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21-Crystallography at Non-Ambient Temperatures and/or Pressures; Phase Transitions

21.01 – High Pressure Crystallography

MS-21.01.01 HIGH-PRESSURE X-RAY DIFFRACTION ON LABORATORY SOURCE. By Y. Akahama and H. Kawamura*, Faculty of Science, Himeji Institute of Technology.

An image plate (IP), which is a digitally readable x-ray photograph with high sensitivity and high dynamic range, is very useful for high-pressure x-ray experiment. The application of the IP system in conjunction with a synchrotron radiation source with the high intensity and low divergence enables us to accurately refine atom positions even in complex, low symmetry structures under high pressure (Fujii, et al. 1989). In practice, however, the advantages of a synchrotron source lead to intense competition for experimental time on central facilities. It is, thus, desirable to develop a laboratory-based IP system in order to remove the limitation of beam time.
Figure 1 shows a block diagram of our laboratory-based IP system. A collimated beam of Mo Ka x-rays (60 kV x 90 mA) monochromatized by a graphite single crystal (001) impinges on the sample in a DAC through a double pin hole collimator. Diffraction lines on the IP, which is disc shape with a radius of 100 mm, is read out by a r-θ-type scanner.

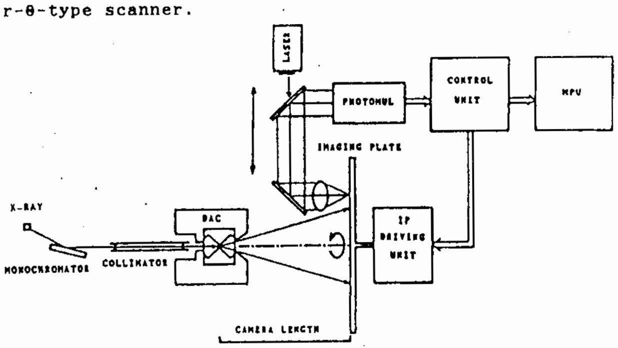

Fig.1 Block diagram of high pressure x-ray diffraction system

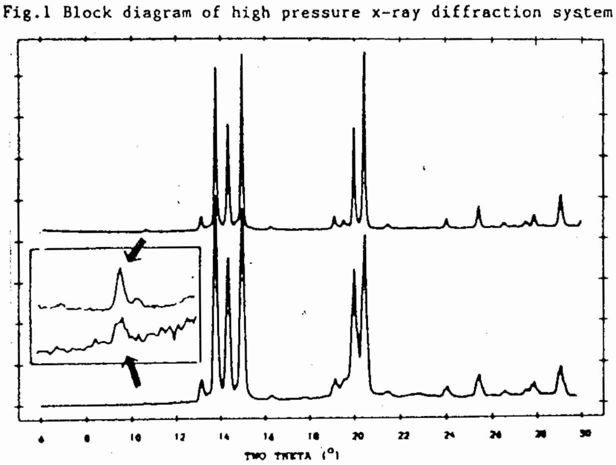

Figure 2 displays a comparison between data taken at the SRS Daresbury and our laboratory on the same sample, InSb at 3.5 GPa (Hatton, et al. to be published). The top trace was obtained in 2.5 hours using the wiggler radiation at station 9.1 at the SRS. The lower diffraction pattern was obtained in 45 minutes at our laboratory. The resolution and signal-to-noise ratio are clearly comparable with the laboratory data displaying only a slightly increased peak width.

REFERENCE

Fujii, Y., Hase, K., Ohishi, Y., Fujihisa, H., Hamaya, N., Takemura, K., Shimomura, O., Kikegawa, T., Amemiya, Y. and Matsushita, T., Phys. Rev. Lett., 63, 536(1989).

MS-21.01.02 ANGLE-DISPERSIVE STUDIES WITH AN IMAGE-PLATE DETECTOR. By M.I. McMahon* and R.J. Nelmes, Department of Physics, The University of Edinburgh, U.K.

Angle-dispersive powder-diffraction techniques, coupled with an image-plate area detector, have been developed at SRS Daresbury for crystal-structure studies at high pressure. Integration of the 2-dimensional diffraction patterns improves the powder averaging and greatly reduces the background noise in the integrated profile - enabling very weak peaks to be observed. The patterns also have high resolution, with a typical FWHM of 0.06° (2θ). The experimental and data-analysis techniques will be described, and illustrated using data from recent studies of II-VI, III-V and group IV semiconductors.

MS-21.01.03 SYNCHROTRON-RADIATION STUDIES WITH DIAMOND - ANVIL CELL AND LARGE VOLUME APPARATUS. By N. Hamaya, Department of Physics, Ochanomizu University, Japan.

Recent developments of high-pressure diffraction experiments at the Photon Factory, KEK, will be briefly reviewed with special emphasis on accurate crystal structure determination, which is now feasible by using reliable intensity data collected with either a combination of the DAC and an Imaging Plate, or a large volume apparatus, MAX80, operated in an angle-dispersive mode. The former technique has been applied to the study of fcc-distorted fcc phase transition in Pr and La. With the aid of a Rietveld analysis, we have identified the crystal structure of the distorted fcc phase to be R $\bar{3}$ 2/m (D^5_{3d}) with Z = 8, determined its positional parameters as a function of pressure, and suggested the association of phonon softening with this transition. In the case of La, its novel re-entrant phase transition scheme, fcc-rhombohedral-fcc, is quantitatively followed in the variation of positional parameters with pressure. Angle-dispersive diffraction on MAX80 is usually carried out using monochromatic high-energy X-rays with E>40 keV in order to avoid large absorption in the material surrounding the sample. High angular resolution to $\Delta 2\theta$ = 0.05° FWHM and high-S/N ratio capability has enabled us to study a complex crystal structure of Bi and MnF_2 at high pressures and high temperatures.

