

05.1-52 NEUTRON DIFFRACTION STUDY OF THE QUADRUPLAR GLASS PHASE OF SOLID DEUTERIUM. By P. E. Sokol, R. O. Simmons, University of Illinois at Urbana-Champaign, Urbana, Illinois, USA, J. D. Jørgensen and J. E. Jørgensen, Argonne National Laboratory, Argonne, Illinois, USA.

We have carried out a neutron powder diffraction measurement on solid deuterium with J=I concentration,  $x=0.49$ , in the temperature range 0.2K-14K. We find that the lattice remains simple hcp ( $P6_3/mmc$ ) at all temperatures. Above 4 K the lattice parameters increase as  $T^{3.6}$ . No deviations in the  $c/a$  ratio is observed in the range studied. Below 4 K an abrupt decrease in the lattice parameter is observed. The lattice constant decreases by 400 ppm between 4 K and 0.2 K. The temperature dependence can, within the experimental errors, be described as linear in T. No discontinuities or anomalies were observed in the lattice spacings, integrated peak intensities, or line widths at the "glass" transition temperature determined by NMR measurements,  $T=0.5$  K. The consequences of this measurement for models of the quadrupolar glass phase of the solid hydrogens will be discussed.

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05.1-54 DO THE RARE EARTH HYDROXIDES SHOW A PHASE CHANGE AT 115 K? K. Ann Kerr, The University of Calgary Calgary, AB, Canada T2N 1N4.

The EPR spectrum of  $\text{Eu}(\text{OH})_3$  doped with  $\text{Gd}^{+3}$  shows a dramatic change in line-shape at about 115 K. To determine whether some feature of the crystal structure was responsible for this change, we determined the cell dimensions as a function of temperature for both  $\text{Eu}(\text{OH})_3$  and  $\text{Y}(\text{OH})_3$  between 97 K and room temperature. In both compounds there is a change in slope near 115 K. Although there have been numerous studies of rare earth oxides at room temperature, we found no low temperature data on this class of compounds.

Three sets of three-dimensional data have been collected from the same single crystal of  $\text{Eu}(\text{OH})_3$  using graphite-monochromatized  $\text{MoK}\alpha$  radiation. Cooling was provided by a cold stream of nitrogen gas obtained by boiling liquid nitrogen. Temperatures of 97, 140 and 190 K were achieved by adjusting the current through a coil in the cold stream. A thermocouple mounted about 3 mm upstream from the crystal showed that the temperature was constant to  $\pm 0.5^\circ$  during each data collection. A six-fold redundant data set (including Friedel pairs) has been collected at each temperature. These have been corrected for absorption and averaged to give 488 reflections in the range  $2.0 < \theta < 50.0$ . Of these 52 had  $I < 2\sigma(I)$  and were considered unobserved.

Refinement is in progress using full-matrix least squares.  $\text{Eu}(\text{OH})_3$  crystallizes in the hexagonal system, with space group  $P6_3/m$  or  $P6_3$ ,  $Z = 2$ ,  $\rho(290\text{K}) = 5280 \text{ gm/cm}^3$ ,  $\mu = 244.6 \text{ cm}^{-1}$ . Cell dimensions are:

	97 K	140 K	190 K
a =	6.3412(7)	6.3419(8)	6.3454(8)
c =	3.6421(6)	3.6425(7)	3.6424(8)
V =	126.83(2)	126.873(3)	127.01(3)

05.1-53 THE INFLUENCE OF THE SYMMETRY OF THE PIEZOELECTRIC CRYSTAL ON THE LOWE ACOUSTIC WAVES. By G.G.Kessenikh, L.A.Shuvalov, V.N. Lujbimov, V.V.Philippov. Institute of Crystallography, Academy of Sciences of USSR, 117333, Moscow, USSR.

The Lowe acoustic waves (A.A.Oliner. Acoustic surface waves, New York (1978), p.47) have been analytically studied for two systems: 1) the piezoelectric substrate loaded by the isotropic layer (authors, Kristallografija (1982) v.27, p. 437-441) 2) the isotropic substrate loaded by the piezoelectric layer (authors, Acusticheskii Zh., (1984) to be published-). The piezoelectric crystals of classes 6, 4, 6mm, 4mm, 622, 422 were considered for both systems. The dispersion equations for phase velocity as a function of the normalized wave vector have been written in an analytical form.

It was shown that the Lowe waves exist both for soft and hard layers. For the case of an isotropic layer on the piezoelectric substrate for 6, 4, 622, 422 classes was obtained the silent zone, where the Lowe wave cannot exist up to the critical thickness of layer. This thickness is proportional to the electromechanical coupling coefficient. Silent zone decreases and then vanishes at about phase transition temperature, 6-- 6mm for example.

The transverse acoustic displacement of the Lowe wave is coupling with the elliptical electric field.

05.1-55 X-RAY AND NEUTRON STUDY OF DISORDER IN  $\text{Ni}(\text{NH}_3)_6(\text{NO}_3)_2$ . By A. Hoser, K. Vogt and W. Prandl, Institut für Kristallographie der Universität Tübingen, West-Germany.

Single crystal investigations made at room temperature confirmed the  $\text{Fm}3m$  space group. As the  $\text{NO}_3$  and  $\text{NH}_3$  molecules occupy sites of symmetry ( $43m$  and  $4mm$  respectively) much higher than their own symmetry, they must be orientationally disordered. The simple model of the  $\text{NO}_3$  group which we used for the x-ray data refinement ( $R_w = 0.065$ ) assumes four equivalent orientations (each perpendicular to the 3-fold axis) forming a tetrahedron together. The H-atoms positions were calculated from the geometry and not refined. After applying the rotational structure factor formalism proposed by Press, Hüller (Acta Cryst. (1973) A29, 252) and extended by Prandl (Acta Cryst. (1981) A37, 811) to the  $\text{NO}_3$  group, the R-factor dropped to 0.027. The radius of the sphere of O-atoms corresponding to the N-O distance becomes  $r$ , close to its correct value and is 1.255(7) Å. The electron density distribution has additional maxima on the 4 axes on this sphere. They indicate how the reorientation of the  $\text{NO}_3$  molecules takes place. The results of the neutron data refinement fully confirm this model. On the other hand we could not find a unique rotational potential (Vogt, Prandl, J. Phys. C (1983) 16, 4753) of the  $\text{NO}_3$  group neither from x-ray nor from neutron data. The reason for this could be rotation-translation coupling ignored by us or more likely dynamical hydrogen bonding which makes the assumption of a time independent rotational potential less valid.