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Under high-temperature and high pressure (HTHP) experiments, materials of small elastic modulus deform easily, and the length of the sample can be hardly predicted which lead to failure of ultrasonic velocity measurement. In this paper, a hydrostatic assembly of the sample for ultrasonic measurements is designed under HPHT, which can prevent plastic deformation. According to the abrupt change of travel time of the sample across the different phase boundaries of bismuth, the correspondent relation of sample pressure and oil pressure of multi-anvil apparatus can be calibrated, and the relation of sample temperature and temperature measured by thermocouple can also be determined. Sample pressure under high temperature is also determined by ultrasonic results. It is believed that the new sample assembly of hydrostatic pressure is valid and feasible for ultrasonic experiments under HTHP.

Keywords: phase transformation of bismuth; ultrasonic measurement; multi-anvil apparatus; hydrostatic pressure

Introduction

Measurement of ultrasonic velocities under high pressure and high temperature (HPHT) is very important for studying the equation of state and constitutive relation of a material. Generally, when the ultrasonic velocity is measured in multi-anvil apparatus [1–3], most scientists use NaCl power, BN power or Pb, etc. as the inner pressure medium. Plastic deformation of the sample will inevitably take place in such a quasi-hydrostatic pressure condition. Li [2] measured the ultrasonic velocity of Mg_2SiO_4 wadsleyite on an octahedral multi-anvil press by using NaCl + BN power as the inner pressure medium, and small deformation of the sample was found by the X-ray imaging technique. Under the quasi-hydrostatic pressure condition, the length of the sample after the experiment could not restore the previous length completely. Wang [3] measured the ultrasonic velocity of copper and aluminium by using Pb as the pressure medium on the hexahedral press, and found that the length of the sample after the experiment changed about 1% shorter than the previous length up to 5 GPa, which will influence the accuracy of ultrasonic velocity under high pressure.

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For materials of a relative large elastic modulus, there is little effect on the length change for the ultrasonic velocity measurement in the condition of quasi-hydrostatic pressure, and which could be neglected. But for materials of the relative small elastic modulus, such as Bi, Pb and Sn, the length of the sample changes greatly under quasi-hydrostatic pressure, and papers about ultrasonic velocity of such materials under HPHT are hard to find. Therefore, it is important to establish the ultrasonic measurement method to meet the need for studying such 'soft' material under HPHT.

Song and Yoneda [4,5] designed a solid–liquid hybrid assembly to measure the ultrasonic velocity of gold, and achieved ultrasonic velocity measurement under hydrostatic pressure on the multi-anvil apparatus by using methanol–ethanol admixture as the pressure medium. Inspired by the method by Song and Yoneda [4,5], Wang et al. [6] proposed a more convenient method for sealing liquid under high pressure to realize ultrasonic velocity measurement under hydrostatic conditions. But all the methods above are preformed at room temperature, and cannot be utilized to measure the ultrasonic velocity of samples under high pressure and simultaneously high temperature.

Based on the method of Wang et al. [6], we have made an improvement on the previous sample assembly. In this way, the new assembly can be further used for ultrasonic measurement under high pressure and simultaneously high temperature. The pressure and temperature in this new assembly are calibrated by the abrupt change of the travel times in Bi sample caused by phase transformations, sample pressure under high temperature is also determined by ultrasonic results.

Experiment set-up

The experiments are performed in a hexahedral multi-anvil press (YL-800t), which can generate pressure up to 5 GPa. The hexahedral cell assembly for HPHT ultrasonic wave experiments of hydrostatic pressure is shown in Figure 1. In this study, the sample of bismuth (0.455 mm long, diameter 6 mm) is affixed to the ceramic buffer rod (12 mm long, diameter 8 mm) by high temperature inorganic glue, and the surrounding of the sample is filled with silicon oil to apply hydrostatic pressure environment. To prevent the silicon oil from extruding out while applying pressure, the silicon oil, sample and Al_2O_3 buffer rod are inserted into a copper sleeve (20 mm long, outside diameter 10 mm, inner diameter 8 mm) by interference fit sealing as proposed by Wang et al. [6]. The heater tube is made of stainless steel for heating the sample. Pyrophyllite tube (outside diameter 12 mm, inner diameter 10 mm) is inserted between the heater and the copper sleeve as an electrical insulator. The thermocouple is placed on the underside of the copper sleeve to measure the on-line temperature of the assembly.

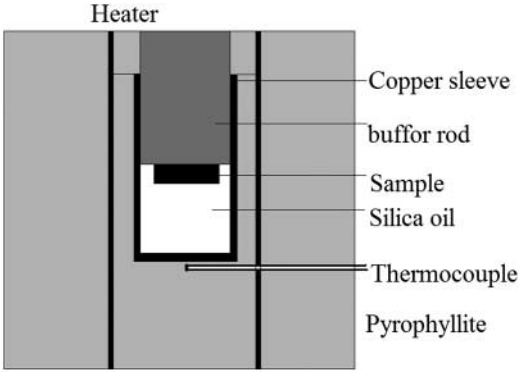

Figure 1. Hexahedral cell assembly for HPHT ultrasonic wave experiments of hydrostatic pressure.

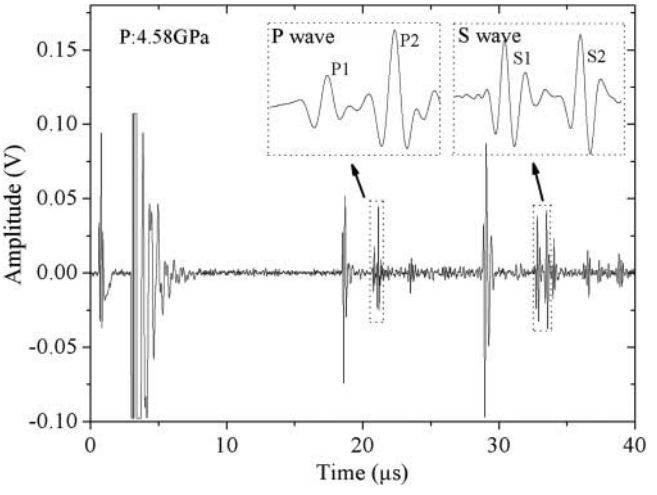

Figure 2. Classical signals of ultrasonic wave under HPHT. P1 and P2 are longitude waves and S1 and S2 are shear waves, which determine the travel time of sample.

The ultrasonic velocities (V_P and V_s) of bismuth are measured with the classical pulse-echo method by using the 5077PR ultrasonic square wave pulser/receiver unit (PANAMETRIC-NDT Incorporation, USA), TDS7054 digital oscilloscope (Tektronix Corporation, USA) and one immersion piezoelectric transducers (PANAMETRIC-NDT x1013) which can generate a 10-MHz longitudinal wave and 5 MHz shear wave in central resonant frequency simultaneously. Detailed descriptions of the ultrasonic measurement system have been given by Liu et al. [7,8]. Figure 2 shows the ultrasonic wave signal of bismuth at 4.58 GPa and room temperature. P1 and P2 are longitudinal wave signals reflected from the upper and lower interface of the sample, and S1 and S2 are shear wave signals reflected from the upper and lower interface of the sample. The travel time of the sample can be determined by the broadband spectroscopy method [6].

The uncertainty of the travel time of sample depends on the system error of the ultrasonic measurement of the pulse echo method technique and the bond effect. The system error of ultrasonic measurement in this experiment is about 1 ns which contains dispersion of ultrasonic waves and resolution of instruments. The thickness of the bond is about 10μm, and it is estimated that uncertainty is about 10 ns for longitude wave. So, the whole uncertainty of the ultrasonic data is about 3%. In this paper, we mainly discuss the phase transformation of bismuth, and the uncertainty of the travel time that is unrelated to the pressure of phase transformation.

Experiment result and discussion

Bismuth is one of the most studied elements at high pressure by electroresistance [9], volumetrically, by XRD [10] and neutron diffraction [11], by measuring elastic constants [12], by shock-wave compression [13,14] and theoretical calculation [15]. In the past few decades, bismuth is being widely used as a pressure calibrator [1,4] on the multi-anvil apparatus for its multi phases at high pressure. Tonkov and Ponyatovsky [16] presented the latest experimental and theoretical information about the phase diagrams of Bismuth. In this paper, we first make full use of the phase graph of bismuth by Tonkov and Ponyatovsky [16] as seen in Figure 3 to calibrate not only the pressure, but also the temperature of our new sample assembly.

At room temperature, the I–II transformation was assumed to be 2.52 GPa [17], the II–III transformation was observed at 2.69 GPa, and the III–IV transformation was measured at 4.27 GPa

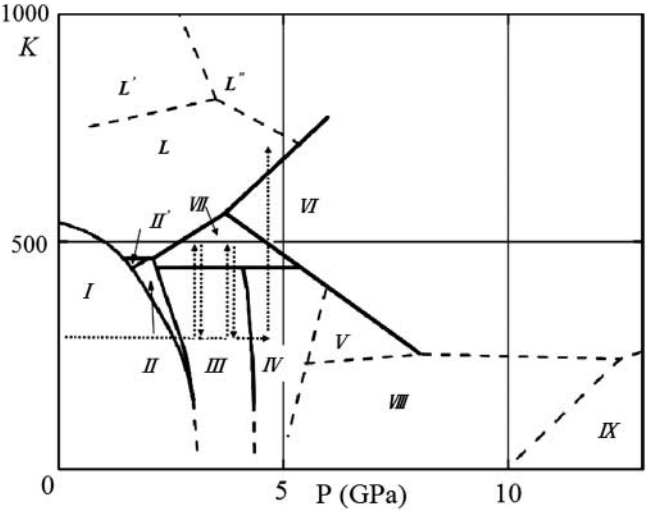

Figure 3. Phase diagram of bismuth by Tonkov and loading path of this study.

[18]. According to the III–IV–VII triple point (4.1 GPa, 448 K), IV–VI–VII triple point (5.4 GPa, 448 K), VI–VII-liquid triple point (3.8 GPa, 569 K) and the melting point (5 GPa, 700 K), it was found that the phase transformation temperature of III–VII and IV–VII is 448 K, which is independent of pressure. At 4.58 GPa, the phase transformation temperature of IV–VII is 448 K, the phase transformation temperature of VII–VI is 510 K and the melting temperature is 654 K, which are used to calibrate the sample temperature and the thermo pressure.

In this study, we increase oil pressure at room temperature first, the sample is heated at 330 kg/cm^2 (3.09 GPa), 400 kg/cm^2 (3.66 GPa) and 520 kg/cm^2 (4.58 GPa), respectively, and the loading path is shown in Figure 3 as the dotted line. With the pressure increasing, the travel times of the sample of both the longitude wave and shear wave are decreasing as shown in Figure 4. At an oil pressure of 258 kg/cm^2, the travel time of longitude changes abruptly as shown in Figure 4(a), meanwhile the echo signal of the shear wave becomes too weak to see. So it is believed that the I–II phase transformation takes place at this pressure. At an oil pressure of 287 kg/cm^2, the travel time of longitude wave begins decreasing and the echo signal of the shear wave appears again, it means that the II phase begins transformation to the III phase. Additionally, relaxation of phase transformation narrows the scale of pure phase II, and we can hardly achieve the slope of travel time versus oil pressure. The travel time of shear wave shows a slope change at around 320 kg/cm^2. The shear wave signal appears again at the oil pressure of 295 kg/cm^2, the amplitude of echo signal increases gradually with pressure increasing, and the slope of travel time versus oil pressure is sharp when oil pressure is less than 320 kg/cm^2, it can be considered that the II phase would transform to phase III entirely. At the oil pressure of 330 kg/cm^2, we begin to heat the sample assembly. The heating process ensures that the II phase of bismuth transforms to the III phase completely. It is presumed to be the reason for a slope change at around 320 kg/cm^2.

The relative volume change $\Delta V/V_4$ is just 1% and the crystal structure does not change [16] at the III–IV phase transformation of bismuth at 4.27 GPa, so the abrupt change in travel time is not found at the this phase boundary as seen in Figure 4. Around the oil pressure of 477 kg/cm^2, the slope of longitude wave travel time versus oil pressure changes from -0.096 to 0.058 ns/(kg/cm^2), the relative change is 165%, and the slope of the shear wave travel time versus oil pressure changes from .-0.211 to 0.086 ns/(kg/cm^2), the variation is 246%. If the sample pressure versus the oil pressure slope changed, both the slope of longitude and shear wave travel time versus oil

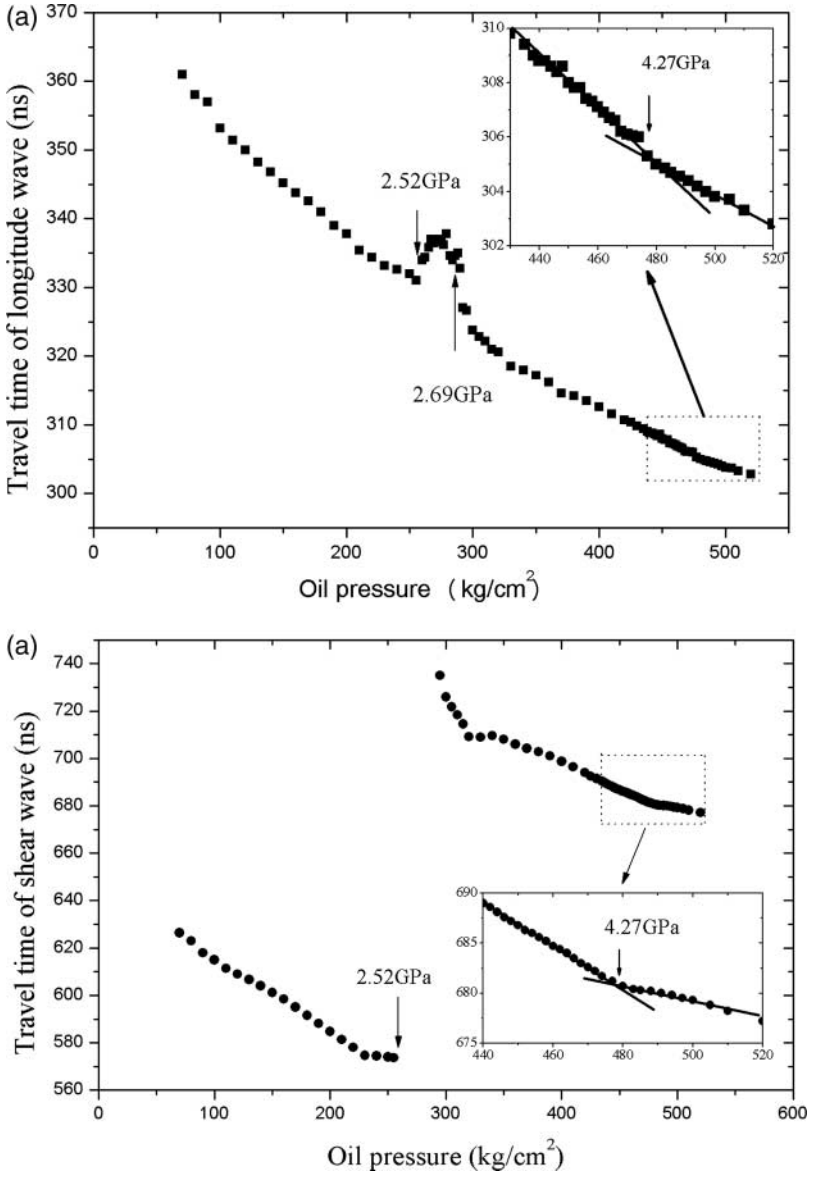

Figure 4. Travel times of sample under high pressure: (a) the travel time of the longitude wave under high pressure, and (b) the travel time of shear wave under high pressure.

pressure should change simultaneously. In fact, the changes of slope are great different in our experiments. So, it is considered that the III–IV phase transformation of bismuth happened at the oil pressure of 477 kg/cm^2. According to the three transformations mentioned above and zero pressure also included, the pressure calibration curve between oil pressure and sample pressure is shown in Figure 5. After polynomial fitting, the pressure calibration curve is expressed as $P = 0.0103 \times P_{\text{oil}} - 2.94 \times 10^{-6} \times P_{\text{oil}}^2$. The calibration curve is characterized by a high linear correlation fitting as seen in Figure 5.

The oil pressure is held at the pressure of 330 kg/cm^2 (3.09 GPa), 400 kg/cm^2 (3.66 GPa) and 520 kg/cm^2 (4.58 GPa) for heating, respectively. When the temperature of measurement over

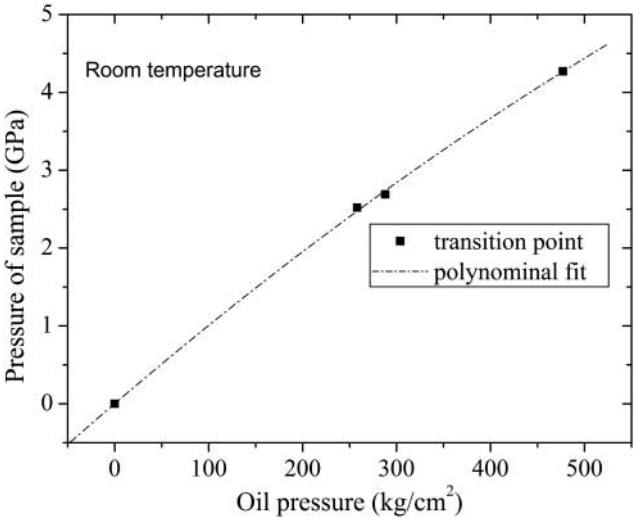

Figure 5. Relation between oil pressure of multi-anvil apparatus and sample pressure.

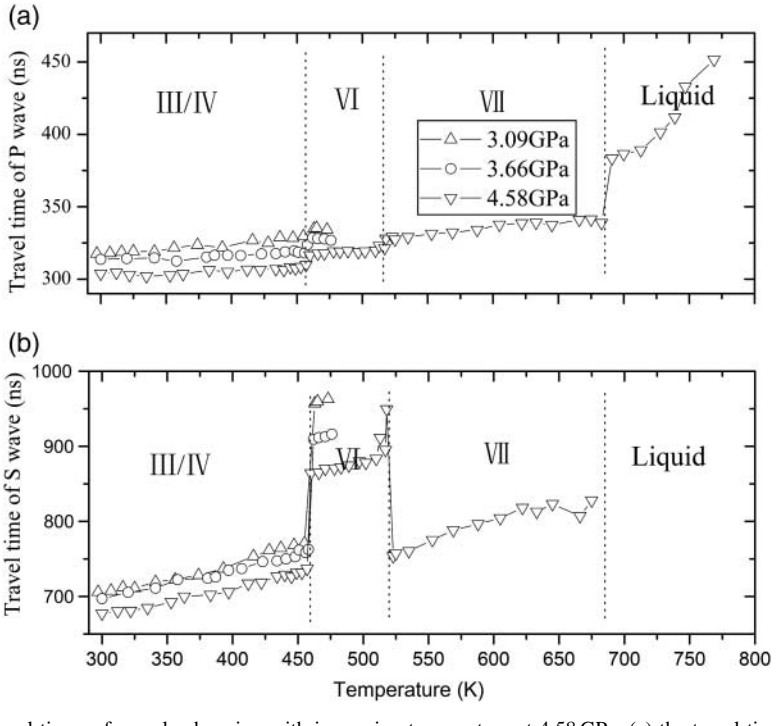

Figure 6. Travel times of sample changing with increasing temperature at 4.58 GPa: (a) the travel time of longitude wave, and (b) the travel time of shear wave.

457 K at 3.09 and 3.66 GPa, the travel times of both the longitude wave and the shear wave increase suddenly as shown in Figure 6. At 4.58 GPa, with the temperature increasing, bismuth changes from IV phase to VII phase, then to VI phase, and finally to the liquid phase in turn. The transformation temperatures of III–VII and IV–VII from phase graph are 448 K are unrelated to pressure. The transformation temperatures by the thermocouple we measured at three pressure

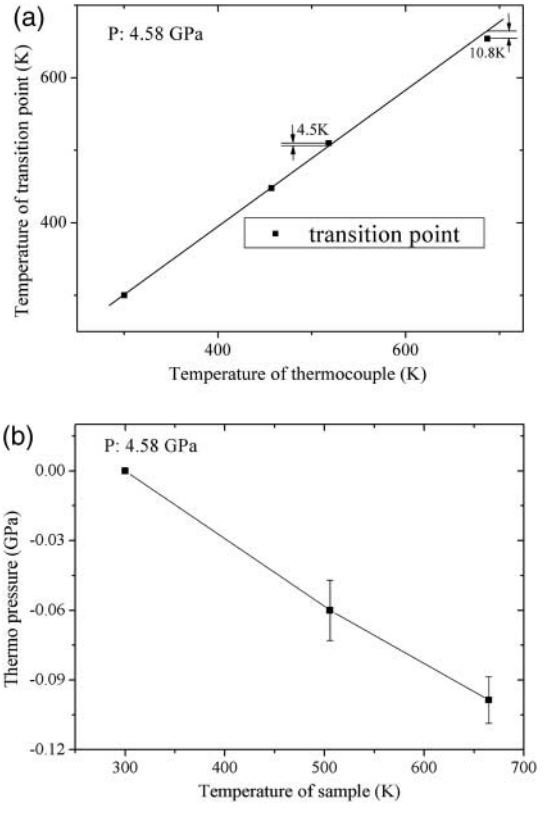

Figure 7. Temperature and thermo pressure calibration of sample by bismuth transformation point: (a) the temperature calibration of sample, and the temperature was just calibrated by room temperature and the transformation temperatures of III–VI and IV–VI (b) thermo pressure calibration of sample.

points are 457 K in Figure 6, and the sample temperature is about 9 K lower than the temperature monitored by the thermocouple. It is indicated that the temperature difference between the sample and the thermocouple was existed. It could be assumed that the temperature gradient is linear with temperature increasing, that the temperature difference between sample and measurement by thermocouple changes linearly with the temperature increasing. So, the sample temperature can be calibrated only by the room temperature and the transformation temperatures of III–VII and IV–VII as shown in Figure 7(a). Bismuth transforms from VII phase to VI phase at a temperature of 518 K, and transforms from VI phase to the liquid phase at the temperature of 687 K. After entering the liquid phase, we find that the travel time of longitude wave increases while the amplitude of signal becomes smaller and smaller, the shape of reflected signals of molten bismuth is distorted and the signal of the shear wave disappears completely. It can be considered that the shape of molten bismuth cannot maintain its original shape because of the surface tension. Naturally, the measurement uncertainty of the travel times of molten bismuth was great, and the travel times could be just used for characterize the melting point.

As we all know, heating could change the sample pressure which would influence the transformation temperature of VI–VII and VII-liquid. In Figure 7(a), we found that the VII-VI transformation point is 4.5 K higher than the fit line and the VI-liquid transformation point is 10.8 K lower than the fit line. According to the transformation boundary of VII-VI and VI-liquid and measurement temperatures, the thermal pressure is calibrated as seen in Figure 7(b). The error bar is presented according to the accuracy of temperature measurement of about 1 K. There are

two reasons influencing the sample pressure while heating the sample by holding pressure. On one hand, the volume of sample and pressure medium would expand with temperature increasing, and the pressure of sample will increase significantly. On the other hand, pressure medium softens with temperature and the pressure gradient of assembly would decrease. The oil pressure could remain unchanged for our multi-anvil apparatus had adaptive ability, and it is considered that the anvil pressure is unchanged. So, the pressure of the sample would decrease with temperature increasing because of pressure medium softening. The two reasons would counteract with each other, and the thermal pressure could be positive or negative for different sample assemblies. The result of negative thermal pressure of this experiment is very normal. The thermal pressure is negative with temperature increasing and the thermal pressure is about -0.1 GPa at 665 K. The volume is very small, and we can neglect the effect of thermal pressure if we use a similar assembly for ultrasonic measurement.

Conclusion

The experiment shows that the sample assembly of the hydrostatic pressure under HPHT is valid, and the pressure medium can be sealed around the sample in the range of 0–5 GPa and 300–750 K. The effect of thermo pressure of our assembly is weak, and it could be neglected if similar assembly is used for ultrasonic measurement. This method could be widely applied for 'soft' materials and also for fragile crystal samples under HTHP.

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