

11.7-19 NEW DESIGN OF A DOUBLE CRYSTAL DIFFRACTOMETER AND SOME APPLICATIONS. By W. Fiedler, W. Heyer, G. Hildebrandt and G. Jähnig, Fritz-Haber-Inst./MPG, Berlin

In a conventional double crystal diffractometer where the second crystal (C II) is adjusted by the main axis, it is quite inconvenient to change diffraction conditions. In order to get a more flexible apparatus we incorporated the following principles.

- 1) The main diffractometer axis (A I) controls the first crystal (C I).
 - 2) The second axis (A II) which adjusts C II is fixed in an eccentric position on a heavy disc which rotates about A I. The axis of a counter holder can be brought into the position of A II. After adjusting A II into the beam reflected from C I, the disc is fixed by raising it a small distance from its ball-bearings.
 - 3) After adjusting C II roughly into reflecting position A II is fixed at its lower end, and the fine adjustments (or repeated runs through the diffraction pattern) are done by torsion of A II.
 - 4) The torsion of the A II axis is provided by a lever which supports the holder for C II. In a distance of 206.3 mm from A II this lever carries a steel ball (of a micrometer screw) which is slightly pressed against a 100 mm long polished glass block mounted on a precision traverse. This traverse is fixed on the disc and translates towards A II. Another micrometer screw adjusts the block slightly obliquely to the translation direction. Thus moving the traverse 0.5 mm (200 steps of a step motor), the angle at C II is changed 1 sec. of arc, with the oblique angle adjusted to 1:500.
 - 5) We used this arrangement to check the quality of calcite crystals with surface topography. With two sufficiently perfect CaCO_3 crystals the effect of the "inverse absorption asymmetry" has been demonstrated; this is reported elsewhere in this volume (W. Heyer, G. Hildebrandt).
 - 6) Initially only to check the reliability and stability of this design, diffraction patterns of silicon crystals have been measured and compared with theory varying the structure factor in the calculations such to get the best fit with the experimental curves (these curves were the result of at least 20 runs through the diffraction pattern, measured overnight with remote control; corrections for a temperature slope were considered if the slope was small and constant, otherwise the measurement had to be repeated). As a result f -values with a mean deviation of about 0.5 % from the supposedly best values in the literature have been measured without difficulty. At present, attempts are being made to combine A II with a light optical system to additionally measure the changes in diffraction angle directly.
- Up to now, the following results have been achieved (f_0 values, i.e. values corrected for anomalous dispersion):

| $hkl \setminus \lambda$ | $\text{CuK}_{\alpha 1}(1)$ | $\text{MoK}_{\alpha 1}(1)$ | (2) | (3) |
|-------------------------|----------------------------|----------------------------|-------|-------|
| 220 | 8.664 | 8.621 | 8.666 | 8.665 |
| 440 | 6.107 | 6.048 | 6.036 | 6.042 |
| 660 | - | 3.891 | 3.850 | 3.860 |

- (1) This work. (2) Mean values, taken from Aldred/Hart, Proc.R.Soc.Lond.A332(1973)223; Bonse/Teworte, J.Appl. Crystall.13(1980)410; Hart/Milne, Acta Cryst.A25(1969)134, and Tanemura/Kato, Acta Cryst.A28(1972)69.
(3) International Tables, Vol.4(1974).

The striking advantage of this particular method for measuring structure factors lies in the fact that only small perfect areas in the crystal surfaces are needed. It is therefore our goal to check the feasibility of the method with less perfect crystals (such as e.g. certain minerals).

11.7-20 ABSOLUTE MEASUREMENT OF THE PEAK SHIFT IN THE CASE OF ASYMMETRIC BRAGG DIFFRACTION
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As is well known, the peak shift in the case of asymmetric Bragg diffraction depends on the angle of asymmetric α (i.e. the angle between the reflecting net planes and the entrance surface of the crystal) and the coherent scattering length b_c (using neutrons) or the atomic scattering factor f (using x-rays). Therefore the precise measurement of the peak shift of the reflected beam principally provides another method for the accurate determination of b_c or f values.

In earlier experiments only the change of the Darwin width or the amount of refraction of the diffracted beam had been determined. With our method the symmetrically and asymmetrically reflected beams are measured by a single experiment which yields absolute values of the peak shift. For this purpose we used all advantages of our double crystal diffractometer at the BER II reactor (HMI, Berlin), i.e. high angular resolution and mechanical and thermal stability. To measure the shift we used the parallel setting of two perfect Silicon crystals. The first one (together with a preset graphite crystal and a collimator) provides the necessary beam preparation. The second one was prepared in such a way, that its surface consisted of a symmetrical and an asymmetrical part with respect to the (111) reflecting net planes (Fig. 1).

In the first test measurement (Fig. 2) neutrons with a mean wavelength $\lambda_0 = 0.2453(3)$ nm and an asymmetry angle $\alpha = 0.3604(2)$ rad (20.65(1)°) were used. We measured a peak shift $\theta_S = 48.5(1.1)$ μ rad corresponding to a coherent scattering length $b_c = 4.13$ fm which agrees within 3% to well established values in the literature.

This technique can easily be improved in various ways, such as using smaller glancing angles which means larger peak shifts (150-200 μ rad) and controlling the absolute shift of the peak with a calibrated optical device (the optical control set up is in the test mode). This method offers especially for x-rays and synchrotron radiation an interesting application for precise structure factor measurements; in the case of synchrotron radiation the higher orders yield additional peak shifts and therefore values for the determination of f .

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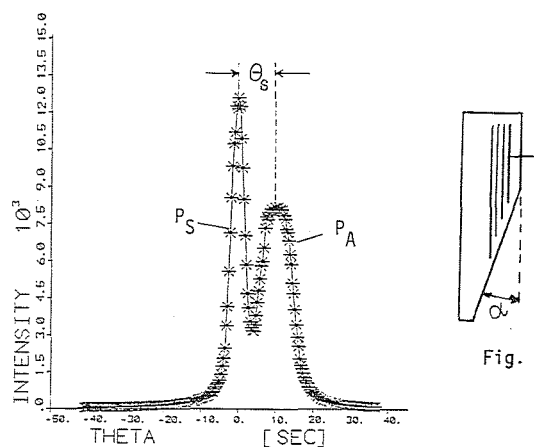


Fig. 1

Fig. 2 Symmetrically (P_S) and asymmetrically (P_A) reflected beam. Peak shift $\theta_S = 10.0(2)$ sec of arc (48.1(1.1) μ rad)