences between  $F$  values for different crystals has identified extinction as the main error