Oral presentation

A new sapphire capillary pressure cell for phase mapping at SNBL BM01, ESRF

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Diamond anvil cells (DAC) are the primary pressure cell design that has dominated high pressure science when kbar pressures are required. They have made data collection (both single-crystal and powder) routine; however, they are not without their limitations. Fine control of pressure is difficult to achieve and measure accurately, particularly at lower pressures (> 1kbar) where in softer molecular systems interesting phase behaviour or compressibility can occur. Currently there is a relative paucity of pressure cell designs made specifically for this lower pressure regime.

Previously a sapphire capillary cell (SCC) was developed for single crystal diffraction measurement at the small molecule beamline I19 at Diamond Light Source. [1] This system used a closed ended sapphire capillary as the pressure vessel for a maximum theoretical pressure of 1500 bar into which a single crystal could be inserted. The pressure generating setup allows for very fine pressure control and measurement (ca 1 bar) both on increasing and decreasing pressure, and the ability to change the hydrostatic medium. This system is seeing increasing interest for high-quality pressure dependent single crystals studies.

Here I present an evolution of the SCC to adapt its use for powder diffraction at the Swiss-Norwegian Beamline BM01 at ESRF. The key development is the pursuit of device to quickly and accurately map phase space with variable temperature and pressure. The large area detector of BM01 and synchrotron radiation reduce collection times to a few seconds and with near instantaneous reaction to pressure isotherms are quick to collect. Sub ambient temperatures are controlled with a Cryostream down to the freezing point of the hydrostatic medium and a custom radiative furnace allows efficient heating to the boiling points of most hydrostatic mediums. The cell allows access to a pressure and temperature range that this is crucial for the burgeoning barocaloric field where pressure driven phase transitions give large uptakes in energy.

Described here will be the technical aspects of the SCC construction, sample preparation, data acquisition and processing tools that have been developed at BM01 to efficiently handle the large volumes of data along with real world examples.

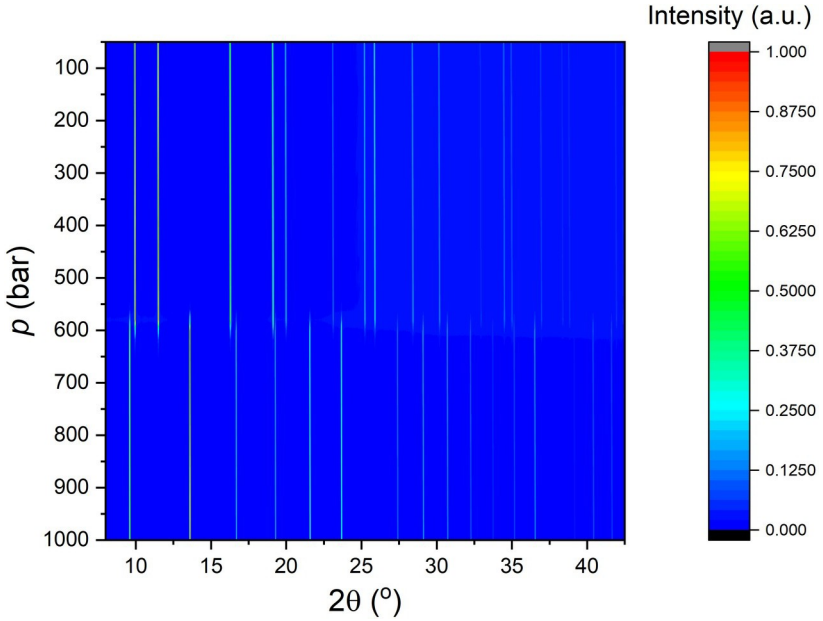

Figure 1. Example of and isotherm showing a first order phase transition on compression from ambient to 1000 bar collected using the SCC.

[1] McMonagle, C. J., Allan, D. R., Warren, M. R., Kamenev, K. V., Turner, G. F. & Moggach, S. A. (2020). *Journal of Applied Crystallography* **53**, 1519-1523.