

Acta Cryst. (1976). A32, 168

Comments on *High-precision orientation of crystals using the Laue method with characteristic X-rays* by G. Christiansen, L. Gerward and I. Alstrup. By G. J. MCINTYRE and Z. BARNEA, *University of Melbourne, School of Physics, Parkville, 3052, Australia*

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It is pointed out that under certain conditions one may need to take account of absorption and other effects in order to achieve the high precision claimed by Christiansen, Gerward & Alstrup [*Acta Cryst.* (1975). A31, 142–145].

In the paper by Christiansen, Gerward & Alstrup (1975) no account is taken of the effects of absorption on the set of reflexions whose intensities are being compared. However, when the planes giving rise to these reflexions make unequal angles with the surface of the crystal, because of miscut, their intensities must be unequal even when they are oriented symmetrically about the incident X-ray beam (Mair, Prager & Barnea, 1971). The balancing of the intensities achieved by the adjustment of the angle of misfit will then result in an orientation error introduced deliberately by the unjustified criterion of equal intensities. This effect of absorption increases with the angle of miscut and with decreasing θ . For instance, for $\theta = 55^\circ$ a 5° miscut will result in a 6% intensity difference between symmetrically oriented reflexions; a 10° miscut will result in a 12% difference. These differences must be compared with the 2–5% precision required to achieve an orientation to within 0.01° .

It should also be pointed out that in non-centrosymmetric crystals care must be taken that the reflexions compared are indeed equivalent when dispersion effects are allowed for. Comparison of non-symmetry-related reflexions is only justified when anharmonic effects are negligible. This is not

necessarily the case even at room temperature. We have observed differences of the order of 3% between the structure factors of non-symmetry-related reflexions in zinc selenide. Differences of this magnitude are also typical of other semiconductor crystals.

The procedure proposed by Christiansen, Gerward & Alstrup could be modified by introducing the effects of absorption into the program calculating the expected Laue pattern. However, since the angle of miscut can only be found in the course of orienting the crystal, the procedure must of necessity become iterative and more laborious. The problem should be avoided by a sufficiently accurate preliminary orientation and corrective polishing, prior to the final high-precision orientation.

References

- CHRISTIANSEN, G., GERWARD, L. & ALSTRUP, I. (1975). *Acta Cryst.* A31, 142–145.
 MAIR, S. L., PRAGER, P. R. & BARNEA, Z. (1971). *J. Appl. Cryst.* 4, 169–171.

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Reply to McIntyre and Barnea's comments on our article *High-precision orientation of crystals using the Laue method with characteristic X-rays*. By G. CHRISTIANSEN, L. GERWARD and I. ALSTRUP, *Laboratory of Applied Physics III, Building 307, Technical University of Denmark, DK-2800 Lyngby, Denmark*

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It is agreed that the miscut must be kept small in the method of Christiansen, Gerward & Alstrup [*Acta Cryst.* (1975). A31, 142–145], as pointed out by McIntyre & Barnea [*Acta Cryst.* (1976). A32, 168]. The comment on dispersion was covered in the original paper.

We agree with McIntyre & Barnea (1976) that our high-precision method implies that the miscut should be kept small during the final adjustment.

We admit that this point was not stressed adequately although it is stated in the reference by Mathiesen, Gerward & Pedersen, cited in our article, that the crystal slice should be cut within a few tenths of a degree from the desired orientation before the final high-precision orientation. For this purpose the first two steps in the orientation procedure discussed in § 3 of our article can be used: (1) The use of the ordinary Laue spots, and (2) the use of the predicted pattern of characteristic spots (without taking their intensities into account).

Concerning the remark on dispersion effects it is clearly stated in our article that the reflexions, the intensities of which are compared, should have the same structure factor. This factor as well as other relevant diffraction data are calculated for each reflexion.

Reference

- MCINTYRE, G. J. & BARNEA, Z. (1976). *Acta Cryst.* A32, 168.