A Correction Procedure for the Errors in Single-Crystal Intensities Due to the Inhomogeneity of the Primary X-ray Beam*

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Abstract

Graphite monochromators are known to give rise to non-homogeneous primary X-ray beams. When intensities of single crystals are measured the effective cross section of a non-spherical crystal in the X-ray beam depends on its orientation in the beam. Therefore, systematic errors in the measured integrated intensities are introduced by the inhomogeneity of the incoming beam. A correction for these errors can be made, knowing the intensity profile of the primary beam and the dimensions and orientation of the crystal in the beam. The correction can conveniently be applied with the absorption correction. Examples of the corrections are given for crystals with rational boundary planes. It is shown that the intensity of an X-ray reflection as a function of the rotation about the scattering vector (ψ rotation) can be calculated with fair accuracy. In some cases (large elongated crystals in an inhomogeneous beam) correction for absorption only may give results which are worse than those with no correction at all.

Introduction

The main disadvantage of graphite monochromators is that they give rise to inhomogeneous X-ray beams. This inhomogeneity causes systematic errors in measured intensities of non-spherical crystals. These are due to the fact that the effective area of the crystal in the beam is a function of the crystal orientation. These errors were considered to be so important by Coppens, Ross, Blessing, Cooper, Larsen, Leipoldt & Rees (1974) that they preferred a β filter instead of a monochromator. A solution for the problem of the inhomogeneity was given by de Boer (1974). His method, of which a pictorial representation is given by Helmholdt & Vos (1977), is based on controlled absorption of the primary beam. A homogeneous region in the beam can be obtained at the expense of the total intensity.

It is the purpose of this note to introduce a practical way to correct for the inhomogeneity error, knowing the intensity profile of the beam and the shape and orientation of the crystal in the beam. The correction can conveniently be performed simultaneously with an absorption correction which makes use of the Gaussian quadrature method. For each grid point used in the absorption-correction programme, the local intensity is taken into account. In this way the error due to inhomogeneity can be calculated and corrected. For a four-circle diffractometer the correction procedure can be tested by comparing observed and calculated intensity variations for a reflection at different azimuthal angles (ψ rotation about the scattering vector).

This correction procedure is applicable only when the centering of the crystal and the intensity profile does not change during data collection.

Experimental

The primary beam intensity profile of our diffractometer (Philips PW1100, Mo $K\alpha$ radiation, flat graphite monochromator, fine-focus tube, 0·8 mm collimator, take-off angle 6°) was determined. The measurements were done with a pinhole (diameter 30 µm) in platinum foil (0·1 mm). The foil was fixed on a goniometer head and by moving the pinhole perpendicular to the primary beam the intensity profile was measured. The geometry adopted in the measurements is shown in Fig. 1.

The results of the intensity profile measurements are given in Figs. 2 and 3, which show the intensity profile and two cross sections respectively. As can be seen from the figures the inhomogeneity in the y direction is very pronounced, while the intensity in the x direction is nearly constant. This effect, mainly due to the orientation of the monochromator has also been found by others (Coppens *et al.*, 1974; de Boer, 1974). The profile given by Tanaka (1978) is quite different

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showing almost circular intensity contours, presumably due to a smaller collimator diameter.

In order to test the correction procedure, two relatively large organic crystals ($\mu = 0.135$ mm^{-1}) of elongated shape were selected. The dimensions of the
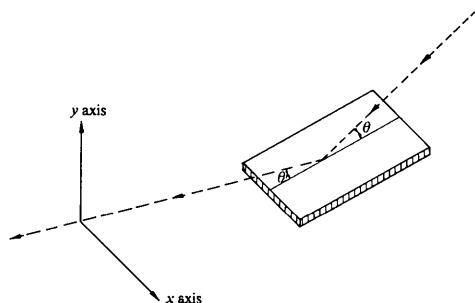

Fig. 1. Monochromator arrangement and coordinate system used.
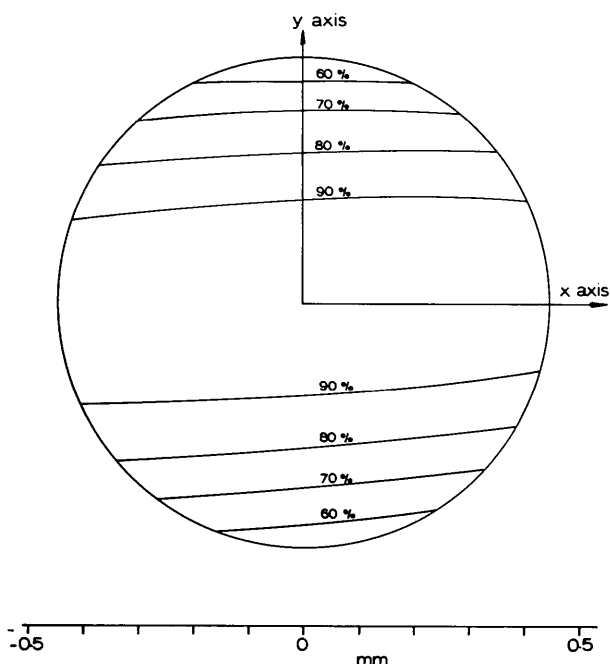

Fig. 2. The measured primary beam intensity profile. Lines of equal intensity are drawn at intervals of 10% of the maximum intensity.
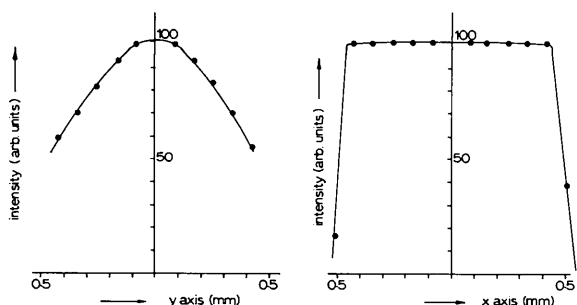

Fig. 3. Vertical (y axis) and horizontal (x axis) cross section of the measured primary beam intensity profile.

crystals were approximately $0.57 \times 0.10 \times 0.21$ mm for crystal 1 and $0.93 \times 0.07 \times 0.17$ mm for crystal 2. The crystals were mounted with the longest edges perpendicular to the axis of the goniometer head. To get maximum effects from the inhomogeneity, reflections were chosen in such a way that the axis of the ψ rotation was approximately parallel to the x axis of Fig. 2. Integrated intensities were measured at regular intervals of $\psi(\Delta\psi = 15°)$. The observed intensity variations for a reflection from crystal 1 and crystal 2 are shown in Figs. 4 and 5 respectively. As can be seen from the figures, appreciable intensity variations are observed especially for the larger crystal.

Calculations

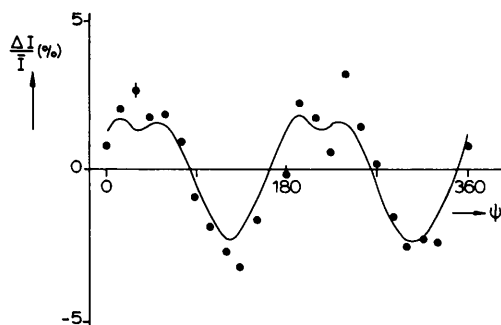In order to keep the calculations simple the intensity profile of the primary beam has been approximated by

Fig. 4. Observed (●) and calculated (solid line) relative variation of integrated intensity for the $\bar{2}00$ reflection of crystal 1.
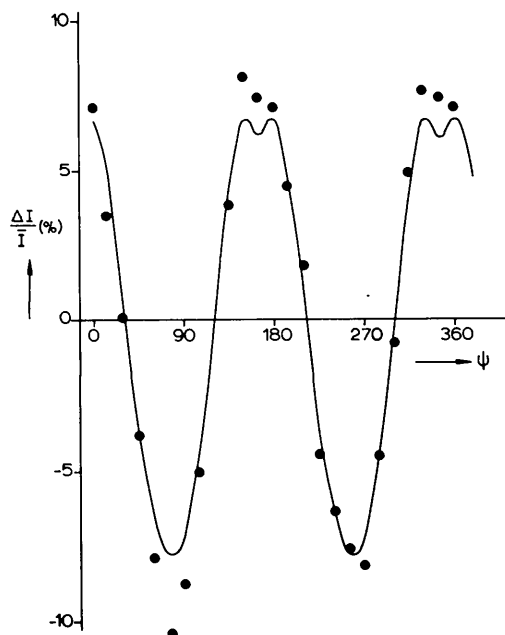

Fig. 5. Observed (●) and calculated (solid line) relative variation of integrated intensity for the $\bar{2}00$ reflection of crystal 2.

a single Gaussian function in the y direction. The inhomogeneity in the x direction has been neglected. A correction procedure taking into account this intensity profile has been incorporated in the absorption program $ACXR$ (Harkema, 1978) which uses a Gaussian quadrature scheme. For each reflection the projections of the grid points on the xy plane of Fig. 2 are calculated. The local intensity at each grid point is then calculated from the approximated intensity profile. In this way the absorption and the inhomogeneity error are taken into account.

Results and discussion

Calculated intensity variations for two reflections are shown in Figs. 4 and 5 together with the observed variations. The variations have been calculated taking into account the inhomogeneity and absorption corrections. A fair agreement between observed and calculated curves is found in both cases. The agreement can be given in a more quantitative way as R factors for the intensities before and after correction. Data on different reflections are given in Table 1. From this table it can

Table 1. *Agreement (expressed as R factors) of the same reflection at different ψ angles before and after correction for absorption and inhomogeneity*

$$R = \frac{\sum_i |I_i - \bar{I}|}{\sum_i I_i} \times 100\%.$$

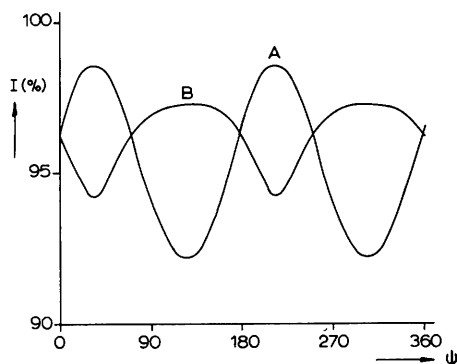| Crystal	Reflection	Uncorrected	Absorption only	Absorption + inhomogeneity
1	$\bar{2}00$	1·79	2·71	0·62
1	$\bar{2}12$	1·52	2·29	0·77
2	$\bar{2}00$	5·77	7·01	0·64
2	$\bar{2}01$	5·33	6·69	0·83
2	$\bar{3}14$	4·33	5·27	1·17

Fig. 6. The total calculated intensity for the $\bar{2}00$ reflection of crystal 1, as a function of ψ, taking into account the inhomogeneity of the primary beam (curve A) and the absorption (curve B).

be concluded that the observed R are of the order of magnitude of 1% after correction.

For the reflection of Fig. 4, the calculated intensity variations due to inhomogeneity and absorption are given separately in Fig. 6. From this figure the interesting observation can be made that absorption and inhomogeneity corrections are out-of-phase. The maximum absorption occurs at the point where the inhomogeneity error is minimal and *vice versa*. At small diffraction angles this is generally true. The inhomogeneity error is minimal when the effective cross section in the xy plane is minimal. At the same point, however, the effective dimension of the crystal in the direction perpendicular to the xy plane, which is related to the absorption at zero diffraction angle, is a maximum. From the curves given in Fig. 6 it can be inferred that, in cases where the inhomogeneity error is of the same order of magnitude or larger than the absorption correction, correction for absorption only gives results which are worse compared with those with no correction at all. This conclusion is corroborated by data given in Table 1, where only absorption correction has been applied. The resulting R factors for the reflections, for all of which the inhomogeneity error is the predominant effect, increase in applying the absorption correction.

This effect, that an absorption correction can give worse results, only applies to absorption corrections which use the shape of the crystal in the calculations. For empirical absorption corrections (*e.g.* Flack, 1977), the final corrections are based on experimental ψ scans of different reflections. When the reflections on which the corrections are based are properly chosen, it can be expected that the correction contains at least some part of the inhomogeneity error. Methods which determine crystal shapes or absorption coefficients from ψ scans (Rigoult, Tomas & Morosini, 1979) are liable to give physically meaningless parameters when inhomogeneous beams are used.

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