Using High-pressure Diffraction to Design and Understand Functionality

Diamonds are crystallographer’s best friends: The new IµS DIAMOND II with diamond hybrid anode technology and high-brightness cathode for very high intensities

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Traditionally, high-pressure crystallography has been associated with the study of minerals from within the earth’s crust and with pressure-dependent studies of inorganic functional materials [1, 2]. More recently, pharmaceutical development has benefitted from organic solid-state chemistry and its perspectives on structure and function in biologically relevant molecules, with high-pressure polymorphism being of critical importance [3]. Of growing interest are also studies on the effects of pressure on drug formulations, since many solid drugs are exposed to mechanical stress during manufacturing, and studies under very high pressure for understanding of the complex mineralogy of the inaccessible deeper interiors of Earth and other planets [4].

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 small and weakly diffracting samples of low symmetry. The volume of the reciprocal space that is accessible in the high-pressure X-ray diffraction experiment is mainly constrained by the opening angle and the overall shape of the Diamond Anvil Cell (DAC) used. 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 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, making the use of strong X-ray sources and sensitive detectors mandatory.

The availability of strong microfocus X-ray sealed tube sources with radiation harder than Mo-Kα, such as the Ag-IµS DIAMOND II, 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.

Our latest IµS family member, the IµS DIAMOND II microfocus sealed tube, delivers intensities very comparable to microfocus rotating anodes, yet maintaining all the comfort of sealed tube systems. It combines our innovative diamond hybrid anode technology [5], that uses diamond as a heat sink in the anode for an increased power loadability [6], with a new cathode with a very high emission rate, resulting in an impressive twofold gain in intensity compared to the previous generation.

This presentation will be reviewing the latest innovations in microfocus sealed tube X-ray sources and recent advances in hardware development, such as detectors and accessories, and will highlight the latest improvements in the APEX4 software, which help in tackling the problems with data acquired in high-pressure experiments.

References: