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2013 Chinese Phys. Lett. 30 054302

(http://iopscience.iop.org/0256-307X/30/5/054302)

CHIN. PHYS. LETT. Vol. 30, No. 5 (2013) 054302

Sound Velocity in Water and Ice up to 4.2 GPa and 500 K on Multi-Anvil Apparatus *

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(Received 4 February 2013)

A new assembly for ultrasonic measurements of water and ice on multi-anvil apparatus has been designed, and the ultrasonic compressional wave velocities in water and ice up to 4.2 GPa and 500 K are achieved. The pressure of the sample is calibrated by the melting curve of ice VII and the transformation pressure of liquid to solid at ambient temperature. The continuous changing process of the sound velocity transforming from water into ice at high pressure is achieved, and the experimental results of sound velocities at high pressure at room temperature on the melting curve of water are consistent with the previous works by Brillouin scattering. It is believed that our new method of ultrasonic measurements of water is reliable, and worth being used for studying more liquids at high pressure.

PACS: 43.35.Ae, 64.70.dj, 62.50.−p DOI: 10.1088/0256-307X/30/5/054302

Liquid water and ice are fundamental components of the Earth and other large icy planets. Water shows many unusual properties because of its polar covalent bonds. It exhibits a maximum in density at 4℃ above its melting point at normal pressure and a maximum in ultrasonic velocity at 74℃. Properties of water at high pressure have been extensively studied by using various techniques, including pressure-volume-temperature relations,[1,2] x-ray and Neutron diffraction,[3−6] Brillouin scattering,[7−13] Raman scattering,[14] ultrasonic measurements[15−17] and theoretical calculation.[18] The elastic modulus is the key to understanding various phenomena in the interior of these planets, while sound velocity measurement is the most powerful method to achieve the elastic modulus. The methods of sound velocity measurement at high pressure include the Brillouin scattering in DAC and traditional ultrasonic measurements on large volume apparatus. The Brillouin scattering in DAC was widely used for measuring the sound velocity of water and ice in the past decades. In the pressure and temperature (PT) ranges up to 25 GPa and 900 K, Asahara *et al.*[13] used a laser heated diamond anvil cell with a combined system of Brillouin scattering and synchrotron x-ray diffraction to study the elastic property of water.

Compared with Brillouin scattering, the traditional ultrasonic method has its advantage of a better signal to noise ratio (SNR) and precision. However, it has only ever been applied on piston-cylinder apparatus for ultrasonic measurements of water, and the PT range is limited to 1.7 GPa and 400 K.[16,17] Although it is very important for studying elastic modulus of liquids, the technique of ultrasonic measurements on multi-anvil apparatus has not been developed due to the technical difficulties of liquid sealing and the length measurement of a liquid sample.

In this Letter, based on the technique of liquid sealing on multi-anvil apparatus[19,20] and a length limiting technique for the ultrasonic measurement of melts, which has been developed recently,[21] a new assembly is designed for ultrasonic measurements of water and ice on multi-anvil apparatus up to 4.2 GPa and 500 K. The pressure of the sample is calibrated by the transformation of liquid water to ice phase VI and melting temperature of ice phase VII. The data of the ultrasonic velocity of water and ice are coordinated with previous works very well.

The experiments are performed on a multi-anvil apparatus (YL-800t), which is capable of generating pressures up to 6 GPa, at the Institute of Fluid Physics of Chinese Academic of Engineering Physics (IFP of CAEP), Mianyang, China.

The cross section of a new assembly for ultrasonic velocity measurements of liquid on multi-anvil apparatus is shown in Fig. 1. This assembly design is based on the hydrostatic pressure method by Wang *et al.*[19,20] and also on the ultrasonic measurement method for melts by Song *et al.*[21] In this study, tungsten carbide (WC, 8 mm diameter, 6 mm length) with a groove (3 mm width, 1.5 mm depth) is used for limiting the length of the sample, and it is af-

*Supported by the National Natural Science Foundation of China under Grant No 40974052, and the National Key Laboratory for Shock Wave and Detonation Physics under Grant No LSD201201002.

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fixed to the polycrystalline alumina ceramics buffer (10 mm diameter, 12 mm length) by inorganic glue at the side. There is no bonding material in the interface between the WC and the ceramics buffer. The previous experiments[19] of ultrasonic measurements demonstrated that no glue will be squeezed into the contact interface if the buffer rod and sample are well polished and the roughness is controlled less than 0.2 micron. The surrounding of WC is filled with water, and a copper sleeve is used to prevent the water from extruding out into the pyrophyllite. A thermocouple is placed at the bottom of the copper sleeve for measuring the temperature of water. There is little difference between the measured temperature with thermocouple and the real temperature of the sample. The measured temperature by the thermocouple is about 10 K[20] higher than the sample temperature at 500 K, and the sample temperature is revised.

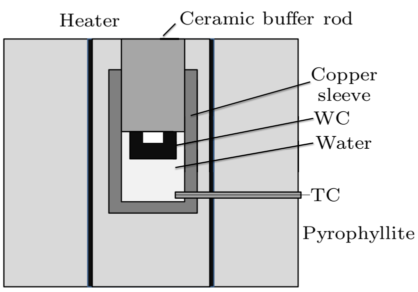

Fig. 1. Cross section of the new assembly for ultrasonic velocity measurements of liquid on multi-anvil apparatus

The pressure and temperature for the loading and subsequently for heating and cooling circles in ultrasonic measurements for water is shown as a dotted line in Fig. 2. The pressure is increased slowly to about 1.2 GPa of ice phase VI at ambient temperature. Then temperature is increased to the melting point of water and decreased at several constant loads.

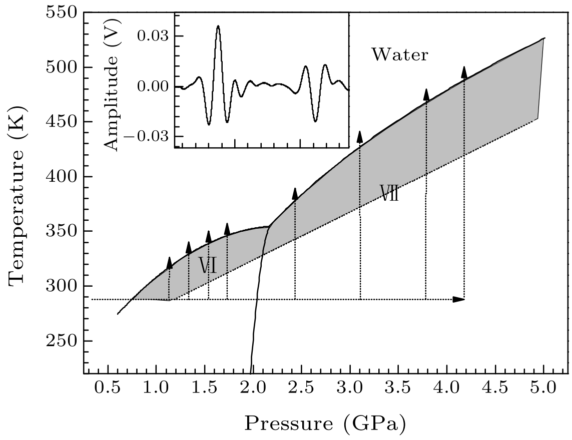

Fig. 2. Loading path of this study. The dashed line is the loading path, the shadowed area is the combined phase area of water and ice, and classical ultrasonic signal of ice is in the top left corner.

The method of ultrasonic measurements has been described by Liu *et al.*[22,23] The typical ultrasonic signals are shown at the upper left corner of Fig. 2. The method of length limiting and correction has been described in more detail elsewhere.[21]

Briefly, the crucial idea to limit the thickness of a liquid sample is the use of cylindrical WC with a groove as an acoustic reflector. According to deformation theory, hydrostatic stress will not contribute to plastic yielding of materials. Thus, the dimension change of a WC reflector under hydrostatic pressure can be estimated by the well known stress-strain relationship for its elastic behavior.

Table.1 Sound velocity in water and ice at high pressure and high temperature.

P (GPa)	T (K)	t (μs)	L (mm)	V_P (km·s^{-1})	P (GPa)	T (K)	t (μs)	L (mm)	V_P (km·s^{-1})
0.47	293	1.336	1.4994	2.244	3.25	293	0.574	1.4958	5.208
0.53	293	1.285	1.4993	2.333	3.47	293	0.563	1.4956	5.310
0.70	293	1.193	1.4991	2.513	3.67	293	0.560	1.4953	5.338
0.80	293	1.149	1.4990	2.609	3.87	293	0.557	1.4950	5.367
0.89	293	1.042	1.4989	2.876	4.05	293	0.550	1.4948	5.433
0.97	293	0.731	1.4988	4.098	4.11	293	0.551	1.4947	5.425
1.10	293	0.677	1.4986	4.427	4.23	293	0.547	1.4946	5.464
1.24	293	0.653	1.4984	4.587	1.14	311	1.076	1.4985	2.785
1.44	293	0.639	1.4982	4.688	1.34	333	1.032	1.4983	2.904
1.63	293	0.627	1.4979	4.777	1.54	351	0.976	1.4980	3.070
1.78	293	0.623	1.4977	4.808	1.73	353	0.940	1.4978	3.187
2.00	293	0.611	1.4974	4.902	2.45	391	0.860	1.4969	3.481
2.17	293	0.609	1.4972	4.918	3.10	435	0.775	1.4961	3.714
2.36	293	0.599	1.4970	5.000	3.73	467	0.732	1.4953	3.904
2.79	293	0.581	1.4964	5.155	4.23	481	0.730	1.4946	3.970

The travel time of a sound wave in a water sample varies obviously with the changing of pressure and temperature as shown in Fig. 3. It is thus evident that the water transforms into ice VI for an abrupt change of travel time. It is hard to judge the transition pressure from ice VI to ice VII because of no obvious change of travel time in the phase boundary. This is consistent with a previous study on crystal ice by Brillouin scattering[7] which also showed that there is no abrupt change of the bulk modulus from ice VI to ice VII. Holding pressure at different points, with the increasing temperature, the travel time of the sample increases stably and slowly at first, then change rapidly like a parabola, and lastly shows an inflexion point of travel time which is believed to be the point of complete melting. Part of the data in this test about travel time, sample length and sound velocity in water and ice at high pressure and high temperature are listed in Table 1. The sample length is calculated by the EOS of tungsten carbide.[28] As shown in Fig. 2, the shadowed area is a combined phase of liquid water and solid ice, in which the travel time changes like a parabola. The area in which the travel time increases rapidly is regarded as the boundary of the combined phase and the pure ice phase. The melting curve of ice VII has been studied accurately by Datchi.[24] Thus, the pressure of sample could be calibrated by the melting curve of ice VII and the transition pressure of liquid water to ice VI. This calibrated pressure varies as a function of oil pressure of the apparatus by polynominal fitting as shown in Fig. 4.

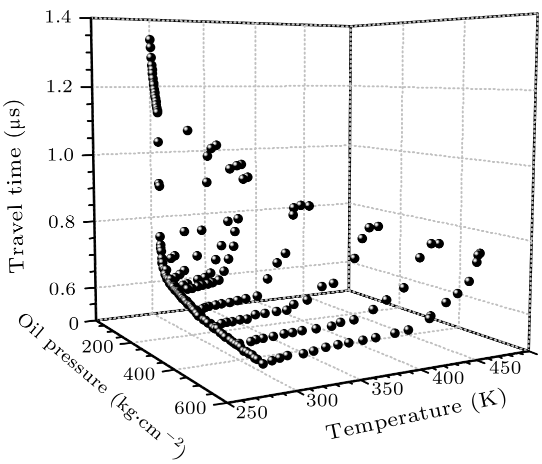

Fig. 3. Travel time of the sample at high pressure and high temperature.

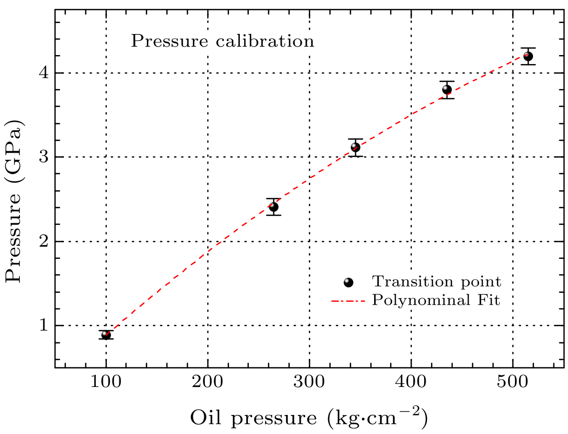

Fig. 4. Pressure calibration of the sample by the melting curve of ice and the transformation pressure of water to ice.

Sound velocities of water and ice at high pressure have been measured by different studies.[7−13] The sound velocity data of previous work are respectively plotted as open circles,[11,12] inverted triangles[7] and triangles[10] in Fig. 5 and the data of this study are plotted as filled circles. Previous data about water/ice are all measured by Brillouin scattering. The elastic moduli of single crystal ice VI and VII have been studied by Shimizu.[11,12] According to the C_{11} and density of ice,[25] we transform the elastic moduli of ice into sound velocities for comparison with our data. The result coincides with previous works very well. The previous sound velocity data of water and ice by Brillouin scattering were just done in separate experiments. Thus, the process of transformation from water into ice could not be described demonstratively. In this study, the sound velocity changing process of water transforming into ice with increasing pressure is easily achieved as shown in Fig. 5. It is clear that the sound velocity of water changes abruptly when water transforms into ice with pressure increasing.

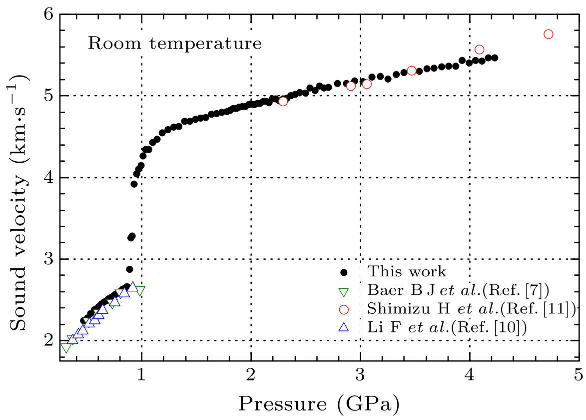

Fig. 5. Sound velocity at high pressure at ambient temperature up to 4.2 GPa. Filled circles, open circles, open converse triangles, and open triangles represent the measured sound velocities in this study, Shimizu(1996), Baer(1998) and Li(2005), respectively.

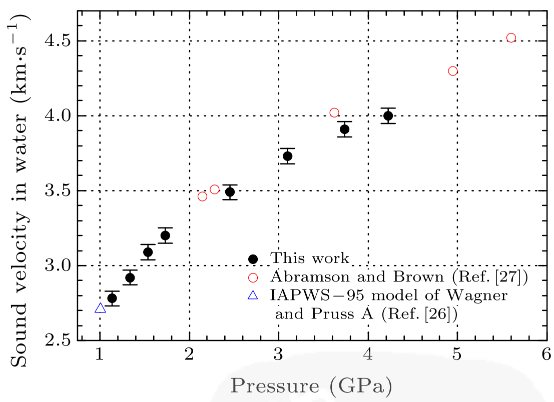

Fig. 6. Measured sound velocities in water on the melting curve of ice. Filled circles, open circles, and open triangles represent measured sound velocities in this study, Abramson and Brown(2004), and the IAPWS-95 model of Wagner and Pruβ(2002), respectively.

Figure 6 shows the relation of the sound velocities and pressure on the melting curve. There is little difference with our results and previous results obtained by Brillouin scattering with the externally heated diamond cell technique.[27] Asahara *et al.*[13] considered that sound velocity on a melting curve obtained by Brillouin scattering has relatively large uncertainty in pressure and temperature.

The uncertainty of ultrasonic velocity mainly concludes the error of travel time and the depth of the WC groove. The SNR of the ultrasonic echo signal is very nice, the travel time error is about 1 ns, and the relative uncertainty of the travel time is less than 0.2%. The depth error of the WC groove is about 3 μm, and the relative uncertainty is less than 0.2%. Considering the system error, it is believed that the whole uncertainty of sound velocity is about 1%.

In summary, we have presented a new assembly for ultrasonic velocity measurements of water/ice on multi-anvil apparatus up to 4.2 GPa and 500 K by using an ultrasonic pulse-echo method. The continuous changing process of the sound velocity from water transforming into ice at high pressure is achieved in our experiments. The sound velocities respectively of water and ice at high pressure at room temperature and of water on the melting curve are consistent with previous results by Brillouin scattering. It is believed that our system of ultrasonic measurements is reliable, and it is worthy to be used for studying more liquids at high pressure.

We thank Professor Zhou Xianmin and Professor Zhu Wenjun at the Institute of Fluid Physics for helpful discussions.

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