Getting the Most Data out of Your High-Pressure Experiment

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Within the last decade high pressure studies have received significant increase of interest. Present typical applications range from the investigation of high-pressure polymorphism of solid-state organics as part of the pharmaceutical drug development to the study of rocks and minerals with an applied pressure of up to 50 GPa.

A major challenge in the field of high pressure crystallography is the acquisition of data of sufficient quality and completeness for a successful structure determination, especially for low symmetry samples. The volume of the reciprocal space that is accessible in the high-pressure X-ray diffraction experiment is constrained by the shape and size of the diamond anvil cell (DAC) used, and is mainly determined by the opening angle of the DAC. In addition, the acquired data often suffer from absorption effects caused by the diamond anvils of the DAC, from high background due to scattering at the gasket as well as from strong reflections of the diamonds used in the DAC overlapping with those from the sample. Further, the samples are usually very small with diameters in the range of 0.2 mm or smaller, sometimes just as small as a few µm.

The structure determination on very small samples has become much easier with the advent of high-brightness microfocus sources, as the increased flux density simply improves the diffracted intensity. The small beam cross section improves the signal-to-noise ratio significantly by minimizing the background resulting from the scattering at the gasket. The availability of strong home-lab X-ray sources with radiation harder than Mo-Kα, such as the Ag-IµS and the Indium METALJET, not only improves the signal-to-noise ratio in a high-pressure experiment by minimizing absorption effects and parasitic scattering but facilitates structure solution and refinement of high-pressure phases. The harder radiation compresses the q-space and, hence, paves the way for data with a higher resolution and an increased completeness and multiplicity.

This presentation will be reviewing recent advances in hardware development, such as X-ray sources, detectors and accessories, and will highlight the latest improvements in the APEX3 software, which help in tackling the problems with data acquired in high-pressure experiments.

Using a D8 VENTURE system, we will be demonstrating the advanced hardware capabilities and processing methods based on data from the monoclinic polymorph of the sulfonium ylid. Further, we will be showing how to increase the completeness of high-pressure experiments by mounting multiple samples in a diamond anvil cell and measuring and processing data concurrently.

The high flexibility of the D8 VENTURE design can be further expanded by adding enhanced features typically only available at synchrotron facilities for studying tiny mineral crystals enclosed in diamond anvil cells. These features include highly accurate, motorized sample positioning and the ability to monitor the intensity of the X-ray beam passing through the diamond anvil cell, as well as an extension for online pressure measurements based on ruby fluorescence. This makes the D8 VENTURE system “a little synchrotron at home”.