High Pressure VI
Data Collection and Analysis

MS18.06.01 THE USE OF IMAGING PLATES IN LABORATORY HIGH PRESSURE X-RAY POWDER DIFFRACTION EXPERIMENTS. J. Haines, J.M. LÇger C.N.R.S., Laboratoire de Physico-Chimie des MatÇriaux, 1, Place Aristide Briand, 92190 Meudon, France

The application of imaging plate (IP) detectors to high pressure diffraction experiments in the laboratory has led to significant increases in sensitivity, resolution, and intensity accuracy as compared to older techniques using film or energy- dispersive diffraction. The integration of two- dimensional IP data results in improved powder averaging and a much higher signal to noise ratio. The data now obtained from both sealed sources and rotating anodes allow for crystal structure refinement using the Rietveld method. A simple, low-cost laboratory IP system will be described along with the data treatment necessary to obtain refinable profiles. Examples of structure refinement of some high pressure metal dioxide phases will be presented. In particular, data obtained for the cubic, high pressure phases of the rutile-type dioxides permitted the oxygen positions to be refined, thereby demonstrating that these phases are not fluorite structured, but rather have a modified fluorite structure. Potential improvements to the technique and future prospects will also be discussed.

MS18.06.02 SOFTWARE FOR HIGH PRESSURE CRYSTALLOGRAPHY USING AREA DETECTORS. A P Hammersley, D Hausermann, M Kunz, and C Reul Wassilew, Synchrotron Radiation Facility, BP 220, 38043 Grenoble Cedex, France

Accurate processing of area detector data is of fundamental importance in High Pressure crystallography. The quality of structural information depends heavily on image processing for distortion and geometrical corrections, and for integration. Detector evaluation, experiment calibration, and scientific data analysis, all share common needs and benefit from versatile software.

Area detector evaluation has emphasized the importance of calibration and correction even for systems with very little distortion. The ultimate test of detectors is the quality of scientific results which they produce, but since this a "convolution" of a large number of factors, many external to the detectors, simple characterisation techniques are of value. To assist in efficient exploitation of experimental facilities automated calibration of beam centre, detector non-orthogonality, sample to detector distance, and wavelength using calibrants is highly desirable.

The integration of 2-D data to 1-D "2-theta scans" is well understood, but the realities of experiments and samples means that flexibility and user interaction are of much value in extracting structural information from the data. Non-ideal powders, with texture, or maybe deviatoric stress make interpretation more complicated and demand new techniques allowing arbitrary selection and integration of different azimuthal regions.

As well as providing sophisticated functionality, software ideally needs to be easy to understand and operate, and provide a large amount of feed-back to the user.

MS18.06.03 2–D ANALYSIS OF NON–IDEAL POWDERS USING AN IMAGE–PLATE DETECTOR S. A. Belmonte, R. J. Nelmes and M. I. McMahon, Department of Physics and Astronomy, The University of Edinburgh, Edinburgh, EH9 3JZ, Scotland.

Structure determination of materials using powder diffraction requires the accurate determination of peak positions and relative peak intensities. Positions and intensities can both be strongly affected by deviations from an ideal powder, which are commonly found in high–pressure studies—such as preferred orientation, deviatoric stress and stacking faults.

Preferred orientation (PO) alters relative peak intensities, sometimes quite dramatically. A general 3–d model has been developed to describe how the PO of a sample is manifested in intensity variations around the rings of a 2–d powder pattern, depending on the orientation of the PO axis with respect to the incident and diffracted beam directions. This, combined with the use of a 2–d image–plate detector to record almost complete Debye–Scherrer rings, opens up a new approach to PO analysis. It will be shown that it is possible in this way to obtain a quantitative description of the PO in a sample—and thereby apply corrections to measured intensities—prior to, and independently of, structure determination and refinement.

The 2–d nature of the analysis will be emphasised. A brief description of the techniques involved in collecting suitable data will be given, as well as illustrations of the analysis of some typical examples. Other 2–d effects caused by non-ideal powders will be presented briefly.

MS18.06.04 SINGLE CRYSTAL STRUCTURE DETERMINATION AT PRESSURES EXCEEDING 20 GPA WITH SYNCHROTRON RADIATION. Stefan Werner, Julie Kim-Zajonz, Jürgen Wittlinger, Heinz Schulz, Institut für Kristallographie und Mineralogie, Ludwig-Maximilians-Universität München, Theresienstr. 41, D-80333 München, Germany

Employing a newly developed diamond anvil cell a pressure of 31 GPa was achieved in an angle dispersive single crystal X-ray scattering experiment on ruby and a pressure of 21 GPa in an experiment on the amorphisation of $ZnCr_2S_4$-spinell.

The principles of this cell design are presented. The cell is loaded with the single crystal specimen, a small ruby crystal as pressure calibrant and cryogenic argon as (quasi)hydrostatic pressure transmitting medium.

Scattering experiments were performed at beamlines D3 and F1 at HASYLAB / DESY.

The centering of the cell on the diffractometer is controlled by the profile of the primary beam which has passed the pressure cell.

A pneumatically driven collimator on the secondary beam was developed to exclude X-rays scattered by the beryllium backing plate facing the detector. Systematic falsifications of intensity data caused by scattering of the diamond single crystals are calculated in advance and are detected by measuring reflection intensities at different ψ-angles.