

11.1-18 X-RAY DIFFRACTION TOPOGRAPHIC OBSERVATION OF THE PARAELECTRIC-ANTIFERROELECTRIC PHASE BOUNDARY IN COPPER FORMATE TETRAHYDRATE. By N. Herres* and H. Klapper, Institut für Kristallographie der RWTH, D-5100 Aachen, Federal Republic of Germany.

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Copper formate tetrahydrate, $\text{Cu}(\text{CHOO})_2 \cdot 4\text{H}_2\text{O}$, is at room temperature paraelectric with space group $P2_1/a$. It becomes antiferroelectric below -38°C by a (mainly) first-order transition. The space group of this phase is reported to be $P2_1/n$, but very weak additional reflections suggest $P1$. The lattice translation c is doubled (e. g. Burger & Fuess, *Ferroelectrics* **22** (1979) 847).

Crystals (3-4 cm diameter) were grown from aqueous solution. (010) and (001) plates (thickness 0.5 - 1 mm) were studied by X-ray topography using the Lang method and $\text{AgK}\alpha$ radiation. A simple cooling chamber with a small temperature gradient due to gravitational thermal convection was applied. Topographs taken at various temperatures around the Curie point show the phase boundary in different positions in the crystal. The boundary, which is visible by strong kinematical contrast originating from inhomogeneous strain in the transition region, is influenced by crystal defects: It preferably follows growth sector boundaries and cracks. For (010) plates (normal to the antiferroelectric axis) both para- and antiferroelectric phase regions have exactly the same 002 reflection position. Topographs of (001) plates in reflection 110, however, reveal an orientational change of the (110) planes across the phase boundary. In the antiferroelectric phase faint fringe contrasts arising from a dense sequence of planar defects (domain boundaries) were observed. Section topographs indicate them to be tilt boundaries. They are visible in non-superstructure reflections and, thus, no (regular) antiphase boundaries. The (only very small) lattice tilt of adjacent domains indicate $P1$ to be the correct space group.

11.1-19 X-RAY TOPOGRAPHIC STUDY OF THE PHASE TRANSITIONS IN LITHIUM AMMONIUM SULPHATE. By H.D. Jennissen, B. O. Hildmann, H. Klapper and Th. Hahn, Institut für Kristallographie der RWTH, 5100 Aachen, FRG.

Lithium ammonium sulphate exhibits two phase transitions: $\text{III}(P12_1/c1) \xrightarrow{-(12^\circ\text{C})} \text{II}(Pc2_1/n) \xrightarrow{-(186^\circ\text{C})} \text{I}(Pcmm)$.

In phase I the tetrahedral Icmm -framework is disordered. It can be described by fourfold splitting of SO_4 , LiO_4 and NH_4 tetrahedra. Phases II and III represent two differently ordered structures giving rise to ferroelectric and ferroelastic domains, resp. (Hildmann, Thesis, Aachen 1980). Large and highly perfect crystals were grown from aqueous solutions. For the X-ray topographic studies a temperature-controlled heating and cooling chamber was used, operating between -60°C and 250°C . Either straight or elliptically shaped isotherms could be introduced into the crystal plate. Plates of various orientations were examined. Series of topographs, taken at various temperatures around 186°C , show the nucleation and growth of phase II inside the phase I-matrix. The phase boundary I-II is visible by strong kinematical contrast arising from inhomogeneous strain due to the misfit of the lattices. Fringe contrast, however, occurs at the boundary in (010) plates. The boundary follows the transition isotherm rather strictly. The significant jump of lattice parameters a and c leads to a relative tilt of the reflecting planes $(\bar{1}0\bar{1})$ directly observable in topographs of (101) plates. Across the boundary I-II the arrangement of tetrahedra is believed to change continuously according to the ordering process and finally to lock in.

In contrast, the boundary II-III is associated with much more stress leading to many cracks in the crystal plates. Since phase III is monoclinic the boundary II-III can clearly be observed in polarized light as well. Under equilibrium conditions the boundary is preferably aligned parallel to $\{101\}$. The structural model of the tetrahedra in the boundary resembles the high-temperature structure I.

11.1-20 PLANAR DEFECTS IN NATURAL BERYLS.

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The beryl crystals $\text{Al}_2\text{Be}_3\text{Si}_6\text{O}_{18}$ from different deposits of the USSR have been studied by the method of X-ray diffraction topography. Save for dislocations, inclusions, needle-shaped channels and growth bands they have fault planes (F.P.). The F.P. are as interference bands systems often adjusted to the growth sector borders $\langle 000\bar{1} \rangle$, $\langle 10\bar{1}\bar{1} \rangle$ and $\langle 11\bar{2}\bar{1} \rangle$ and as single regions and ribbons subject to hexagonal symmetry (Fig. I). Sometimes the isolated F.P. occur limited by dislocations from both sides (Fig. 2). Interference bands possess different frequency and width, and their quantity in the F.P. given depends on the wave length of the radiation applied. The F.P. described appeared to be analogous to the defects shown on the report topographs (Herres, Lang, *J. Appl. Cryst.* (1983), **16**, 47). They should be referred to the stacking faults of deformation origin, connected with the anisotropy of thermal beryl expansion along a and c axes at temperature below 300°C .



Fig. 1. X-ray topograph of a basal slice of beryl. $(10\bar{1}\bar{0})$ reflexion.

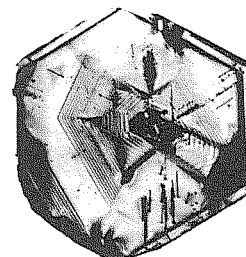


Fig. 2. X-ray topograph of $(11\bar{2}0)$ slice of beryl. $(10\bar{1}\bar{0})$ reflexion.