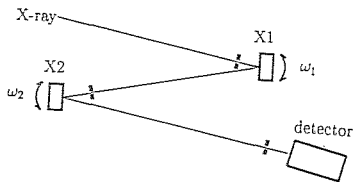
PS01.11.03 EXAMINATION OF HIGH-ANGLE DOUBLE-CRYSTAL X-RAY DIFFRACTOMETRY (HADOX). By Y.Fujii, Y.Soejima and A.Okazaki Department of Physics, Kyushu University, Fukuoka 812-81, Japan

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 to 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 schematically shown. X-rays are successively diffracted by two crystals X1 and X2; the Bragg's law is given by $d_1 \sin\theta_1 = d_2 \sin\theta_2$ where suffixes 1 and 2 denote X1 and X2 respectively. In HADOX, the two arrangements A and B can be used, X1 and X2 being used as follows:

in A, X1 as a monochromator and X2 as a specimen, and
in B, X1 as a specimen and X2 as an analyzer.

In both A and B, a change in d of the specimen crystal can be determined from a change in ω_2. In A, however, the change in ω_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 white X-rays, and the intensity will be a serious problem. Combinations of crystals with FWHM in ω 50, 300 and 500 μrad are examined in connection with the determination of $d(T)$ and the characterization of crystals.



PS01.11.04 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 gyrotropic effects, when masked by birefringence, can be determined reliably with the so called 'HAUP' (High Accuracy Universal Polarimeter) method /1/ if further information is provided from modelling of the phase-retardation /2/. In a previous method (WS-HAUP in the Clarendon Laboratory), the phase-retardation is modelled by changing the wavelength with a high-pressure lamp-monochromater 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 /3/.

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 gyrotropy, 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.

/1/ Kobayashi, J., Uesu, Y., *J.Appl.Cryst.* **16** (1983) 204-211.
/2/ Devarajan, V., Glazer, A. M., *Acta Cryst.* **A42** (1986) 560-569.
/3/ Kaminsky, W., Glazer, A. M., *Ferroelectrics* (1996) in press.

PS01.11.05 MONOCHROMATIC X-RAY INTENSITIES VIA A 'BALANCED' TUBE EXPERIMENT. S. Maes, A. T. H. Lenstra, Department of Chemistry, University of Antwerp (UIA), Universiteitsplein 1, 2610 Wilrijk, Belgium

Diffraction intensities are accurate (reproducable), but unprecise due to the lack of monochromaticity in the incident X-ray beam. For a sealed tube equipment we find $\Delta\lambda/\lambda$ of ~14% and ~3% for a C(002) and a Si(111) monochromator. Routine data collections were made with a Mo-tube using ammonium bitartrate as reference structure. With C(002) as monochromator we arrive at R_u=3.23%, R_w=4.24% and S=1.48; with Si(111) at R_u=3.83%, R_w=4.41% and S=1.28 In both experiments the reflection intensities are the sum of characteristic Mo-K_α radiation and an unavoidable 'white continuum' component. The white continuum has an intensity distribution given by Kramer's formula $I(\lambda) = K Z [E(\text{tube})-E(\lambda)] / E(\lambda)$ So for a fixed tube voltage E(tube) $I(\lambda)$ only depends on the atomic number Z. This enables us to measure the 'white continuum' contribution separately by replacing the Mo-tube by e.g. a Cu-tube. The rather small 0.71Å Cu-tube intensities were measured and resulted in R_u=4.63%, R_w=4.95% and S=1.33 for graphite and R_u=5.89%, R_w=5.98% and S=1.28 for silicon. With Si(111) we find a constant ratio for I(0.71Å;Mo)/I(0.71Å;Cu). For the graphite monochromator we find $I(0.71Å;Mo) \approx I(0.71Å;Cu)\ \exp[+2B' \sin^2\theta/\lambda^2]$ with B'=0.5Å^2. This illustrates that a large $\Delta\lambda/\lambda$-error interferes with the ADP's. Subtraction of I(0.71Å;Cu) from I(0.71Å;Mo) produces a 'monochromatic' Mo-K_α dataset, which fits almost ideal to the structure factor equation exploited in least squares. The Si(111) defined datasets result in R_u=3.61%, R_w=4.04% and S=1.18, which is a significant improvement compared to I(0.71Å;Mo) alone. Subtraction of the graphite datasets eliminates the systematic intensity errors. Refinement yielded R_u=3.79%, R_w=5.09% and S=1.79 These indicators are all larger than the single I(0.71Å;Mo) measurement. However, the 'monochromatic' model shows ADP's ≈0.97 B(0.71Å;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).

PS01.11.06 ON THE USE OF LABORATORY EXAFS SPECTROMETER WITH SIEMENS 18 kW ROTATING ANODE. B.Mierzwa, Z.Kaszkur, J.Pielaszek - Institute of Physical, Chemistry, Polish Academy of Sciences, Warsaw, Poland.

EXAFS laboratory spectrometer was constructed for using in preliminary laboratory studies of catalysts. Standard Θ - 2Θ 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 Kα absorption edge performed on Fe - foils showed that the data obtained were comparable to synchrotron data but at the significant expense of time of measurements.

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 instabilities of the performance of the rotating anode affect 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 each of the phases of the main.