11.7-13 PRECISION MEASUREMENT OF STRUCTURE FACTORS BY DYNAMICAL DIFFRACTION.

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As has been shown (Bonse & Teworte, 1980), a very accurate (i.e. on or below the 0.1 % level) determination of structure factors can be performed by evaluating relative heights and positions of the intensity oscillations on the wings of Laue case rocking curves. These oscillations occur by interference of the two excited wave fields inside the crystal provided that absorption is not too high (i.e. the product of crystal thickness t and absorption coefficient μ is less than 1). Contrary to the well-known Pendellösung method with wedge crystals only a small perfect region of less than 1 x 1 mm^2 is sufficient for our measurements. The method had been tested on silicon (Bonse & Teworte, 1981) and is now being applied to other crystals.

References:

U. Bonse, R. Teworte, J. Appl. Cryst. (1980), 13, 410-416

U. Bonse, R. Teworte (to be published)

11.8-01 PARTIAL COHERENCE OF X-RAYS.

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Profiles of diffraction lines 111, 222, 331 of three kinds of nickel powders were measured in arrangements without and with a crystal monochromator. The high angle monochromator, designed by Čermák (J. Phys. E: Sci. Instrum. (1972) 5, 1124) utilizing the reflection angle 72° selected from the doublet Cu-Kα only 1/5 of the half-width of Kα_1. The sample with narrowest lines was used as a standard for the estimate of diffraction component from measured and instrumental profiles by the Fourier deconvolution. Integral breadths of deconvolutions were calculated also from the relations based on assumptions on analytical shapes of profiles. Statistical analysis shows that (1) in the arrangement with monochromator the pure diffraction profile is systematically narrower (as much as by 50 % of the arithmetic mean of the values in both arrangements), (2) this difference decreases with the increasing reflection angle, (3) the mentioned difference is relatively smaller for the sample with less perfect crystal lattice. This phenomenon was theoretically predicted by Bezirganian (Doklady AN Arm.SSR (1963) 37, 197; Izv. AN SSSR, ser. fiz. (1964) 28, 882) who applied theory of partial coherence of visible light (Born, Wolf (1968) Principles of Optics, Oxford: Pergamon Press) to kinematical theory of the Bragg reflection. Individual atoms in the focus of X-ray tube radiate indepedently and only a finite time. In the first approximation there are finite wave trains of harmonic waves of length $l = \lambda^2/\Delta\lambda$, where λ is the mean wavelength and $\Delta\lambda$ the effective breadth of spectral distribution of the primary radiation. Values $l = 21.97 \times 10^{-7}$ m and $l = 0.62 \times 10^{-7}$ m resp. follow from the mentioned formula for our experimental arrangements with and without monochromator resp. Wave trains reflected for a higher $\Delta\lambda$ on all atom planes in a crystal need not meet in the point of interference. Finite wave trains increase in this way the breadth of pure diffraction profile. Inclusion of the spectral distribution in the instrumental profile and the usual deconvolution cannot be thus sufficient, because the spectrum influences directly the pure diffraction profile. Interpretation of profiles (e.g. an estimate of particle size) may be distorted by appreciable errors. Comparison of structural data obtained in various experimental arrangements may be rather problematic. Space coherence defined by geometry of the arrangement may influence breadth of lines, too. Theoretical predictions of the effect of space coherence were presented by Kuběna (Czech. J. Phys. (1968) B 18, 777, 1233; (1970) B 20, 106). General relation for the interference intensity of partially coherent light (Born, Wolf, ad ibid.) indicates a shift of diffraction lines due to a coherence change. Our experimental results confirm: (4) the centroids of pure diffraction profiles differ in both arrangements as much as by 0.01°, (5) this difference decreases with increasing reflection angle and (6) with broader lines.

11.8-02 WAVELENGTH DEPENDENT X-RAY SCATTERING FROM PYROLYTIC GRAPHITE

By J L Lawrence, Physics Department, The University of St Andrews, North Haugh, St Andrews, Scotland, KY16 9SS.

Using a parallel beam of x-rays, the absolute integrated intensities and the absolute reflectivities from the (001) planes of a crystal of pyrolytic graphite for l=2 and l=4 were measured over a range of wavelength from 0.5 to 1.54 A. The crystal had a mosaic spread of 0.26 degrees.

The Second Order Reflection [l=2]

The measured integrated intensities are only about one-third of the kinematic values, showing that the scattering cannot be assumed to be a kinematic process. Assuming the multiple scattering equation of Zachariasen [Theory of X-ray Diffraction in Crystals, Wiley, New York, 1945, p162] a much closer agreement with the measured data is obtained, the measured values for all wavelengths being slightly greater than the calculated values. The maximum reflectivities were also slightly greater than the calculated values and the proportion of the once, three-times, five-times etc scattered radiation is estimated. The highest integrated intensity and reflectivity occured at 0.710 A.

The Fourth Order Reflection [l=4]

The integrated intensities were about one-third lower than the kinematic values and the integrated intensities and reflectivities were both lower than those obtained from Zachariasen's equation. The maximum integrated intensity and reflectivity occured about 1.0 A.

The absoption factors used throughout the calculations were those of the author [Acta Cryst(1979) A35 p316].