ESRF	**Experiment title: Mechanical properties of Ta under high pressure by single crystal x-ray diffraction ; determination of an absolute pressure scale**	**Experiment number**: HS-1831
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Shifts: 18	**Local contact(s)**: A.C. Dhaussy	

Names and affiliations of applicants (* indicates experimentalists):

- ***Dewaele Agnès – CEA/DPTA – BP12 – 91680 Bruyères le Châtel – France**
- ***Berhanu Michael – same address**
- ***Loubeyre Paul - CEA/DPTA – BP12 – 91680 Bruyères le Châtel – France**

Report:

The aim of these experiments were the following:

(1) determination of a reference equation of state (EoS) for Ta, in the Mbar pressure range, that would allow the calculation of an absolute pressure scale;

(2) estimation of the pressure and the plastic strain effect on the yield stress Yc of tantalum

For this purposes, monochromatic single crystal X-Ray diffraction has been used.

(1) Tantalum EoS:

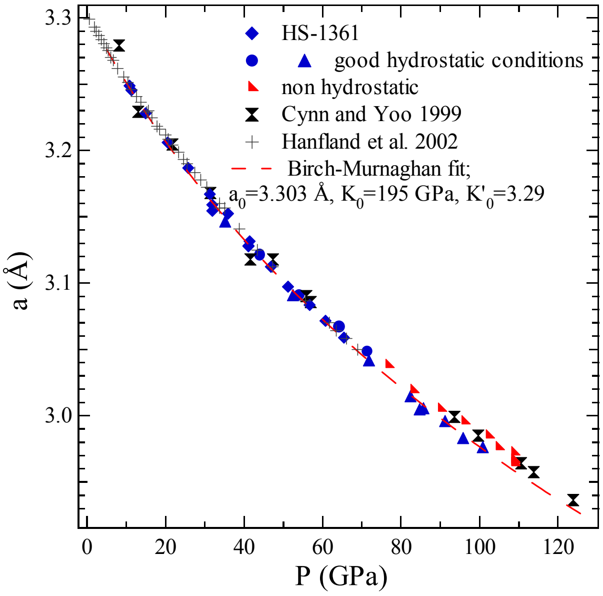

Figure 1: Ta lattice parameter as a function of P at 298 K

Two runs have been performed up to 110 GPa. Small tantalum single crystals (~3 µm size) and a small ruby chip were embedded in helium in a Re gasket, and compressed between two ultrahigh pressure diamonds anvils. Unfortunately, the first run has not been performed in good hydrostatic conditions from 70 to 109 GPa because of an helium leakage trough one diamond. The second run has been performed in quasi-hydrostatic conditions up to 101 GPa. This has been checked by comparing the lattice parameter based on various interreticular distances, for all single crystals; the variations *Δa/a* were of the order of $\pm 1.5.10^{-3}$ for low pressure points and $\pm 3.10^{-3}$ for the highest pressure achieved. This includes the variations of *a* in a single crystal and between the different crystals. Both results are presented on **figure 1**, together with literature data ([1], [2]) and HS-1361 results. Hanfland et al. [2] measured the compression of tantalum up to 69 GPa at ESRF, using the ruby pressure scale [3]. Their data are in excellent agreement with ours. But Cynn and Yoo [1] measurements lead to a lower ultra-high pressure compressibility. Their pressure calibration was based on gold EoS [4]. This suggests that either this pressure scale is not compatible with the ruby pressure scale, or these measurements were not performed in good hydrostatic conditions.

(2) Yield stress evolution under high pressure

Two experimental runs under high deviatoric stress have been performed. In these runs, the sample, embedded in Ne, was compressed directly between the anvils. In each run, several interreticular distances of the same single crystal have been measured, and the subsequent deviatoric stress has been calculated. This method has been first developped for white beam x-ray diffraction (see experimental report HS-1361), and successfully applied on monochromatic x-ray diffraction during this experiment. We also estimated the plastic strain by measuring the space between the diamonds by white light interferometry.

For the first experiment, we used a single crystal of 25 µm thickness and 75 µm diameter, compressed between two 400 µm tip diamonds. This sample has been strained at a constant pressure (20 GPa) by cycling the membrane pressure, to increase the cumulated plastic strain from 0 to 20%. For each strain step, stress and strain have been measured. This allowed us to establish a strain hardening curve under high pressure (figure 2), in the large plastic strain range. The strain hardening factor $(dYc/d\varepsilon^p)/Yc(\varepsilon^p=0)$ is 8.5 at ambient pressure [5]. Here, this factor is approximately 40 at 20 GPa. At $\varepsilon^p=0$, the pressure effect is $dYc/dP=0.0075$. These data show that strain hardening dramatically increases with pressure. Thus, the drastic pressure effect on the yield stress previously reported in the literature ([6] for instance) could be a coupled effect of pressure and plastic strain.

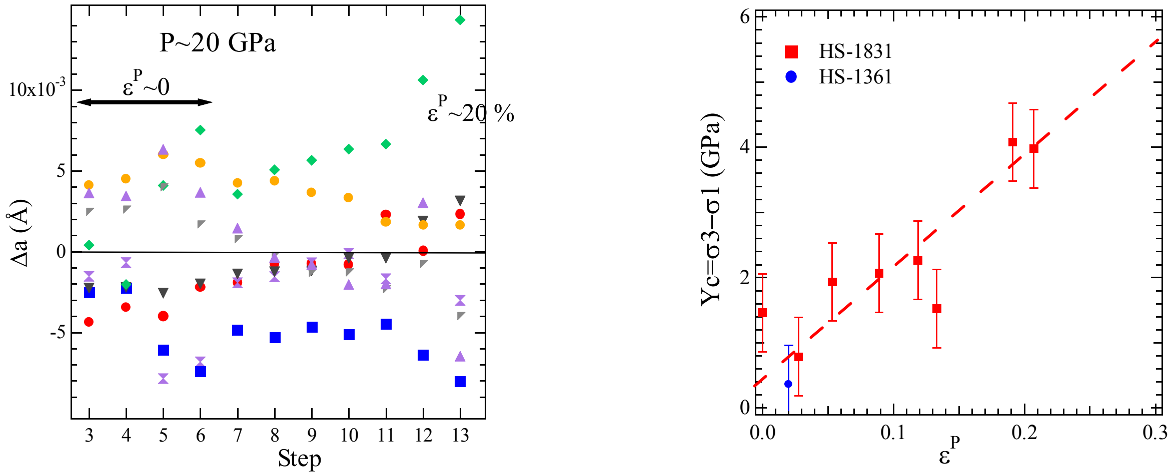

Figure 2: left: evolution of $\Delta a=(dhkl*\sqrt{(h^2+k^2+l^2)})-(dhkl*\sqrt{(h^2+k^2+l^2)})_{av}$ for several planes as a function of the strain step. The plastic strain was negligible up to step 5, when the diamonds went in contact wth the sample. **Right:** stress-strain curve at P=20 GPa.

The second experiment has been carried out in the small plastic strain ($\varepsilon^p<10\%$), up to 89 GPa. Lattice parameter variations are shown on **figure 3.** They have not yet been interpreted in terms of stress tensor.

References:
[1] Cynn and Yoo, Phys. Rev. B, 59, 8526-8529 (1999)
[2] Hanfland et al., J. Appl. Phys., 91: 4143-4148 (2002)
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[4] Heinz and Jeanloz, J. Appl. Phys., 55, 885 (1984)
[5] Bridgman, J. Appl. Phys., 24, 5, 560-570 (1953)
[6] Hemley et al., Science, 276, 1242-1245 (1997)

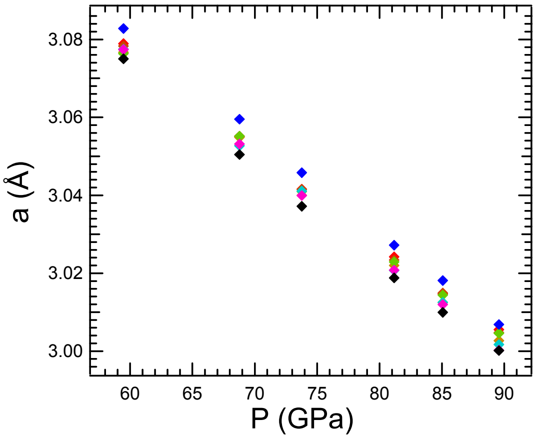

Figure 3 : evolution of a $dhkl*\sqrt{(h^2+k^2+l^2)}$ for several planes