



	Experiment title: New diamond anvils to perform static measurements up to 500 GPa (continuation)	Experiment number: ME-1380
Beamline: ID27	Date of experiment: from:19 to:21/07/2015	Date of report: 24/07/2015
Shifts: 6	Local contact(s): P. Parisiadis	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Dewaele Agnès*, Ocelli Florent*, Loubeyre Paul*		

Report:

The aim of this proposal (continuation of ME-1358) was to reach higher pressures with the static diamond anvil cell device using new shapes of diamond anvil tips.

We have slightly modified the design of diamond anvil tips taking into account observations made in ME-1358. A tore (as proposed by Bridgman for larger high pressure devices [1]), with similar dimensions but with a slightly softer profile than in ME-1358 has been machined on the two diamond anvils tips. The machining has been performed in a scanning electron microscope equipped with focused ion beam (FIB), as previously.

The gasket was made of rhenium and a small sample of gold was placed in a 4 microns diameter hole made with FIB in the pre-indented gasket.

We have been able to test one design in the planned beamtime (6 shifts). This time was necessary to focus the X-ray beam, align the diamond anvil cell and slowly increase pressure and perform measurements.

The change of tore design resulted in a drastic increase of the pressure reached: ~585 GPa (instead of 330 GPa at maximum for ME-1358), measured with the ambient temperature equation of state of the rhenium gasket [2]. This pressure is in the same range as pressures reached with double-stage diamond anvil cells [3]. This suggests that new ultra-hard materials, such as nanopolycrystalline diamond used in Ref. [3], are not mandatory to reach higher pressure in diamond anvil cells. The maximum gasket pressure vs membrane pressure (proportional to the force applied on the diamonds) is plotted in the inset of **Figure 1**. The experiment terminated with the failure of one of the diamond anvils.

At each pressure step, the X-ray diffraction signal of the gold sample and rhenium gasket has been recorded at the center of the diamond tip, as well as the deformation of the diamonds with X-ray absorption scans (not shown here). Every 50 GPa, a pressure distribution map in the gasket was measured using rhenium gasket X-ray diffraction signal and equation of state [2]. These distribution maps exhibit a very large pressure gradient (up to 60 GPa/micron) in the inner part of the tore but also a relatively homogeneous zone with a ~8 microns radius at the center of the tip. The equation of state of the gold sample measured using the rhenium gasket as a pressure X-ray calibrant is plotted in **Figure 1** (green points). The (111) X-ray diffraction line of gold is used for this measurement. This equation of state agrees with the extrapolation of the equation of state measured in quasi-hydrostatic conditions [4] (blue curve). In other words, the extrapolated equations of state of gold [4] and rhenium [2] agree with each other in the 600 GPa pressure range. The non-hydrostatic

pressurizing conditions strongly influence the (200) diffraction line of gold, as expected from previous measurements [4].

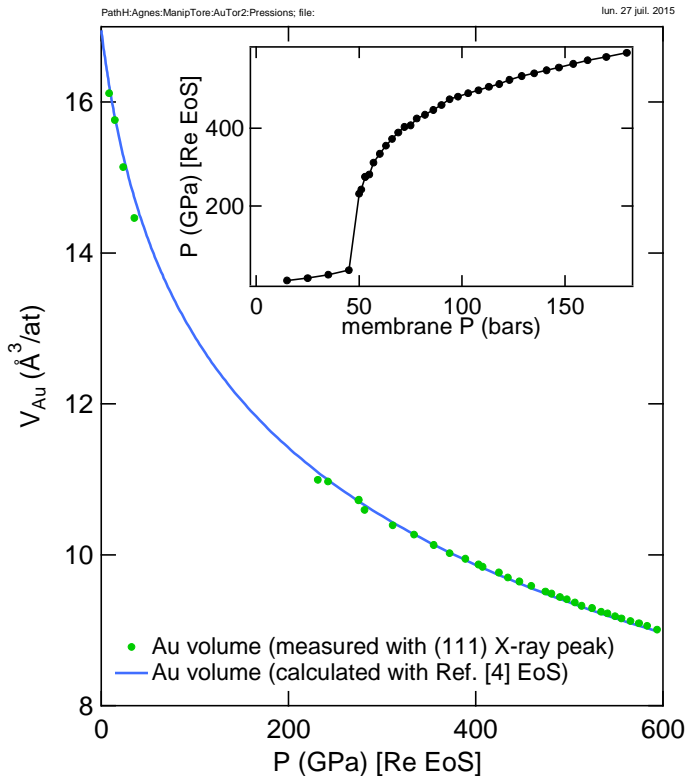


Figure 1: Green points: gold atomic volume, measured using the (111) X-ray diffraction peak of this metal (it is the least affected by non-hydrostatic pressurizing conditions, see Ref. [4]) vs pressure estimated using Re equation of state [2] and (100) and (101) peaks. It is compared with the equation of state extrapolated from Ref. [4], continuous blue line. **Inset:** Pressure measured at the center of the diamond tip, using rhenium gasket equation of state, vs membrane pressure.

The good consistency between X-ray diffraction data measured for these two metals proves the possibility of performing reliable measurements in the toroidal diamond anvil cell device, in the 600 GPa pressure range, if a micro-focused X-ray beam is used. The next step of our research will be to load softer materials (alkali halides, liquids or rare gases) to improve the pressurizing conditions in this device.

References:

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- [4] Anzellini *et al.*, Equation of state of rhenium and application for ultra high pressure calibration, J. Appl. Phys. 115, 043511, 2014
- [3] Dubrovinsky *et al.*, Implementation of micro-ball nanodiamond anvils for high-pressure studies above 6 Mbar, Nat. Comm. 3, 1163, 2012
- [4] Takemura and Dewaele, Isothermal equation of state for gold with a He-pressure medium, Phys. Rev. B 78, 104119, 2008