

11.7-20 PHYSICAL ESTIMATION OF X-RAY TRIPLET PHASES IN MOSAIC CRYSTALS - CURRENT RESULTS AND APPLICATION IN CRYSTAL-STRUCTURE WORK. By F. Mo and B.C. Hauback, Inst. for røntgenteknikk, Universitetet i Trondheim - NTH, N-7034 Trondheim - NTH, Norway.

It has been known for over 30 years that simultaneous diffraction of N beams ($N > 2$) in a perfect crystal gives rise to intensity perturbations which are determined by the phases of the beams involved. More recently it has been realized that N -beam phase effects can be significant also in mosaic crystals. We reported our first successful phase measurements with truly mosaic specimens in 1982 (Thorkildsen & Mo, ECM-7, Jerusalem, 1982, Abstr. p.6), and later showed (ECM-8, Liège, 1983, Abstr. p.258) how non-dispersive factors cause broadening of the line profiles, leaving for observation a much reduced phase signal farther away from the exact N -beam point.

The possible usefulness of physical phase acquisition in crystallographic work is limited by several factors, and it is necessary to explore the potential of the method. We have measured triplet phases from an organic, mosaic crystal with unit-cell volume $\sim 3000 \text{ \AA}^3$ using standard X-ray equipment: a 4-circle diffractometer and filtered Mo radiation from a fine-focus tube. Of a selected set of interaction maxima, phase could be assigned to 77%, of which 95% later proved to be correct. The phases were used to solve the unknown structure ($M_r = 298.4$, $Z = 8$) by direct methods. This appears to be the first reported case on the solution of an unknown structure based on experimental phases. Our results also indicate that a standard laboratory set-up may be used to measure phases for crystals with considerably larger unit cells. Moreover, the experimental conditions can be refined in various ways. - A review of theoretical conditions and some important consequences for the experimental strategy will be given. Possible advantages of this method for special types of structure problems will be discussed.

11.7-21 DIRECT OBSERVATION OF BRAGG-LAUE SURFACE DIFFRACTION. By Cícero Campos and Neusa Lopes Sanjurjo, Instituto de Física - UNICAMP - C.P. 6165, 13.100, Campinas, SP, BRAZIL.

Multiple diffraction of X-rays is systematically produced on the Renninger geometry (M. Renninger, Z. Phys., 1937, 106, 141) by making the primary incidence plane coincide with the diffractometer plane, in order to observe the primary scattered intensities. A new scattering geometry is proposed, in which the secondary plane lies on the diffractometer plane. This geometry makes possible the direct observation of the secondary beam surface propagation. As a matter of fact, the primary crystallographic direction should be made parallel to the ϕ axis of the single crystal diffractometer and the ω and 2θ axes should be fixed at $2\theta_B$ for the secondary reflection. Finally, the primary reciprocal lattice point reaches the Ewald Sphere when the sample is positioned on the χ value. The symmetry of the primary direction gives the number of observable secondary beams. Experiments carried out on Si (111) provided the image for the 222, 113, 000 three-beam and 113, 000 two-beam cases. The 100, 246, 000 three-beam case for InGaAsP/InP (100) epitaxially grown film was also observed. The experimental set up and superficial nature of the beam propagation will be discussed.

11.8-1 OBSERVATION OF PHASE TRANSITION ON K_2SnCl_6 SINGLE CRYSTAL BY MULTIPLE DIFFRACTION OF X-RAYS. By C. Campos* and M.M.F. d'Aguiar Neto, Instituto de Física da UFBA - 40000 Salvador, Bahia, Brasil.

The splitting of the coplanar six beam 000, $\bar{2}22$, $\bar{2}24$, 006, 222, 224 multiple diffraction line for the hexahalocomplex K_2SnCl_6 single crystal was directly observed by means of the divergent beam technique. It happens when the specimen undergoes its first structural phase transition (Henkel, W. et al., Z. Phys. (1980) B37, 321) from high to low temperature. The experimental set up using a 6° incident beam divergence is similar to that presented by Chang S.L. (Appl. Phys. Lett. (1980) 37, 819) who also calculated the theoretical angle line splitting. In our case, this angle appears as $\Delta\theta = 0.493 (\Delta a/a - \Delta c/c)$ for $\text{CuK}\alpha$. The obtained value agrees with the change in the lattice parameter given in the literature (Henkel W. et al., Z. Phys. (1980), B37, 321). Measurements were performed at room temperature and right below 263K. At low temperature, the picture for the primary reflection 006 exhibits a line broadening associated with the domain structure acquired by the crystal due to the phase transition process. A posterior similar measurement for the same 006 line reveals that such domain is irreversible.

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Support from FINEP.

11.8-2 EQUATIONS FOR DIFFUSE SCATTERING FROM DISORDERED MOLECULAR CRYSTALS. By Rita Khanna and T.R. Welberry, Research School of Chemistry, Australian National University, CANBERRA, Australia.

General equations have been developed for diffuse scattering due to substitutional and orientational disorder in molecular crystals. Previous studies on disordered molecular crystals have been either qualitative or semi-quantitative and have in general neglected size-effect contributions from static and dynamic displacements. The equations for diffuse scattering from alloys due to Hayakawa and Cohen [Acta Cryst. (1975), A31, 635] were the starting point for our work and they were modified for use with molecular crystals. Although the same basic principals are involved, there are some fundamental differences between diffuse scattering from molecular crystals and metallic alloys. The atomic scattering factors which are used in the analysis for alloys are replaced by molecular structure factors. These are in general complex and rapidly varying functions in reciprocal space and depend strongly on the position vectors of the various atoms constituting the molecule. As the molecules may have different orientations in different sublattices, the molecular structure factors can vary from one sublattice to another. In addition, alloys tend to be basically simple structures of high symmetry whereas molecular crystals often have low-symmetry with complex arrangements of intermolecular contacts.

The modelling of the contribution to diffuse scattering from size-effect distortions is much more difficult to quantify for molecular crystals. There are two types of molecular motions involved. First, the translation of the molecule as a whole gives rise to size-effect terms similar to those for metallic alloys. Secondly, the libration of the molecule about some axis through its centre of mass modifies the molecular structure factor and gives rise to an additional diffuse scattering term.

The derived equations have been applied to the isostructural isomers of dibromo-diethyl-dimethylbenzene, which show very different disorder diffuse scattering. The basic procedures for data analysis and separation of various components of diffuse scattering have been developed.