Preferred orientation in diamond anvil cells: a new approach using time-of-flight neutron diffraction

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Powder diffraction is an established means to determine crystallographic structure and is a critical tool for studying many 'real-world' systems where perfect single-crystal samples are unavailable. It is especially important in extreme-conditions research, where reconstructive phase transitions often obliterate single-crystal samples. A key requirement for powder diffraction is that the sample is formed of a well randomized powder, where crystallites do not preferentially orient along particular directions relative to the beam. Yet, opposed-anvil cells, which underpin the vast majority of high-pressure research naturally, induce a strong uniaxial stress gradient, which creates a significant risk of preferred orientation (PO).

We have developed a new approach where, by combining large azimuthal opening diamond anvil cells, large, highly pixelated detectors and time-of-flight (TOF) neutron diffraction, we can directly measure PO *in situ*. The key to this approach is that the TOF technique allows simultaneous angle and wavelength resolution. A broad, white beam of incident neutrons generates a continuum of Debye-scherrer cones, each sampling subsets of crystallites with different orientations relative to the beam. As each pixel records a full, wavelength-resolved, diffraction pattern, it is possible to extract distinct powder pattern for each subset of crystallites.

Here we will present our initial implementation of this approach, using high-pressure measurements of D_2O ice VII on the SNAP diffractometer at the SNS. We were able to achieve a 4° resolution across our full coverage angle of ~60° in 2θ (see Figure 1). We will discuss our implementation of an attenuation correction for the upstream diamond, which is a critical component of the data reduction.

Using this approach, we have been able to introduce limits on the maximum amount of PO that is consistent with the measured data. Further, we have investigated the possible impact of this potential PO on refined atomic-positions of the D atoms. The results of this study have important implications for measurements of H-bond symmetrisation in high-pressure studies of ice. Moreover, they introduce important considerations for many other high-pressure neutron diffraction studies using opposed anvil cells.

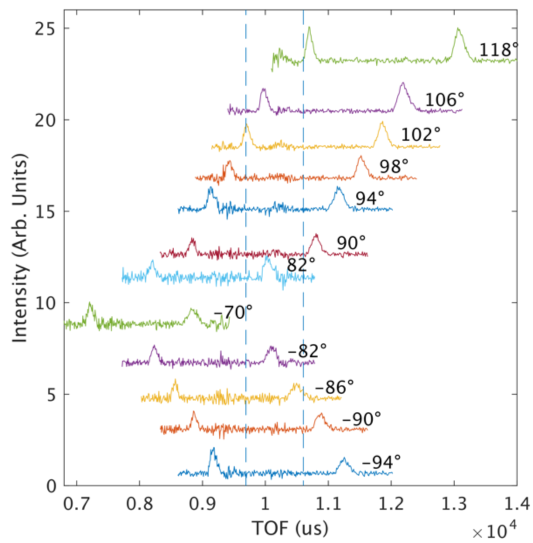

Figure 1. Indicidual powder diffraction datasets collected at different angles. The TOF axis is directly proportional to wavelength or, equivalently, d-spacing at a given 2θ.