ELASTIC MODULI OF POLYCRYSTALS UNDER HIGH PRESSURE. APPLICATION TO TA UP TO 5 GPA

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Abstract

The objective of this work is to determine the parameters of equation of state of polycrystalline metals, especially the adiabatic bulk modulus B and its pressure derivative. To describe the elastic behavior under high pressure at room temperature and assuming the metal as isotropic, the three elastic moduli (E-Young modulus, G-shear modulus, B) and the Poisson ratio ν can be calculated as a function of acoustic velocities and material density. We use a large volume Paris-Edinburgh cell, originally developed for high-pressure neutron diffraction and adapted to perform ultrasonic wave velocities measurements. This set-up allows to measure ultrasound velocities as a function of pressure up to 10 GPa.

This paper is especially applied to the elastic characterization of Ta. Our results are in good agreements with X-rays diffraction measurements (for this abstract, up to 5 GPa) and with ultrasonic measurements using a low-pressure set-up up to 1 GPa.

Introduction

The mechanical characterization of materials requires the determination of the elastic moduli (E-Young modulus, G-shear modulus, B) and the Poisson ratio ν . Furthermore, these moduli are the parameters needed to implement the constitutive laws and the equation of state. All these moduli depend on pressure. For our specific applications, our interest focuses on polycrystalline metals.

Experimental set-up

Low pressure cell

This set-up is used as a reference for the ultrasonic measurements between ambient pressure and 1 GPa [1]. The figure 1 shows the low-pressure cell. A handed pump with low viscosity oil is used to transmit pressure to the sample. A plate of $LiNbO_3$, which is sticked to the surface of the sample, can generate longitudinal or shear ultrasonic waves.

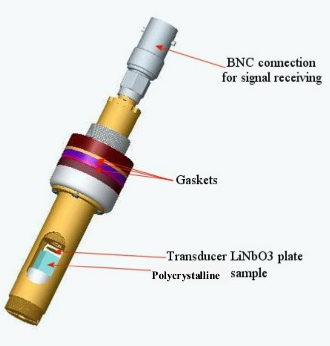

Figure1: Low-pressure cell

Large volume pressure cell : Paris-Edinburgh cell

The experimental cell, developed by [1], is shown in figure 2. An hydraulic press, which generates the necessary thrust, is connected to the lower part of the cell (blue unit in figure 2). The high-pressure chamber is placed between two opposite tungsten carbide toroidal anvils. The piston drives up the lower anvil to the upper anvil. A boron epoxy gasket is placed between the two anvils.

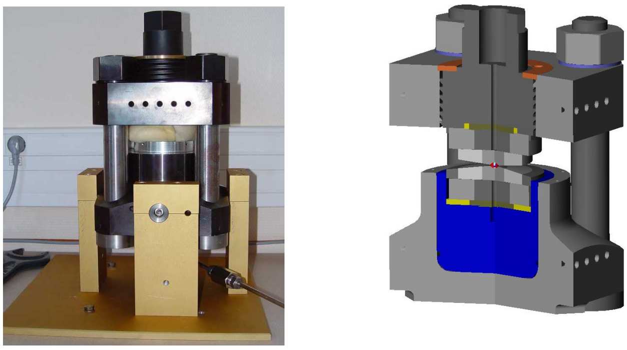

Figure 2: Paris-Edinburgh cell

This experiment requires cylindrical samples 1,9 mm in diameter and 3 mm in length. The sample is encased in a boron nitride (BN) cylinder, which is used as a pressure-transmitting medium. Compressed NaCl powder is located below and around the sample. The pressure value is determined by measuring the NaCl lattice parameters using the Murnaghan's equation of state. This device is put into the gasket (figure 3).

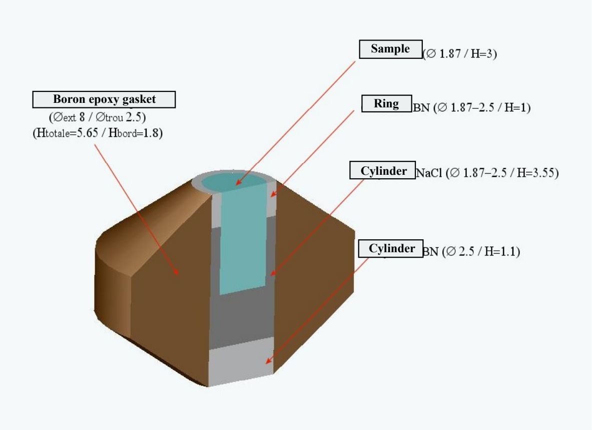

Figure 3: Sample and gasket

A plate of $LiNbO_3$, which is stacked to the rear surface of the upper anvil, can generate longitudinal or shear ultrasonic waves. The first echo is due to the anvil-sample interface and the second one to the back face of the sample.

Pressure calibration

Because all future experiments will be performed inside a glove box for nuclear material characterization, both ultrasonic and pressure measurements cannot be performed simultaneously. Some neutron diffraction experiments on different materials were used to obtain a pressure calibration. The measurement accuracy is estimated at ± 0,35 GPa, including the uniaxial component of the strain.

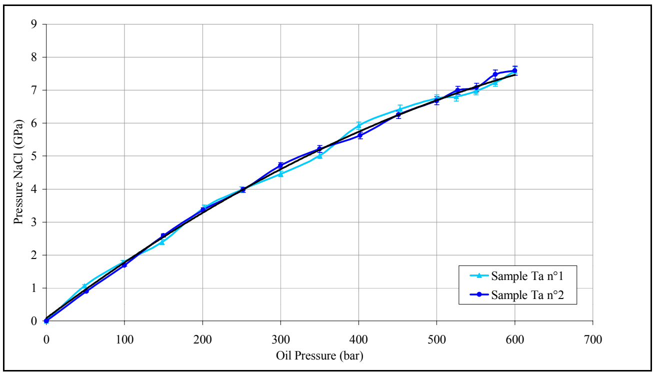

Figure 4: pressure calibration for tantalum samples

Determination of acoustic velocities

The pressure dependence of the ultrasonic transit time of the longitudinal and shear waves of tantalum (Ta) is measured using the low pressure set-up between ambient pressure and 1GPa, and the Paris-Edinburgh cell between 1,5 GPa and 5 GPa. For longitudinal and shear wave transit time, we calculated the acoustic velocity versus pressure. The variations of elastic velocities as a function of pressure obtained using these techniques are shown on figure 5. Measurements of velocities using an ultrasonic contact method with piezoelectric transducers. We can observe an increase about 7 % of the velocity of shear wave (Vt) and about 3 % of the velocity of the longitudinal wave (V_l). Furthermore, the values obtained by ultrasound at high pressure are in good agreement with those obtained at low pressure.

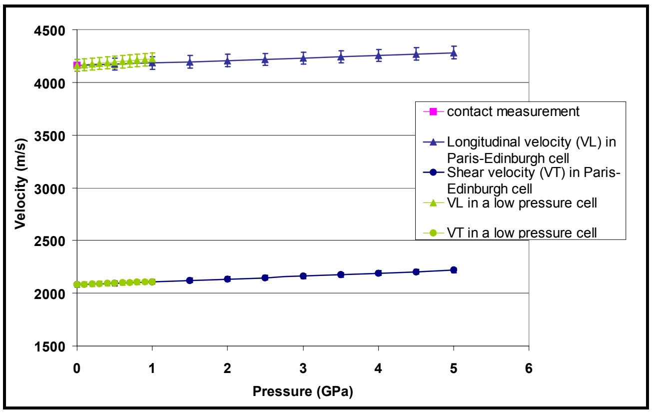

Figure 5: Pressure dependence of elastic velocities

Behavior of the elastic moduli versus pressure

The solid considered as a polycrystal is supposed to be isotropic, the four elastic moduli and the sound velocity c are then calculated as a function of acoustic velocities and ρ the material density.

$$c=\left(v_L^2-\frac{4}{3}v_T^2\right)^{1/2} \qquad E=\rho v_T^2\frac{3v_L^2-4v_T^2}{v_L^2-v_T^2}$$

$$G=\rho v_T^2 \qquad \nu=\frac{v_L^2-2v_T^2}{2\left(v_L^2-v_T^2\right)}$$

$$B=\rho\left(v_L^2-\frac{4}{3}v_T^2\right)$$

All the pressure dependencies of elastic moduli of Ta are shown on figures 6 to 9. Our results (low and high pressure) are compared with earlier published results [2,3]. We can observe a discrepancy between our results and those of the other group, especially for the shear modulus and the adiabatic bulk modulus. Differences in the studies samples such as single or polycrystalline sample, or in treatments, may be explain this discrepancy.

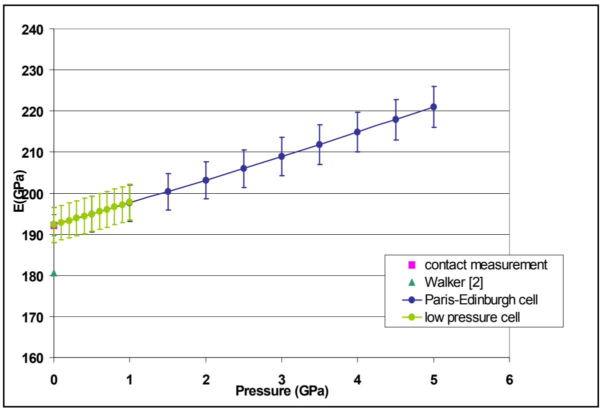

Figure 6: Pressure dependence of Young modulus

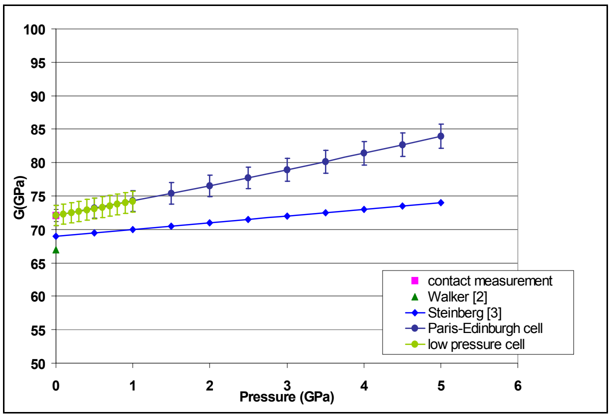

Figure 7: Pressure dependence of shear modulus

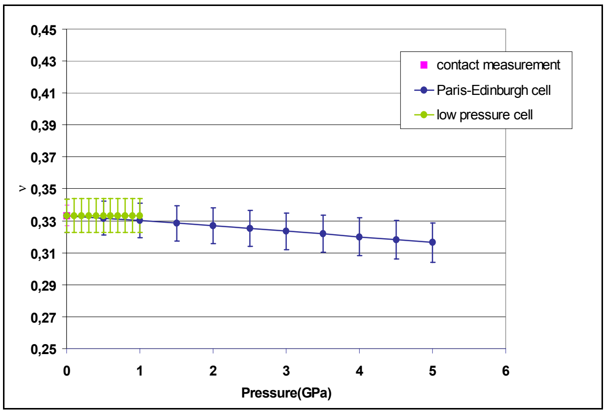

Figure 8: Pressure dependence of Poisson ratio

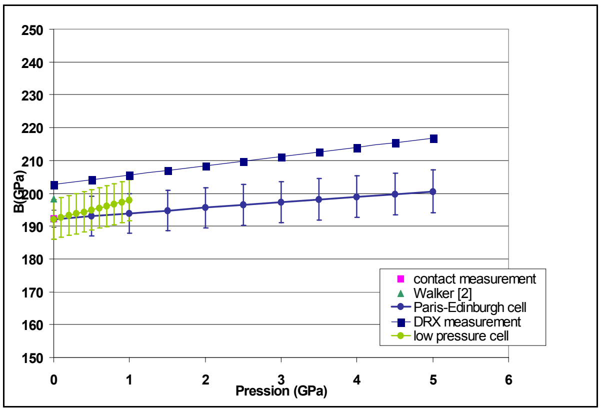

Figure 9: Pressure dependence of adiabatic bulk modulus

The elastic moduli of tantalum show a linear behavior up to 5 GPa. The pressure dependencies of acoustic velocities and of the elastic moduli are reported in table 1.

Table 1: Pressure dependencies of acoustic velocities and elastic moduli of tantalum obtained with the two set-up.

Paris-Edinburgh cell	**Low pressure cell**
$V_l = 4160 + 24\ P$ (m/s)	$V_l = 4160.8 + 60.6\ P$ (m/s)
$V_t = 2079.6 + 27.5\ P$ (m/s)	$V_t = 2081.1 + 29.4\ P$ (m/s)
$E = 191.9 + 5.7\ P$ (GPa)	$E = 192.3 + 5.5\ P$ (GPa)
$G = 71.9 + 2.3\ P$ (GPa)	$G = 72.1 + 2\ P$ (GPa)
$\nu = 0.334 - 0.003\ P$	$\nu = 0.333 - 0.0002\ P$
$B = 192.1 + 1.7\ P$ (GPa)	$B = 192.1 + 5.7\ P$ (GPa)

Conclusions

Current results confirm the great interest of using an ultrasonic technique for accurate measurement of the elastic properties of materials under high pressure. This technique permits to determine the pressure dependence elastic moduli of a polycrystalline metal. In this study, this technique is especially applied to the elastic characterization of tantalum. Within the same scope, the next study will be focused on solid, which presents solid phase transition between 1 and 10 GPa.

Acknowledgments

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References

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