ESRF	Experiment title: Calibration of the diamond anvil Raman gauge to above 500 GPa in a toroidal-DAC	Experiment number: HC-3950
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Report:

The aim of this proposal was to calibrate the Raman of diamond edge as an optiocal pressure determination for laboratory experiments up to 500 GPa using toroidal diamond anvil cell. The t-DAC is based on a toroidal design of the anvil culet that compensates for the deformation of the anvil under very high load and so enables to pass the accepted pressure limit of conventional DAC. The stress distribution and deformation of the diamond anvil tip are different than for a conventional DAC. In particular we wanted to answer the following questions:

- Can the raman diamond edge be measured using the toroidal DAC?
- Is the existing Akahama's calibration, currently used up to 300 GPa, valid?

Two toroidal-DACs were prepared, both using 25 μ m culet toroidal-shaped diamond anvils. One loaded with Au and Pt 1 μ m in diameter pieces embedded in Ne. The second DAC with only argon. Only the DAC loaded with argon enabled to reach the 400 GPa range.

A portable Raman spectrometer was brought on ID 27 nbeamline with an opticval head that could be translated in front of the DAC on the XRD stage. Thev Raman signal was excited by a 660-nm-wavelength. A very discernible Raman diamond edge could be measured up to the maximum pressure. The pressure was measured from the volume of the rehenium gasket at the interface with the sample. A good agreement with the presure obtained with the diamond frequancy edge is obtained if the Akahama's scale 1 is used. Scale 1 has been proposed for pressure below 300 GPa. A scale 2 has been proposed above 300 GPa but seems to overestimate the pressure. More measurements are now needed to perform a calibration of the diamond edge up to 500 GPa.

These first results have been published in Nature Communication 9, 2913 (2018).



Fig. 6 Argon equation of state data. a Raman spectrum collected at the center of the toroidal-DAC at the end of run 5. The position of the high-frequency edge of the diamond anvil (minimum of the differential spectrum) is indicated by a red disc. It corresponds to a pressure of 420 GPa using the high-pressure calibration³⁵. b Volume of argon measured in run 5, compared with the equation of state obtained by a fit of lower pressure data points^{51,52} (black line). Insets: photographs of the sample. The image at 429 GPa is blurred by birefringence of the diamond anvil under high stress; the red color seen on the diamond tip is attributed to the closure of the diamond band gap³².