

15.5-2 HIGH PRESSURE X-RAY DIFFRACTION MEASUREMENTS ON POTASSIUM NITRATE USING SYNCHROTRON RADIATION. By D. M. Adams,¹ P. D. Hatton,² A. E. Heath,¹ and A. Norman.¹

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Potassium nitrate (KNO₃) exhibits complex polymorphism with seven polymorphs in the pressure range 0.0-4.0 GPa. We have studied this material at room temperature as a function of pressure up to 9.3 GPa using energy dispersive powder diffraction at the SERC Synchrotron Radiation source (SRS). We have confirmed the structure of phase IV refined by neutron diffraction at 0.36 GPa (Worlton et al., *Physica* 136B (1986) 503), orthorhombic Pnma, $z = 4$. The compressibility measured in the range 0.3-9.3 GPa is found to be anisotropic with the axial compression ratios $a:b:c = 1.00:0.64:0.50$. The differing merits of both X-rays and neutrons for high pressure studies are discussed.

crystal. We confirmed dislocations grew steadily in whitish part in (a). After the transformation to bcc, the spot in the same reflexion point as in (a) consists of a lot of crystallites almost in the same orientation within several tens minutes as is seen in (b). Internal strains induced in the martensitic transformation from hcp to bcc are easily relaxed mainly by slip. With decreasing temperature, a part of the crystal (b) transform into the hcp structure with very large strain as seen in (c). In the martensitic transformation from the hcp into bcc structures, the (0001)_h plane is converted into the (110)_b plane uniquely. Meanwhile, in the reverse transformation each of six (110)_b planes has equal possibility to transform into the (0001)_h plane. Therefore a hcp crystal transform into a bcc crystal containing some imperfections, but a bcc crystal transform into a large number of hcp crystal grains via the state shown in (c).

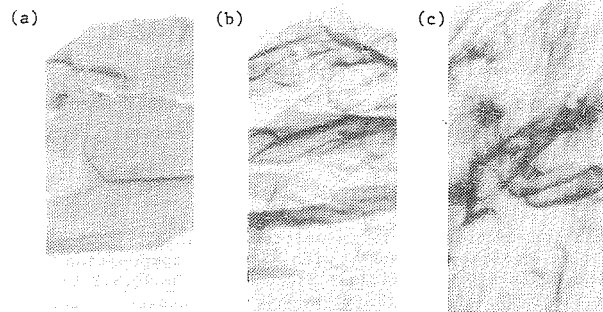


Figure. A part of section topographs of the martensitic transformation in helium-4 from (a) hcp phase at 27.9 atm via (b) bcc phase at 28 atm and (c) mixed state at 28 atm to hcp phase.

15.6-1 OBSERVATIONS OF MARTENSITIC TRANSFORMATION AND SOME BEHAVIORS OF LATTICE IMPERFECTIONS IN HELIUM-4 CRYSTALS BY SR X-RAY TOPOGRAPHY. By Hideji Suzuki, Tetsuo Nakajima* and Izumi Iwasa**, The Research Institute for Iron, Steel, Other Metals, Tohoku Univ., Sendai, * Photon Factory, National Laboratory for High Energy Physics, Tsukuba, ** Dept. of Phys., Fac. of Sci., The Univ. of Tokyo, Tokyo, Japan.

Solid helium is a representative of quantum crystals, because of very large zero point amplitude of about 30 % of lattice spacings. SR X-ray topography is the only feasible method for in situ observation of quantum mechanical behaviours of dislocations in helium crystals at temperatures of mK region. However, we have to solve some inevitable technical difficulties caused by irradiation heating, the small atomic scattering form factor of helium and the uncontrolled orientation of a helium single crystal.

This experiment is at a preliminary stage to develop techniques to get over all difficulties and is limited to temperatures of liquid He-4. We succeeded to take some topographs by TV and nuclear research plates (Ilford L4), in which imperfect structures and their behaviours with temperatures and pressures can be observed.

The sample chamber is made of stainless steel with two Be windows of 1.2 mm thick and 10 mm in diameter. The size of a helium crystal in the direction of incident X-ray beam is 10 mm. Helium crystals in the hcp structure were grown under 35.6 atm by cooling the sample chamber from its bottom. Usually we obtained a large crystal together with a few small crystals at the corners of the sample chamber. A nuclear research plate was set at 58 mm behind the center of the sample chamber, to take transmission type X-ray topographs.

We took section topographs in beam of $0.1 \times 6.5 \text{ mm}^2$ to obtain well-resolved images of sub-structures as shown in Fig. (a), (b) and (c). The topograph (a) is a part of the reflection from (2134) plane of an as-grown hcp

15.6-2 EXPERIMENTAL RESULTS OF TRIPLET PHASE DETERMINATION USING SYNCHROTRON RADIATION. By E. Weckert, H. Bondza and K. Huemmer, Inst. für Angewandte Physik, Lehrstuhl für Kristallographie, Bismarckstr. 10, University of Erlangen, FRG.

Recent progress of experimental triplet phase determination for noncentrosymmetric light atom organic structures with medium size unit cells is reported. The phase information can be extracted from the three-beam profiles of a Renninger psi-scan experiment (K. Huemmer, H. Billy, *Acta Cryst.* (1986), A42, 127-133). The measurements were carried out with a special Psi-Circle-Diffractometer (K. Huemmer, H. Billy in *Crystallographic Computing 3*, (1985) ed. G.M. Sheldrick et. al.) installed at the storage ring DORIS in Hamburg. The six circles of the diffractometer are computer controlled, the angular resolution is 0.001° . There are several advantages using synchrotron radiation. The high energy resolution (Si,Ge(111) two-crystal monochromator) and the small divergence of the incident beam results in an excellent angular resolution of the multiple-beam psi-scan profiles. The total angular width of the three-beam interaction range is smaller than 0.1° . This is mainly due to mosaicity of the crystals, which is indicated in the three-beam psi-scan profiles but cannot be seen in two-beam profiles. The wavelength can be adjusted so that there is no overlap with adjacent multiple-beam cases. But some problems arise from the source instabilities. A number of triplet phases were determined and compared with the calculated values of the known crystal structures. It is shown that the triplet phases can be determined with an accuracy of about 45° comparing the profiles of the two centrosymmetric correlated three-beam cases. Then, the absolute configuration can also be fixed.

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