ESRF	**Experiment title: Single crystal elastic constants of Ta by inelastic X-Ray scattering under high pressure – Determination of an absolute pressure scale**	**Experiment number**: HS-1595
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Report:

The aim of this experiment was to measure the elastic constants of a single crystal of tantalum, hydrostatically compressed in a diamond anvil cell. Combined with X-Ray diffraction data, these constants allow calculation of an absolute pressure scale by a simple integration.

We studied two samples (A and B), loaded in Ne in a 400 μm-culet diamonds anvil cell. Both were single crystals of Ta, thickness 25 μm, diameter 75 μm, oriented (110), provided by D. Farber and C. Ruddle, LLNL (USA).

Sample A has been studied with a 17.8 keV monochromatic x-ray beam (energy resolution 2.5 meV). It exhibited 0.3° FWHM mosaicity. We measured acoustic modes in the second Brillouin zone, located around a (110) peak (the most intense in the bcc structure). The most intense inelastic signal arises from the longitudinal mode. This mode propagates with the velocity $c=\sqrt{((C_{11}+C_{12}+2C_{44})/2\rho)}$. For a given momentum transfer **k**, related to the position of the detector, the energy of the longitudinal phonons is deduced from x-ray dispersion spectrum (see **Figure 1** for an example). Subsequent high pressure phonon dispersion curves are plotted in **Figure 2**. For sample A, the large scatter between the phonon energies obtained for different momentum transfers can be attributed to the presence of parasitic phonons, or to the too large energy resolution. At 19 GPa, sample A began to be close to the diamonds and its crystalline quality decreased.

To improve the quality of the measurements we thus decided to work on sample B with an x-ray energy of 21.7 keV. At this energy the photon flux is weaker, but the resolution is higher (1.5 meV). The mosaicity of the sample was 0.45° FWHM at 16 GPa. Because of the weakness of the signal, 15 hours have been necessary to collect only two data points. However, their agreement gives us a good confidence in our dispersion curve measurement; the obtained velocity (4400 ± 200 m/s) is 2 % smaller than predicted by extrapolation of low pressure data [1].

The inferred elastic constants are plotted on **Figure 3**, together with an extrapolation of low pressure (P<0.5 GPa) acoustic velocity measurements [1] and data based on X-ray diffraction [2]. The pressure derivative of $(C_{11}+C_{12}+2C_{44})/2$ has been estimated to be approximately 5, in good agreement with [1]. Data need to be obtained at higher pressure.

Unfortunately, mosaicity was too large to quantitatively exploit the transverse modes. In fact, these modes were partially hidden by the central elastic peak, enlarged by mosaicity. More work on the sample is needed to address this problem.

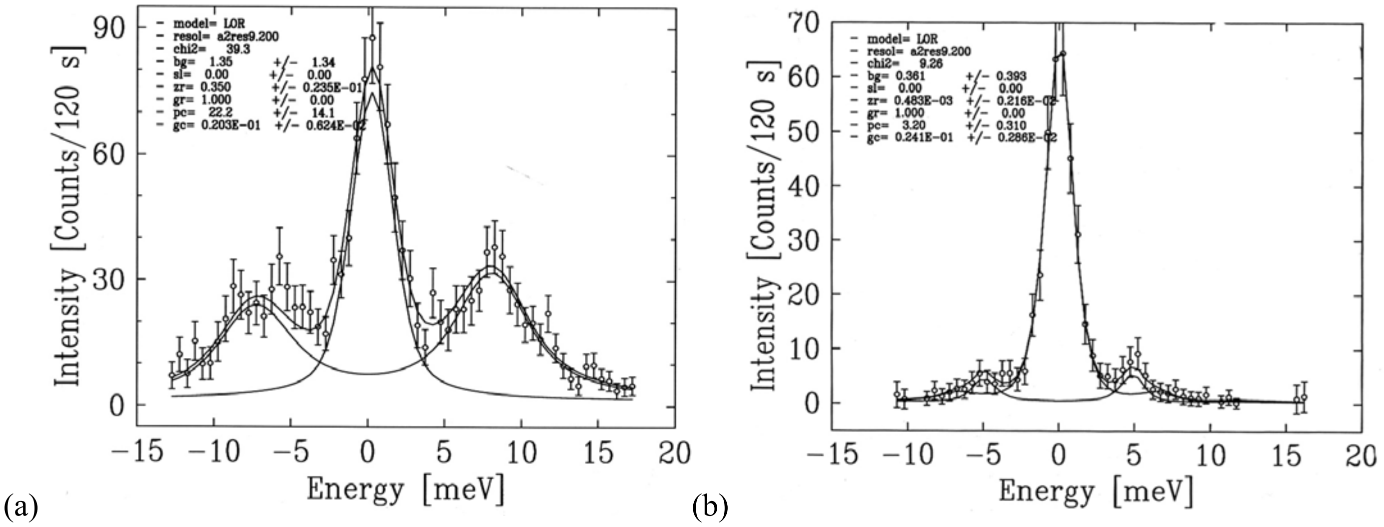
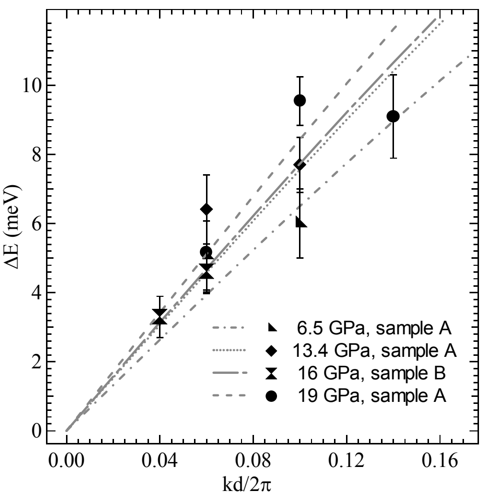


Figure 1: (a) spectrum obtained with sample A at 13.5 GPa at 17.8 keV (Si_{999}), at Q=(1.1, 1.1, 0), ΔE=7.7±0.2 meV. (b) spectrum obtained with sample B at 16 GPa at 21.7 keV ($Si_{11\ 11\ 11}$), Q=(1.06, 1.06, 0); ΔE=4.61±0.1 meV.



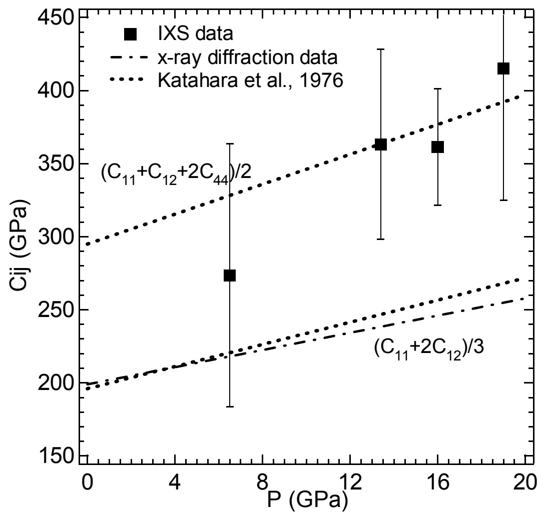


Figure 2: Dispersion curves in tantalum; d=a/√2 (distance between 110 planes); **k=Q-G$_{110}$** is the wave vector of the phonon, and a simple form ΔE=(hc/πd)sin(kd/2) is assumed.
Figure 3: Elastic constants deduced from these dispersion curves.

To conclude, this experiment confirmed the feasibility of single crystal elastic constants direct measurement under high pressure in a diamond anvil cell. Measurements in correct agreement with other techniques have been obtained. We determined the optimum conditions for x-ray scattering on tantalum or other heavy metals samples. In particular, the use of the highest energy possible (21.7 keV) is valuable. We still need to work on the single crystal quality, in order to be able to observe the acoustic transverse modes. We will then obtain good measurements of all elastic constants of Ta in the 60 GPa pressure range.

References

[1] Kahatara et al, J. App. Phys., 47, 2, 434-439, 1976
[2] H. Cynn and Yoo, unpublished results, 2001