

Angle dispersive diffraction with diamond anvil cells

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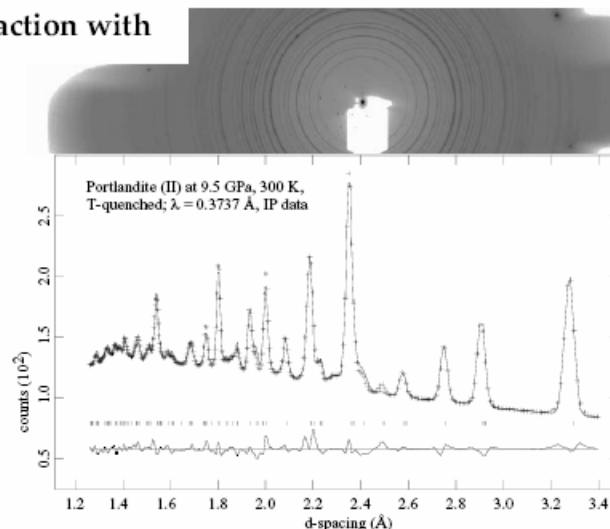
Over the past year we have conducted a number of studies using angle dispersive diffraction at high pressures using diamond anvil cells. Used many years at the CHESS B1 station, energy dispersive diffraction, originally demonstrated for diamond anvil cell synchrotron work by Buras [1], provides a simple method for collecting a wide range of d-spacings from a sample with a limited x-ray 2-theta. This technique has been a mainstay of powder diffraction at high pressure for many years in spite of the limited resolution of the solid state detectors employed. This limitation makes it difficult or impossible to resolve the closely spaced diffraction lines seen in many experiments at moderate pressures. In addition, the detector sees only a small portion of the diffraction cone, exacerbating texturing problems normally seen in diamond anvil cell experiments.

Angle dispersive diffraction, while not a new technique, offers a new way of solving more complex structures at high pressure [2]. This has come about because of the availability of both image plates, providing two dimensional image data in electronic form, and inexpensive computers with enough memory and disk space to routinely handle large data sets. The experimenters collect Debye-Scherrer rings from high pressure samples and read the data into a computer. Figure 1 shows an example of an experiment involving a solid-solid phase transition. Since large crystallites form during the transition the pattern becomes spotty: an energy dispersive experiment would miss this important aspect and

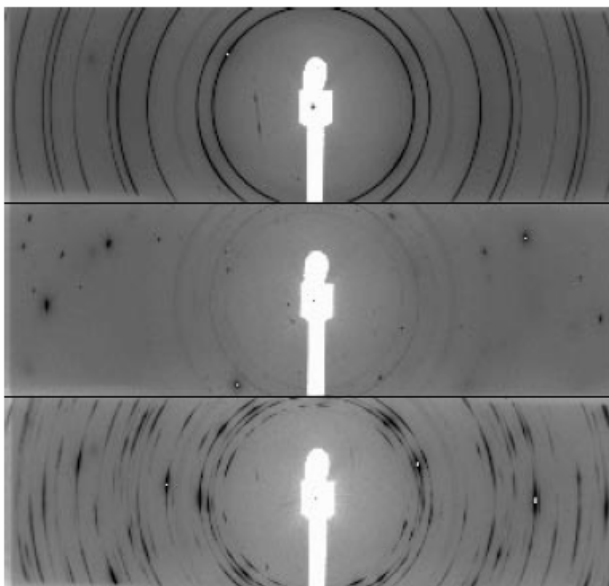
(Figure 1) Yogesh Vohra's group from the University of Alabama at Birmingham have studied Cerium metal and Cerium Thorium alloys [5] recently using angle dispersive diffraction. Cerium is a classic example of a material showing an electronic transition: at 0.7 GPa a FCC to FCC phase transition takes place with a volume collapse of 16%. This study concentrates on the post volume collapse phases and on crystal grain growth during the phase transformation to the alpha Uranium structure. This is the first observation of such a phenomenon in metals. (top) Before transforming to alpha Uranium (4 GPa), (middle) just after the transformation (6 GPa), and (bottom) beyond the transformation (8 GPa).

might incorrectly conclude the material becomes amorphous. The next step in the analysis involves using software to transform the images into 2θ-intensity plots. This allows the user to find the structures and lattice parameters of best fit. Figure 2 shows the results of fitting a new high pressure phase of Portlandite found during a recent run on D-line.

Currently at CHESS we have 3 software packages installed for analyzing powder diffraction data taken with image plates: 1) IMP, developed at CHESS, transforms image files (roughly 10 megabytes) into an equivalent diffractometer scan. IMP can output either an ASCII file for your favorite plotting package or else



(Figure 2) Angle dispersive diffraction at D1 late last fall yielded a new high pressure phase of $\text{Ca}(\text{OH})_2$ [6]. In this experiment Portlandite prepared at 9.5 GPa and 500C in a diamond anvil cells gave a refractive x-ray spectrum at high pressure and room temperature. The program "IMP" reduced the image on the top to a histogram allowing the program "GSAS" to refine the fit to the P21c monoclinic space group with $a=4.887(2)\text{\AA}$, $b=5.834(2)\text{\AA}$, $c=5.587(2)\text{\AA}$, $\beta = 99.74(2)$ degrees. The bottom shows the fit plotted as the solid line and the data as "+" marks. This experiment was first tried unsuccessfully using energy dispersive diffraction.



a GSAS file for further analysis (see below). 2) PLATYPUS developed at the University of Edinburgh by Ross Piltz [3] serves a similar function as "IMP". 3) GSAS developed at Los Alamos National Laboratory by Allen Larson and Robert Von Dreele [4]. This comprehensive package allows the user to use Rietveld refinement to analyze the powder patterns.

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