Single-crystal X-ray Diffraction at Extreme conditions

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The advantages of using single crystals over powdered samples in X-ray diffraction experiments are well known [1]. Analysis of single-crystal X-ray diffraction data has traditionally allowed us to obtain explicit solutions of complex structures, detect small structural distortions, retrieve accurate displacement parameters as well as provide chemical characterization of new materials. The single-crystal X-ray diffraction method is becoming more and more appealing in the high-pressure research community nowadays [2]. It is now possible to study in great details the crystal structure, physical and chemical properties of minerals and materials, important for materials science, even in the megabar pressure range using the diamond anvil cell [3]. Even at high pressure, where the coverage of the reciprocal space is restricted by the diamond anvil cell design, single-crystal X-ray diffraction data provide more information than the one-dimensional diffraction patterns collected from powdered samples.

Here we review the sample and diamond anvil cell preparations that are necessary prior to a single-crystal X-ray diffraction experiment. In addition we describe the data collection procedures at the GSECARS beamline (sector 13), and we discuss the data processing using various software. A few examples on carbonate minerals and 3d transition metal oxides are presented in order to demonstrate not only the challenges but also the advantages of using single crystals for solving the structures of complex high-pressure polymorphs or novel compounds, as well as to better constrain the compressibility and the high-pressure structural evolution of known compounds.

[1] P. Dera **(2010)** *All different flavors of synchrotron single crystal X-ray diffraction experiments.* High-Pressure Crystallography, Springer, Dordrecht, p11-22.
[2] T. Boffa-Ballaran et al. **(2013)** *Single-crystal X-ray diffraction at extreme conditions: a review.* High Pressure Research, 33, p453-465.
[3] M. Bykov et al. **(2018)** *Synthesis of FeN_4 at 180 GPa and its structure from a submicron-sized grain.* Acta Crystallographica Section E, 74, p1392-1395.