Solving structures from complex multigrain, multi-phase samples at extreme conditions

Abstract:

Laser-heating of samples in the diamond anvil cell is often used to trigger re-crystallization and crystal-growth processes that may also be accompanied by phase transformations and/or chemical reactions. The resulted X-ray diffraction patterns usually appear “messy”: a mixture of diffraction rings with texture and a chaotic cloud of spots merged with reflections of the high pressure medium and pressure standards. In the great majority of these cases so far, the diffraction data of such complex multigrain, often multi-phase, samples are integrated to one-dimensional patterns and are treated as powder samples. Such approach could be proven ideal for samples that are consisted of readily known compounds and known structure types; data processing and interpretation seems easy and straightforward. But what if none of the known phases matches our pattern? What if our 1D patterns consist of so many peaks (overlapping and/or broad) that basically any phase in the literature could be fitted? And what if this one weak satellite reflection that we observed in our patterns is actually a sample reflection from a phase that we are about to miss? In this talk we will show how one could extract the outmost information possible from such complex samples by using single-crystal X-ray diffraction techniques. This data approach allows explicit structure solution of complex compounds, detection of small structural distortions, accurate determination of displacement parameters and chemical characterization of novel materials. We will review the sample and DAC preparations that are necessary prior to such an experiment, describe the data collection procedures, and discuss the data processing with various software. A few examples, on carbonate minerals and various metal oxides, but also weakly scattering compounds, such as CO_2, will be presented in order to demonstrate not only the challenges but also the great merits of the method.