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Single-crystal neutron diffraction in diamond anvil cells with hot neutrons

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A diamond anvil cell (DAC) is the most versatile tool to study structures and physical properties of (non-)crystalline materials at high pressures. The range of experimental techniques in a DAC is very broad: synchrotron and x-ray diffraction, inelastic x-ray scattering, optical and vibrational spectroscopies, transport and magnetic measurements, etc. While high-pressure single-crystal x-ray
diffraction in diamond anvil cells is nowadays a standard technique, there are scarcely any single-crystal neutron diffraction studies in a DAC, that would present complete structural refinements, reported in the literature [1-3]. This is mainly due to a limited sample volume available in the sample chamber of the DAC. Up to now, even at the most advanced neutron facilities it is difficult to routinely study crystals with volumes well below 1 mm^3 due to the low flux of the neutron beams. The requirement for large samples hinders a joint use of x-ray and neutron single-crystals diffraction upon compression. The combination of both techniques is highly advantageous for detailed studies on crystalline compounds, as neutron diffraction plays a crucial role in those cases where x-ray diffraction fails to provide information on, for instance, magnetic order or compounds containing light elements.

Recently, we have been exploring the feasibility of single-crystal neutron diffraction in a DAC on the single-crystal diffractometer HEIDI at the Heinz Maier-Leibnitz Zentrum (MLZ) in Garching (operated by the Institute of Crystallography at the RWTH Aachen University together with the Jülich Centre for Neutron Science). The instrument is installed at the hot source and offers short neutron wavelengths (0.55 Å < lambda < 1.2 Å) with high fluxes to obtain both high resolution and large Q range. We have tested several existing DACs at HEIDI to optimize the most efficient scattering geometry i.e., transmission and radial (panoramic) geometries. Currently, the minimum crystal size that could be studied there in a DAC is about 0.1 mm^3. The signal-to-noise ratio of the measured Bragg reflections improves significantly after minimizing the beam size and when using hot neutrons due to reduced scattering/absorption of the neutron beam by the components of the DAC. It is possible to determine the orientation matrix of the
crystal using standard searching routines in the reciprocal space while the diffracted intensities are measured with a point detector. The acquisition time is about 10 minutes per reflection. The obtained data could be used for complete refinements of structural models with the program Jana2006.

We have now developed optimized DACs for measurements at room and low temperatures. Our results demonstrate that single-crystal neutron diffraction in the DAC with hot neutrons at the MLZ is feasible. Upgrading the single-crystal diffractometer HEIDI, i.e., the monochromator to operate at short wavelengths and the neutron guide to minimize the beam size, will allow for much smaller crystals.

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