ESRF	**Experiment title:** Calibration of the Raman diamond pressure gauge to above 500 GPa in a toroidal-DAC	**Experiment number**: HC4227
Beamline: ID15b	**Date of experiment**: from: 02/06/2021 to: 06/06/2021	**Date of report**: 09/09/2021
Shifts: 12	**Local contact(s)**: Gaston Garbarino, Michael Hanfland	*Received at ESRF:*

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Report:

Scientific background and objectives

The diamond anvil Raman phonon frequency shift at the interface with the sample is now currently used as a pressure measurement above 100 GPa for laboratory experiments. However, this spectroscopic pressure measurement could largely overestimate pressure above 400 GPa. The aim of this proposal is to accurately calibrate the Raman diamond pressure gauge up to 500 GPa using two calibrants, copper and diamond, both embedded in Ne, by relating their XRD volumes to pressure through their EOS.

Experimental details and results

The X-ray and Raman measurements were performed on the ID15B beamline. A 30 keV x-ray beam was focussed down to 2-3 microns thanks to refractive lenses from ID06 and tested for the first time on the ID15during this experiment. Data were collected on an Eiger 2 detector that offers a large dynamic range and a very weak electronic noise. The Raman measurements were performed online using a portable Raman microscope brought from our laboratory. It is important to note that the sanitary conditions have strongly complexified the organization of the experiment which required our presence at ESRF to install and control the Raman bench. Two membrane diamond anvil cells (MDAC) were equipped with Boehler-Almax anvils of large X-ray aperture (+/- 35°). In the first MDAC, FIB machined toroidal anvils were used (culet diameter of 30 microns) . The second MDAC was equipped with anvils with beveled culets of diameter 40 microns. Small single diamond crystals of 2 to 3 microns in thickness have been carefully selected using an optical profilometer. A small grain of Copper was also added in the sample cavity. The MDACs were loaded with neon at 1400 bar and ambient temperature.

The X-ray beam was slightly too large for the sample (5-6 microns in diameter) in the toroidal DAC, so the X-ray patterns were heavily polute by the gasket diffraction. These sample sizes actually require a submicron beam which should be available on the ID27 beamline in the next semester.
The second DAC had a sample size of 10-12 microns in diameter, which was sufficient to extract a clean diffraction pattern from the diamond single crystal. The volume measurements on diamond and copper were performed up to 200 GPa, pressure at which anvils failed (see figure 1a). The diffraction data were complemented by Raman measurements of the anvil culet and the diamond single-crystal (see figure 1b). Twelve pressure points were collected, with each point requiring approximately one hour for the sample pressure to stabilize.

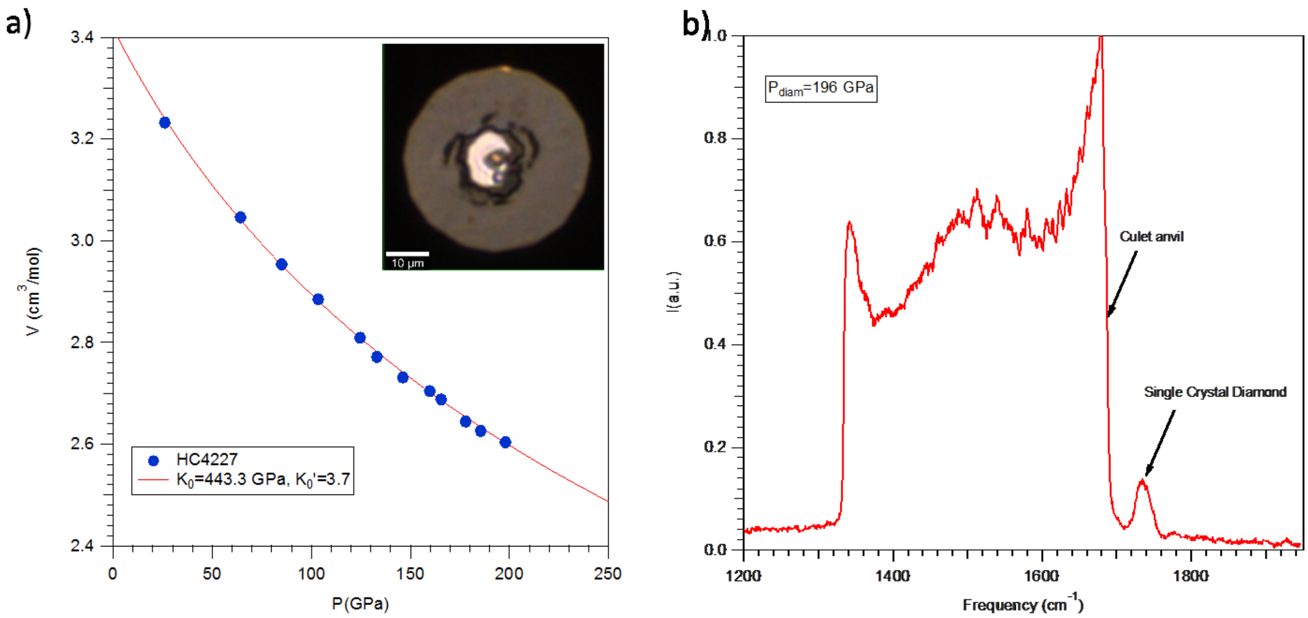

Figure 1: a) XRD volume of diamond vs pressure. The pressure is given by the frequency shift of the diamond anvil raman phonon. b) Typical Raman spectra collected on the Diamond single crystal. Above 20 GPa, the Raman signal of the anvil and the single crystal are well separated.

Despite the sanitary conditions, this experiment can be considered a success because we have demonstrate that the XRD signal from tiny diamond crystal in the DAC could be measured accurately under extreme conditions. The continuation of this project at higher pressure with toroidal anvils requires a sub-micron X-ray beam and will be possible on the ID27 beamline.