ESRF	**Experiment title:** New diamond anvils to perform static measurements up to 0.5 TPa: validation on the Ta behaviour	**Experiment number**: ME-1358
Beamline: ID27	**Date of experiment**: from: 18-21/06/14 and 03-05/04/14	**Date of report**: 26/08/2014
Shifts: 16	**Local contact(s)**: M. Mezouar, P. Parisiadis, V. Svitlyk	*Received at ESRF:*

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Report:

The aim of this experiment was to test new shapes of anvil tips designed to reach higher pressures with diamond anvil cells and to measure the ultra-high pressure behaviour of tantalum.
The concept of these new tips was inspired by the toroidal modification of Bridgman anvils for large volume high pressure apparatus [1] and by a very recent work [2]. It consists of machining a toroidal hole on the diamond flat tip with focused ion beam (FIB) (**Fig. 1**). This tore was supposed to reduce the gasket flow toward the ouside of the diamond. Inside the tore, a central flat zone would allow inducing a large pressure gradient in the very thin gasket and thus reach ultra-high pressures as in Ref. [2].

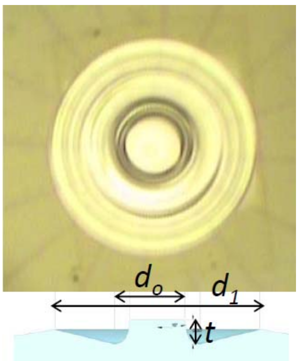

Fig. 1 : Geometry of the tores machined in the diamond anvil. d_0=16 µm for all runs, see **Table 1** for d_1 and t.

The sample was a Ta grain, placed in a ~4 µm diameter hole FIB drilled in a rhenium gasket. We have performed four experiments: one reference with a diamond with a classical shape, and three experiments with FIB machined anvils (see **Table 1**). For each run, the pressure has been very smoothly increased using an automated pressure driving system coupled with our membrane diamonds anvil cells. At each pressure step, the pressure was measured using tantalum sample X-ray diffraction signal and equation of state [3] and a measurement of the deformation of the diamonds was recorded using X-ray absorption scans (not shown here). Every few pressure steps, a pressure distribution map in the gasket was measured using rhenium gasket X-ray diffraction signal and equation of state [4]. These measurements provide reference data which will be used for a finite element modeling of this mechanical system, which is currently performed in our laboratory.

Name	Tore depth t (µm)	Tore outer diameter d_1(µm)	Gasket	Max. pressure (GPa)
TaTor3	No tore	Culet diameter 25 (µm)	Re	280
TaTor1	2	60	Re double	335
TaTor4	5	60	Re	309
TaTor6	2.8	40	Re	240

Table 1: Conditions of the four experimental runs.

The evolution of pressure as a function of membrane pressure measured for all runs is presented **Fig. 2,** together with pressure distribution maps for two runs.With classical diamonds, the pressure gradient on the culet is around 6 GPa/µm, whereas with tores, the pressure gradient is steeper (around 25 GPa/µm for run TaTor4) and localized on the central flat culet. One of our goals, to create a zone with large pressure gradients, was thus reached. However, we have noticed a large extrusion of the rhenium gasket from the toroidal hole, leading to a mechanical instability for the deepest tore studied (TaTor4). In future experiments, we want to limit this extrusion by a change of the tore design, the use of a composite gasket and a work on the diamond surface to increase the diamond/gasket friction coefficient. In the run TaTor6, we reached only 240 GPa: the tore diameter was too small, which lead to the bridging of the diamonds at d_1 and to the diamonds failure.

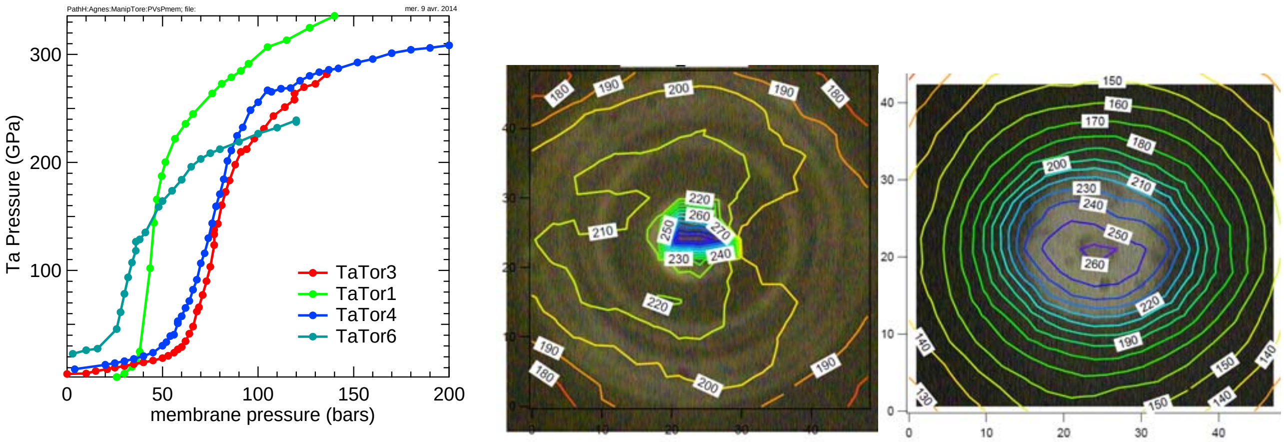

Figure 2: left: reached pressure, measured with tantalum equation of state, as a function of the membrane pressure which is proportional to the force applied on the back of the diamonds; center: pressure distribution map in run TaTor4 (pressure in GPa, position in microns); right: pressure distribution map in run TaTor3, diamonds with no tore (same units). The pressure maps are superimposed on photographs of the anvils culet.

To finish, our measurements prove that Ta and Re equations of state [3,4] are consistent in the scanned pressure range.

Our tests are promising as we managed to create ultra-high pressure gradients in the metallic gasket, and we have already reached pressures ~50 GPa higher with toroidal anvils than with classical anvils. We want to extend these tests with the ideas presented above. In addition, we want to check if the pressure reached has not been limited by a chemical-mechanical effect, Ta being reactive with diamonds under pressure. This can be done by using another sample material.

References:

[1] Eremets, High pressure experimental methods, Oxford University Press, 1996

[2] Dubrovinsky *et al*., Implementation of micro-ball nanodiamond anvils for high-pressure studies above 6 Mbar, Nat. Comm. 3, 1163, 2012

[3] Dewaele *et al*., Equations of state of six metals above 94 GPa, Phys. Rev. B 70, 094112, 2004

[4] Anzellini *et al*., Equation of state of rhenium and application for ultra high pressure calibration, J. Appl. Phys. 115, 043511, 2014