EXAMINATION OF HIGH-ANGLE DOUBLE-CRYSTAL X-RAY DIFFRACTOMETRY (HADOX). By Y. Fujii, Y. Soejima and A. Okazaki, Department of Physics, Kyushu University, Fukuoka 812-81, Japan.

The HADOX method has been used since the seventies in the determination of the temperature dependence of the lattice spacing \(d(T)\) in a relative precision of \(10^{-6}-10^{-7}\), and in the characterization of crystals. In the circumstances where synchrotron X-rays are available, the performance of the method is systematically reexamined for crystals of different qualities. In the figure given below, the experimental arrangement in HADOX is shown. X-rays are successively diffracted by two crystals \(X_1\) and \(X_2\); the Bragg’s law is given by \(d \sin \theta_1 = d \sin \theta_2\) where suffixes 1 and 2 denote \(X_1\) and \(X_2\) respectively. In HADOX, the two arrangements \(A\) and \(B\) can be used, \(X_1\) and \(X_2\) being used as follows:

In \(A\), \(X_1\) as a monochromator and \(X_2\) as a specimen, and in \(B\), \(X_1\) as a specimen and \(X_2\) as an analyzer.

In both \(A\) and \(B\), a change in \(d\) of the specimen crystal can be determined from a change in \(\omega_2\); in \(A\), however, the change in \(\omega_2\) may include contributions from a twist of the specimen holder etc. when the temperature is varied. In \(B\), the X-ray wavelength at the detector varies when \(d_1\) varies. If a conventional X-ray source is used, this may require to use a white X-rays, and the intensity will be a serious problem.

When d1 varies.

For the monochromator we find \(I(0.71 \text{\AA}; \text{Mo})/I(0.71 \text{\AA}; \text{Cu})\) and \(I(0.71 \text{\AA}; \text{Cu})/I(0.71 \text{\AA}; \text{Mo})\). For the graphite monochromator we find \(I(0.71 \text{\AA}; \text{Mo})/I(0.71 \text{\AA}; \text{Cu}) = \exp(2\pi^2 \sin^2 \theta/\lambda^2)\) with \(B=0.5\times 2\). This illustrates a large \(\Delta \lambda/\lambda\) interference with the ADP's. Subtraction of \(I(0.71 \text{\AA}; \text{Cu})\) from \(I(0.71 \text{\AA}; \text{Mo})\) produces a 'monochromatic' Mo-K\(\alpha\) dataset, which fits almost ideal to the structure factor equation obtained in least squares. The Si(111) defined datasets result in \(R_w=3.61\%\), \(R_m=5.04\%\), and \(S=1.79\). These indicators are all larger than the single I(0.71\AA;Mo) measurement. However, the 'monochromatic' model shows ADP's of 0.97 B(0.71\AA;Mo). So B has become practically equal to B(neutron), which means that \(\Delta \lambda/\lambda\) is the driving force behind the hitherto unexplained inequality B(neutron)<B(X-ray).

THE ‘TILTER’: A NOVEL POLARIMETER FOR FAST OPTICAL ACTIVITY MEASUREMENTS IN BIREFRINGENT CRYSTAL SECTIONS. A. M. Glazer, W. Kaminsky, Clarendon Laboratory, Department of Physics, University of Oxford, England.

Optical activity and other gyrotronic effects, when masked by birefringence, can be determined reliably with the so called ‘HAUP’ (High Accuracy Universal Polarimeter) method. If further information is provided from modelling of the phase-retardation \(\phi\). In a previous method (WS-HAUP in the Clarendon Laboratory), the phase-retardation is modelled by changing the wavelength with a high-pressure lamp-monochromator arrangement. In the new ‘TILTER’ system, this modelling is established by scanning through sections of the crystal by tilting it with respect to the wave vector to select different values of the birefringence \(\phi\).

Using a laser as a light source and varying the angular parameters of the setup, which are related to the polarizers and the sample, it is possible to separate the gyrotrony, birefringence, orientation of the indicatrix and parasitic contributions. The time required could be minimized to about 2 minutes for a complete scan which has to be compared with about 24 hours in the case of the previous ‘HAUP’-system in the Clarendon Laboratory.

By scanning across a crystal section and repeating the tilt-scan procedure, it is now possible to produce maps of optical activity within a day’s time. First results show larger variation of optical activity in homogeneous samples than expected. However, twinning and domain-structures are easily analysed by the new technique.

EXAFS laboratory spectrometer was constructed for using in preliminary laboratory studies of catalysts. Standard G-20 goniometer has been adapted with monochromator mounted at the goniometer axis and two especially build proportional counters (with the sample placed in between) on the arm of the goniometer. The radius of the monochromator curvature is changeable during the measurements to keep the setting close to the Rowland geometry. Silicon, about 200mm thick, (110) and (111) oriented crystals were used as monochromators.

Initially the system was used with a standard 1.5 kW sealed-off X-ray tube. The measurements of Fe K\alpha absorption edge performed on Fe foils showed that the data obtained were comparable to synchrotron data but at the significant expense of the measurement time.

To increase the intensity of the measured spectra and shorten the time of measurements the spectrometer was installed on Siemens 18 kW rotating anode with Mo target. As it will be shown the obtained spectra were of much inferior quality than expected from possible lower energy resolution. Therefore a series of long-lasting tests was performed showing how insubstantial the performance of the rotating anode affects the quality of the measurements.

Special attention was paid to the possibility of X-ray focus displacement, total intensity fluctuations and thermal expansion of the rotating anode target itself. As results show the most important are the long term beam fluctuations rising up to 10% of the overall intensity of the emitted X-rays which are highly correlated with minute instabilities the effective interphase voltage of the mains, in spite of the very high stability of all the phases of the main.