13.1-3 SINGLE CRYSTAL NEUTRON DIFFRACTION STUDY ON HEAVY AND NORMAL ICE VI AT 0.9GPa. By W.F. Kuhs, D. Londono, Institut Laue-Langevin Grenoble, J.L. Finney Birkbeck College London and H. Ahabahs Mineralog. Institut, Marburg.

More than 10 crystalline phases of ice are known to exist as a function of pressure and temperature. They have considerable interest for our understanding of hydrogen-bonded systems and water-water interactions. Most of the ice phases are formed under pressure : Due to experimental restrictions all neutron diffraction studies have been performed on powders and/or on samples recovered to ambient pressure. We report on the first in-situ single crystal neutron diffraction study, on ice VI at ~0.9GPa. A sapphire anvil pressure cell was used which allowed optical control during the (in-situ) crystal growth. The crystals finally obtained had a volume of ~2mm^3. Data were collected on 2 samples at a neutron wavelength of 0.85A on the fourcircle diffractometer D9 of the ILL. The intensity data were corrected for sample and cell absorption. An isotropic correction for thermal diffuse scattering was applied using the known mean longitudinal and transversal velocities of sound. The structures were refined to weighted R-factors between 2 and 3% (on F). The structure model included higher order terms to describe the deformations of the atomic densities due to the molecular disorder. Undoubledly the oxygen atoms are displaced opposite to the bisectrix of the water molecule. When this disorder is taken into account the O-H bondlength becomes ~0.97A in very good agreement with the values observed in ordered ices. A similar study on H_2O ice VI is planned, in which a position sensitive detector should not only improve the statistical quality of the data, but also help to assist in eliminating spurious peaks from the sapphire anvils.

13.1-4 D2B, A NEW VERY HIGH RESOLUTION NEUTRON POWDER DIFFRACTOMETER AT ILL GRENOBLE. A.W. Hewat, ILL, BP156X, 38042 Grenoble FRANCE

The latest results on the D2B high resolution neutron powder diffractometer on the ILL high flux reactor will be presented, together with a description of the machine and its associated furnaces, cryostats and pressure cells.

The machine has been designed for Rietveld refinement of relatively complex structures, such as zeolites, from powder data alone. Intercalated hydrogenous materials can be located relatively easily with neutron diffraction.

The precision of the structural parameters rivals that of good quality single crystal work, so that chemically significant information can be obtained. For example, the Zachariason / Brown-Shannon effective charges can be determined, and electronic ordering examined, in mixed valence compounds such as iron silicates. Apart from the fact that samples can be real materials rather than single crystals, the speed of data collection means that structures can be examined as a function of temperature (or pressure).

Resolution of 0.0005 in d-spacing has been obtained by using extreme backscattering together with Soller collimators of only 5 minute divergence. Data collection is rapid on the high flux reactor due to the very large composite focussing monochromator and the 160 degree multidetector.

D2B will be complemented by similar very high resolution x-ray powder diffractometers on the European Synchrotron at the same Grenoble site.

13.1-5 REMARKS ON ASYMMETRIC BRAGG REFLECTION. By W. Treimer and G. Hildebrandt, Department of Mathematics/Physics of the Technische Fachhochschule Berlin and Fritz-Haber-Inst. der MPG, Berlin.

Asymmetric Bragg reflection changes the ray geometry of the exit beam and the dynamical properties of diffraction. Its use for topography or beam preparation is commonly known. With the help of two successive asymmetric Bragg reflections a two dimensional enlargement of a factor of 25 was already realized (Boettinger W. et al., Rev.Sci. Instr. 50, 1979,26). This geometrical effect as well as the altered properties of the exit beam i.e. its collimation after Bragg reflection (narrowed or enlarged) may be compared with light lenses.

Another less known effect is the shift of the center of the diffraction pattern due to asymmetric Bragg reflection (see e.g. Matsushita, T. & Hashizume, H., in: Handbook on Synchrotron Radiation, Vol.1, North Holland Publ. 1983,261). In the case of neutron diffraction this shift could absolutely be measured and used to determine the coherent scattering length b_c of Silicon with an accuracy comparable with standard methods (Treimer, W. & Berger, G., Phys.Lett. A110,1985,173). The shift $\Delta\Theta_s$ is proportional to λ^2 and to (1+1/b), where $b=\sin(\Theta_B-\alpha)/\sin(\Theta_B+\alpha)$ the asymmetry factor and α the angle between crystal surface and lattice planes. This "wavelength" dependent shift was already tested with white neutrons (Treimer,W.: Final report of the BMFT-supported project 1983-1985) where the experimental conditions had to be kept rather coarse due to intensity problems. Only the 111 reflection could be separated from the shorter harmonics -333,444 etc.- which appeared as a single peak. In the case of x-ray diffraction, the shift $\Delta\Theta_s$ is much more pronounced, mainly due to a larger "atomic scattering factor". Assuming a Bragg angle $\Theta_B=45^o$ and an asymmetry angle $\alpha=44,5^o$, the shift $\Delta\Theta_s$ is large enough to separate harmonics up to high orders from each other (e.g. the peaks of the 888 and 999 reflections are still apart more than 4 sec of arc, or the 10 10 0 and 12 12 0 more than 6"). Using these properties, a double crystal arrangement consisting of a symmetric monochromator and an asymmetrically cut analyzer crystal will work as a tunable, harmonic-suppressing perfect synchrotron monochromator. Experiments on this subject are already in progress.