ESRF	**Experiment title: Calibration of the diamond anvil Raman pressure gauge to above 500 GPa in a toroidal-DAC.**	**Experiment number**: HC4884
Beamline: ID27	**Date of experiment**: from: 3/05/2022 to: 6/052022	**Date of report**: 03/10/2022
Shifts: 12	**Local contact(s)**: M. Mezouar and B. Wehinger	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Paul Loubeyre*, CEA ; Gunnar Weck*, CEA; Florent Occelli*, CEA.

Report:
Scientific background and objectives

The aim of this proposal was to accurately calibrate the Raman diamond pressure gauge up to 500 GPa. The first goal was to determine the pressure scale up to the 500 GPa range by measuring the volume of three calibrant Au, diamond, Cu in Ne pressure transmitting medium. The second goal was to calibrate the spectroscopic pressure gauge based on the Raman of diamond at the tip. That gauge is currently used for all laboratory measurements above 200 GPa, but its calibration is debated.

This proposal was the continuation of proposal HC-4227 which could not be fully performed on ID15B. The X-ray spot size (larger than the requested 2 µm diameter even by using refractive lenses) was not adapted to extract a sufficiently clean diffraction pattern from a 1 µm grain size samples of diamond and of metals in the t-DAC. Data could however be collected up to 200 GPa (see report HC-4227), using standard bevelled diamond anvil.

Experimental details and results

For the run , 4 DAcs were prepared for this campaign:

- DAC#1 with 30 µm flat bevel anvils with 2 µm in diameter samples of C, Au and Cu loaded in Ne.
- DAC#2 with 30 µm toroidal anvil with a pit in one culet to avoid bridging the diamond sample at very high pressure and loaded with 2 µm in diameter samples of C, Au and Cu in Ne.
- DAC#3 with toroidal diamond of 24 µm diameter culet, hence dimensionned to go to over 450 GPa. The sample chamber was loaded with Au only.
- DAC#4 with toroidal diamond of 16 µm diameter culet, hence dimensionned to go to over 600 GPa. The sample chamber was loaded with Au only.

Data were collected to 168 GPa in DAC#1. Diamond broke probably because of the bridging of the diamond piece between the two anvils. The XRD spectra obtained are much improved over those obtained on ID15B (see figure below).
The sample in DAC#2 became instable above 60 GPa. The thickness of the gasket at start was increased too much to take into account the volume of the pit.
DAC#3 and DAC#4 went up above 400 GPa but broke before the maximum pressure expected. In both cases the qualitry of the sample was perfect but the pressure ramp was performed in less than 10 hours. That is much faster than what is currently done. The elastic deformamation of the diamond anvil went too fast. Excellent data were obtained and the calibration of the Raman pressure gauge could be made using the Au and Rhenium EoS. Future experiment will be performed by statrting at 300 GPa. The slow increase of pressure to assure an optimized elastic deformation of the toroidal anvil will be made in opur home laboratory.

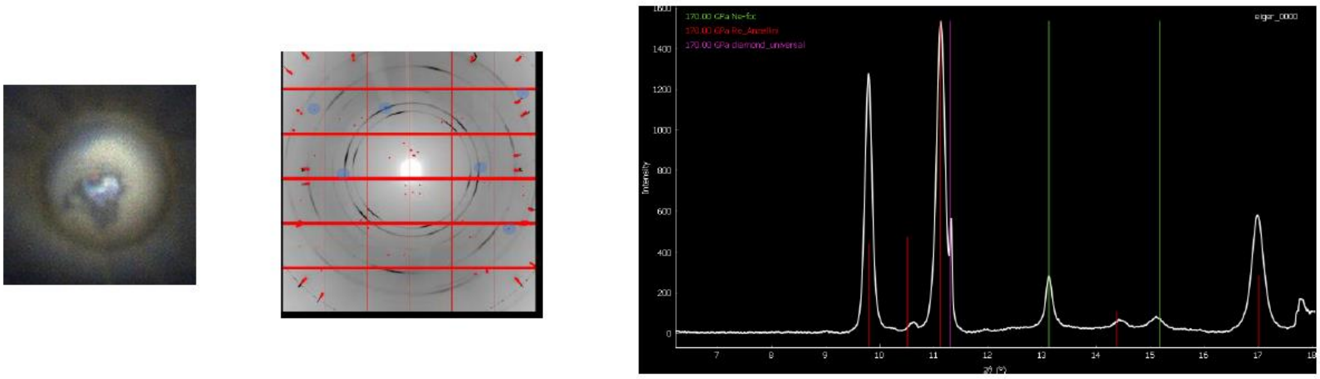

Figure 1: Sample (C, Au, Cu in Ne) and XRD spectra taken at 168 GPa in DAC#1

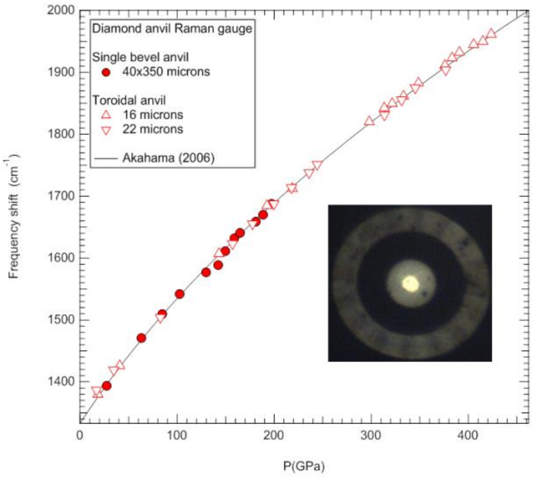

Figure 4: Diamond Raman frequency at the anvil-sample interface versus pressure, measured from the Au and Re volume. The Au sample in a Rhenium gasket in te toroidal DAC #3 is shown in the inset.