ESRF	**Experiment title:** Melting of tantalum in a laser-heated diamond anvil cell detected by x-ray diffraction	**Experiment number**: HS-3532
Beamline: ID27	**Date of experiment**: from: 01-03-2008 to: 03-03-2008	**Date of report**: 22/08/08
Shifts: 9	**Local contact(s)**: M. Mezouar	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*Dewaele A, *Loubeyre P, CEA, France

*Guignot N, Synchrotron Soleil, France

Report:

The aim of the proposal was to characterize the melting of tantalum in a diamond anvil cell, using an X-ray diffraction diagnostic: the appearance of a typical pattern of x-rays scattered by the liquid sample (a diffuse ring). It is possible on the ID27 beamline of ESRF. This beamline allows to simultaneously laser-heat, measure temperature by pyrometry, and record X-ray diffraction signal from a small area of a sample in a diamond anvil cell.

Our goal was to confirm, or infirm, the discrepancy between the melting curve of tantalum measured in laser-heated diamond anvil cell, LHDAC (Tm~3700 K at 100 GPa [1], almost the same as at ambient pressure: 3300 K) and the melting point reported by shock compression (Tm~8000 K at 270 GPa [2]). For that purpose, pressures in the range of 50-100 GPa had to be reached, a domain in which the melting curve was reported to flatten when the LHDAC technique was used [1].

During this experiment, four sample assemblies have been tested to the conditions summarized in **Table 1.** The sample was a tantalum foil of ~5 microns thickness. Different gaseous or solid pressure transmitting media have been used: sodium chloride NaCl, argon, neon, single crystal sapphire plates Al_2O_3. In some runs, diamond anvils coated with a few microns of alumina have been used. It allowed us to test the effect of the sample assembly, of the melting point of the pressure transmitting medium, and of its chemical reactivity on the temperatures reached during laser heating and on the observation of melting of tantalum.

Run	Anvils culet size (μm)	coating	Pressure range (GPa)	Temperature range (K)	Pressure medium	Gasket
1	300 x 500	N	24-30	300-~4000	$Ar+Al_2O_3$	Re
2	150 x 350	Y	70-120	300-~5000	Ar	Re
3	100 x 300	N	75-85	300-~5500	NaCl	Re
4	150 x 350	Y	100-120	300-~4500	Ne	Re

Table 1: conditions of each experimental run. The pressure/temperature ranges correspond to pressure/temperatures at which X-ray diffraction signal of the sample was recorded.

We observed that tantalum reacts, probably with argon, at moderate pressure (run 1, P<30 GPa) but not at higher pressure (run 2). A chemical reaction also occurred when NaCl pressure transmissing medium was used (run 3), but only after heating at extremely high temperatures (T>6000 K). In runs 2, 3 and 4, a new phase of tantalum was observed, but the bcc phase was the one in equilibrium with the liquid phase (see **figure 1**). We believe that this new phase was the product of a reaction between tantalum and the rhenium gasket, an hypothesis which needs to be confirmed by experiments with an other gasket material.

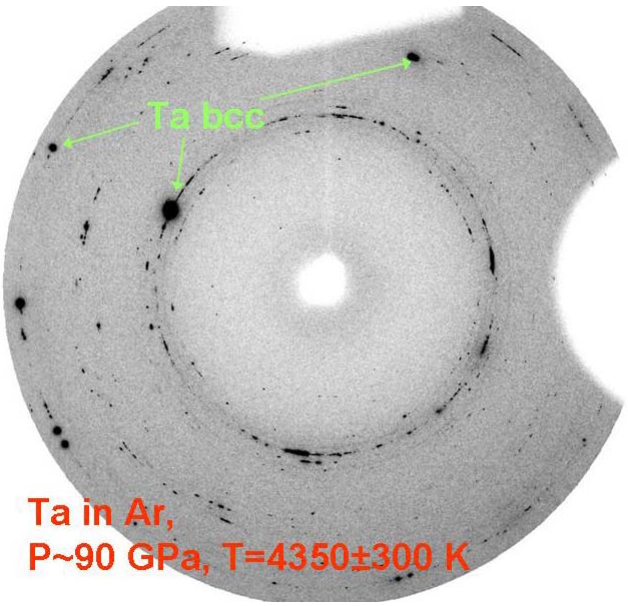

Figure 1: Monochromatic X-ray diffraction spectrum of a tantalum sample, laser-heated in argon pressure transmitting medium to 4350±300K. The diffracted signal allows identifying the solid bcc phase of tantalum (powder rings + round spots which characterize single crystals, probably crystallized from the melt), solid argon and a fluid phase (diffuse ring).

The X-ray diffraction signal of molten tantalum has been observed in runs 2 and 3 (see **figure 1**), at temperatures higher than ~4000 K above 70 GPa. The temperatures reached during run 4 (4500 K) was probably not high enough to observe any melting. These observations, even if they need to be confirmed by additional runs, suggest that the melting curve of tantalum around 100 GPa is 500 to 1000 K higher than the previous claims [1]. Six additional shifts are planned for experiment HS-3532, which will be used to clarify the issue of chemical reactivity between tantalum and rhenium, and better constrain temperature metrology.

References :

[1] D. Errandonea *et al.*, Phys. Rev. B **63**, 132104, 2001
[2] M. Ross *et al.*, J. Phys. Chem. Solids **67**, 2178, 2006