A new sapphire capillary cell: a moderate pressure cell for powder diffraction at The Swiss- Norwegian Beamlines at ESRF

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Lattice expansion on heating and compression under pressure are common feature of all the solids. Due to structural complexity met in many functional materials such as MOF, organic ferroelectrics and many others, structural response on external stimuli is an intricate interplay of atomic re-arrangements, displacements, rotations and deformations of atomic groups. There are also structural collective phenomena coupled with lattice deformation, such as phase transitions, chemical and positional ordering, diffusion and intercalation to mention a few. When these phenomena co-exist and interplay with thermal expansion and/or compression under pressure , new functional properties may emerge: Invar effect, negative thermal expansion, and barocaloric properties serve as examples.

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 moderate pressure regime.

Here I present a sapphire capillary cell (SCC) that has been optimised 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 pressures up to 1000 bar. 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 use a wide variety of hydrostatic mediums. 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. Both temperature and pressure control are automated allowing highly autonomous phase space mapping.

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.

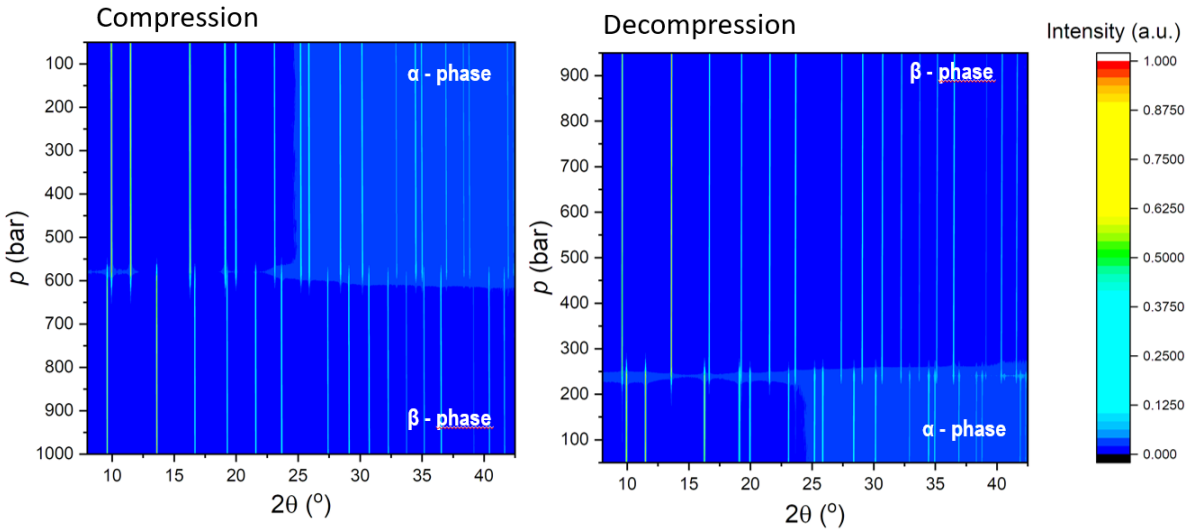


Figure 1. *Example of an isotherm showing a first order phase transition on compression from ambient to 1000 bar collected using the SCC with 10 bar steps.*