Towards Pressure Calibration to 20 GPa in Multi-Anvil Apparatus: Example of Au

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Introduction

Among the increased applications of multianvil apparatus with synchrotron X-radiation source, NaCl is probably the most widely used internal pressure standard in high pressure research due to availability of the large body of experimental data as well as a continuous effort to update and refine the scale. Ruoff and co-workers proposed a method to determine absolute pressure based on simultaneous measurements of length and ultrasonic travel times for isotropically compressed materials. Using this method, the transition pressure of mercury at 0°C has been successfully determined at 0.76 GPa. The adaptation of synchrotron X-radiography into the multi-anvil high pressure apparatus has facilitated length measurement at high pressure and high temperature. In these experiments, a combined analysis of ultrasonic velocities and density using finite strain theory provides not only the determination of the elastic moduli and their pressure dependence independent of pressure measurement, but also the absolute pressure. In the following, we report our effort in extending absolute pressure determination towards 20 GPa using a double-stage multi-anvil apparatus (T-25) installed at synchrotron beamline 13-ID-D, GSECARS, Advanced Photon Source (Argonne, IL, USA)[1].

Methods and Materials

Major components of the experimental set-up include the multi-anvil press, the solid state detector, the X-ray imaging system, and the ultrasonic measurement system. The detailed layout of these components, installed at 13-ID-D/GSECARS, has been described elsewhere. The sample is placed next to an internal acoustic buffer rod enclosed in the MgO octahedron and is surrounded by a mixture of and NaCl plus boron nitride (BN) (NaCl:BN = 10:1 by weight). The mixture of NaCl plus BN around the sample serves for two important purposes: (1) to provide a pseudo-hydrostatic pressure environment for the sample; and (2) to serve as a secondary pressure standard. During the experiment, X-ray diffraction patterns from both the sample and the NaCl are collected using energy dispersive X-radiation source at a diffracting angle of 6.5°. The X-ray imaging system, consisting of a YAG scintillator and a CCD camera, captures the image of the cell assembly during high presusre experiment. The brightness contrast between the sample and its neighboring regions results from the difference in the X-ray absorption coefficients of the various materials in the high-pressure cell assembly. In this cases, the sample distincts from itself from the surrounding materials due to its high absorption coefficients, serves well in delineating the sample boundaries. To obtain sample lengths at high pressure and high temperature conditions, the very last image at the end of the experiment (zero pressure) is used as the reference image to retrieve the change in pixels between neighboring P-T conditions by cross-correlations of the intensity profiles at the center of the sample boundaries. Typically, a change of 0.25 pixel can be resolved using cross-correlation method. For a sample with a dimension of about 1000 pixels, a precision to resolve length change at 0.025% can be achieved.

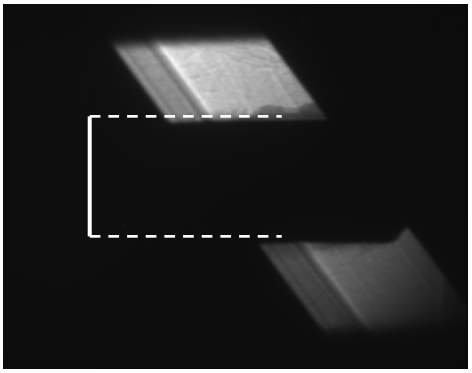

Fig. 1 X-ray image of sample at 16 GPa(dark region between dashed lines). L ~ 0.6 mm.

. By contrast, when the sample dimension in pixels is directly measured from an image, it has a total uncertainty about 2-4 pixels, which gives 0.2-0.4% in precision, regardless of the sample state at high pressure and high temperature.

A polycrystalline specimen of gold was used in this study. The sample was confirmed to be fine grain-sized (5-10μm), and free of cracks and porosities. Both surfaces of the sample were polished with 1 micron diamond paste finish and parallel within 0.1 degree. The final sample size used in ultrasonic experiment was about 2 mm in diameter and ~1.0 mm in length. After the sample was assembled into the cell assembly pressure was increased to a designated peak value (~15 GPa) at room temperature and and data were collected along compression at room temperature.

Results and Discussion

Knowing the velocities and density at a series of elastic strains along isothermal conditions, the absolute pressure can be calculated using finite stain equations (1)-(4),

$$P = -3K_{0T}\varepsilon(1-2\varepsilon)5/2(1+3(4-K_{0T}')\varepsilon/2) \quad (1)$$

$$K_T = K_{0T}(1-2\varepsilon)5/2(1+(5-3K_{0T}')\varepsilon) \quad (2)$$

$$K_T = K_S/(1+\alpha\gamma T) \quad (3)$$

$$\varepsilon = 0.5[(\rho/\rho_0)^{2/3}] \quad (4)$$

where ε is the Eulerian strain, α thermal expansivity, γ the Gruneisen parameter, and K_T and K_S isothermal and adiabatic bulk moduli, respectively.

Fig. 1 is a plot of the measured travel times as a function of pressure determined from Eos of NaCl. Fast decrease of travel time in both P and S waves is observed which are believed to correspond to the length shortening of the sample due to the fact that gold is soft compared to the other materials in the sample assembly. Using the sample length obtained at high pressures, the velocities are obtained. The results are presented in Fig. 2. Monotonic increase in both P and S wave velocities are observed. These data are processed using a finite strain procedure as described in [2], and the results for bulk and shear moduli, as well as their pressure dependence are obtianed. The bulk and shear moduli, Ks=219 (2) GPa and G=27(1) GPa are

compatible with previous studies. Interestingly, the pressure derivative for the bulk modulus is higher than many Earth minerals, but shear preperty is similar. These experiments are still preliminary and further experiments with improved cell assembly to keep sample from flowing will improve the accuracy for pressure calibration purposes.

Acknowledgements

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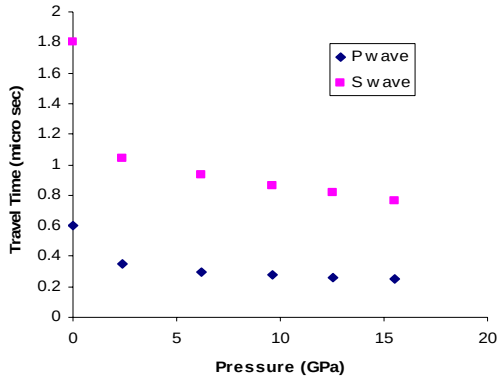

Fig. 1. Observed travel time for P and S waves at high pressure for Gold.

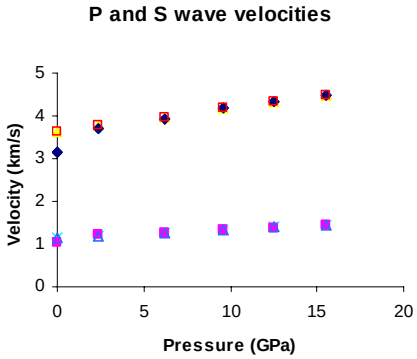

Fig. 2 P and S wave velocities og Gold at high pressures. Solid symbols: experimental data; Open: Fit.

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