SHORT COMMUNICATIONS

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The learnt-profile method in four-circle diffractometry: the variation of reflexion peak width.

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Abstract

The anisotropic functional form previously suggested for the variation of reflexion peak width in reciprocal space does not adequately deal with cases of high anisotropy when measurements are made at different azimuthal angles. A small modification produces considerable improvement.

We have previously reported an application of the learnt-profile method to single-crystal diffraction data collection on a four-circle diffractometer (Clegg, 1981). Essential to the method is the derivation and use of an anisotropic functional form for the width of the α_1 and α_2 components of each reflexion, so that the correct scan parameters can be chosen. We used a seven-parameter function of the form

$$W = \sum\sum a_i a_j A_{ij} + B \tan\theta,$$

where the a_i are the direction cosines of the diffraction vector relative to a crystal-fixed set of Cartesian axes, and A is a symmetric 3 × 3 tensor. The term $B \tan\theta$ allows for wavelength dispersion within α_1 and α_2.

This form has been found to be very satisfactory in most cases, but it assumes that W is independent of the azimuthal angle for which the reflexion is measured. If all reflexions are measured at or close to the bisecting geometry (azimuthal angle = 0), or if the width function is approximately isotropic, this shortcoming is unimportant. If, however, different azimuthal angles are to be used (*e.g.* for application of empirical absorption corrections) and the width function is highly anisotropic, a poor match of expected and measured reflexion width is obtained, and the learnt-profile method breaks down. Such anisotropy of the width function can be caused by non-uniformity of crystal dimensions (*e.g.* for needle and plate crystals), anisotropy of mosaic spread, or both. Thus it is closely related to anisotropy of secondary extinction. Reference to the work of Coppens (1969) and Coppens & Hamilton (1970) suggests that the necessary improvement to the functional form for W is to replace the direction cosines a_i by d_i, the direction cosines of the vector perpendicular to the diffraction plane. Incorporation of this change into the data collection procedure improves the match of expected and measured reflexion widths for measurement in non-bisecting geometries and makes the method applicable for azimuthal scan measurements even in extreme cases of anisotropy.

In such cases, when data are to be collected at different azimuthal angles, it is advantageous to include measurements at a range of azimuthal angles in the preliminary routine which determines the width parameters and an initial normalized reflexion profile, so that these are obtained under conditions as similar as possible to those under which data collection takes place.

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Least-squares refinement of unit-cell parameters from precession photographs.

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Abstract

A method is described for the determination of reciprocal-cell parameters by least-squares refinement from (x,y) Cartesian coordinates of reflexions on precession photographs.

An advantage of the precession method of single-crystal X-ray photography over the Weissenberg method is the production of an undistorted record of each reciprocal-lattice level. Reciprocal-unit-cell parameters can easily be obtained by direct measurement of angles and distances on the film. With a suitable single mounting of the crystal, two zero-layer

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