

11.7-21 EXPERIMENTAL EVIDENCE OF THE "INVERSE ABSORPTION ASYMMETRY", MEASURED WITH X-RAYS IN CALCITE. By W. Heyer and G. Hildebrandt, Fritz-Haber-Inst./MPG, Berlin

It is well known (in both Bragg and Laue cases) that the relationship between the wave field produced in a perfect crystal and the lattice depends directly on the direction of the incident wave. In most cases (e.g. in all reflections of monoatomic crystals), refraction and absorption increase with increasing diffraction angle ("Prins-Kohler asymmetry"). If, however, polyatomic crystals contain atoms with strongly different scattering and absorption powers, the inverse can happen: decreasing absorption with increasing scattering angle leads to the "inverse absorption asymmetry". This has been predicted already in 1962 by G. Borrmann (Z.Kristallogr. 120(1964)143, and J.M. Cowley recommended to use the 222 planes in calcite or the 110 planes in sodium nitrate for an experimental proof; but using the Laue case, he found a clear indication of the effect only with  $\text{NaNO}_3$  (Acta Crystallogr. 17(1964)33).

As mentioned elsewhere in this volume (Fiedler, Heyer, Hildebrandt, Jähnig) we used a double crystal diffractometer with a somewhat unconventional design for the detection of the above effect in highly perfect calcite crystals in the Bragg case (222 reflection,  $\text{CuK}_\alpha$  radiation). These crystals were selected using surface topographical methods (Berg-Barrett, Lang, Double Crystal); artificial 111 and 655 surfaces were prepared by grinding and etch-pit-free etching with diluted (2%) CP 4.

Considering symmetrical and asymmetrical reflections, there exist nine possible (n;-n) arrangements (net planes of the crystals nearly parallel); in three pairs, however, FWHM and shape are predicted to be the same, only the percent reflection differs. Out of the six remaining configurations, three are suited to reveal the diffraction pattern of the test crystal: high resolution together with high intensity offers the quasi-dispersion-free V-S-arrangement (Renninger's notation: crystal I with grazing incidence, crystal II in symmetrical reflection), whereas the other two settings show a small dispersion effect due to the different refraction by the crystals, depending on the amount of asymmetry. Using the crystal with a 655 surface as crystal I, the asymmetry factor is  $b = -0.055$  (with  $\theta_{222} = 15.76^\circ$  and the asymmetry angle  $14.18^\circ$ ). With a symmetrical 222 reflection at crystal II, a diffraction pattern with a FWHM of 2.15" and a percent reflection of 63% should result; the measured values were 2.88" and 48%, resp., the discrepancy probably being due to crystal defects. The integrated intensity, however, agreed to 1.2% to the expected theoretical value (comparable to Renninger's results on the cleavage plane (211); Acta Cryst. 8(1955)597). In Fig. 1 the measured pattern (curve with points) is compared to a calculated one, which results from a reduction of the theoretical pattern to 48% reflectivity, keeping the integrated intensity constant. The asymmetry lies clearly on the correct side; this is even more evident if the measured pattern is reflected at an ordinate traversing the maximum (Fig. 2). Therefore these measurements yield a clear evidence of the expected effect.

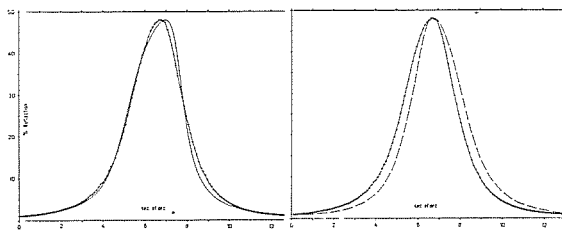


Fig. 1

Fig. 2

Theory ——— ; meas. - - - - - ; meas.(refl.) - - - - - .

11.7-22 THE ANGULAR DEPENDENCE OF ATOMIC X-RAY DISPERSION CORRECTIONS. By G. Hildebrandt, J. Kühn, Fritz-Haber-Inst./MPG, Berlin-Dahlem, Germany, and H.K. Wagenfeld, Dept. Appl. Phys. RMIT, Melbourne, 3001, Australia

X-ray dispersion corrections have been calculated in the past by several authors using different theoretical approaches. In addition, there are now a number of experimental determinations of dispersion corrections which allow comparison with the theoretical data. A critical issue is the angular dependence of dispersion corrections. Although the theoretical treatment for the calculation of this dependence is correctly presented in I. Waller's paper (Z. Phys. 51 (1927) 213) on the dispersion of x-rays in atoms and has been calculated by H. Hönl (Ann. Phys. (5) 18 (1933) 625) accordingly for the K-shell of atoms, not much notice has been taken by some authors who attempt to calculate this angular dependence. They simply multiplied the oscillator strengths for dipole transitions by the corresponding electron form factor (i.e. the Fourier transform of the electron charge distribution) for the electron in consideration, (Prins, J.A.: Z. Phys. 63 (1930) 477 (formula 9 page 428); Zachariasen, W.H.: Theory of X-Ray Diffraction in Crystals (formula 3.31, page 96) Dover Publ. Inc., New York 1967; James, R.W.: The Optical Principles of the Diffraction of X-Rays (formula 6.47, page 324), Bell and Sons Ltd., London 1962). Waller's formula shows that the angular dependence in the "anomalous dispersion" term is, apart from the Lorentz factor (i.e. the polarization factor), only due to the retardation terms in the atomic transition matrix elements for the in- and outgoing x-rays. Retardation effects are neglected in the dipole approximation since the retardation factors are put equal to one. The "dipole" dispersion corrections are therefore not angular dependent. The second term in a Taylor expansion of the retardation factors corresponds to "quadrupole" transitions.

The corresponding dispersion corrections depend on the scattering angle and the wave length independently. These terms are small relative to dipole terms. One of us (Wagenfeld, H.K.: Theoretical computations of x-ray dispersion corrections. In: Anomalous Scattering, Ed. S. Ramaseshan and S.C. Abrahams, IUCr, Munksgaard, Copenhagen 1975) pointed this out at the conference on anomalous scattering in Madrid (1974) where numerical calculations of anomalous dispersion terms and their angular dependence have been reported; however incorrect numerical results were included in the final publication of the conference proceedings (Hazel, A.C., pp. 48-50 in: Anomalous Scattering, see above). False assumptions concerning the angular dependence can lead to significant errors in some structure determinations. The points mentioned in the manuscript presented in Madrid, however, are well accepted by scientists working in the field of dynamical x-ray diffraction on perfect crystals, and the theoretical data have been, in some cases, experimentally confirmed. We will now present revised numerical dispersion data for different elements, x-ray wave lengths, and scattering angles based on an extended Hönl calculation.