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Ultrasonic experimental study on the elasticity of aluminum to 4.1 GPa in multi-anvil apparatus

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ABSTRACT

Ultrasonic experiments have been performed to measure compressional and shear wave velocities of polycrystalline aluminum under hydrostatic pressure up to 4.1 GPa at room temperature in a multi-anvil apparatus. The sample pressure was determined by the new Z-cut quartz calibrant. Two types of data processing methods, three-order finite strain method and the Anderson's method, were utilized to calculate the elasticity of aluminum. The results from this work are in good agreement with previously reports and so it demonstrates the accuracy and convenience of our experimental methods, including the validity of the Z-cut quartz calibrant for pressure determination. We believe it's valuable for the measurements of elasticity of other material in multi-anvil apparatus, especially those experiments using compressible specimens which have small elastic moduli, and lacking of X-ray source.

Credit Author Statement

Wei Song: Methodology, Investigation, Writing – original draft. Qizhe Tang: Resources, Data curation. Chang Su: Resources, Formal analysis. Xiang Chen: Investigation. Yonggang Liu: Conceptualization, Writing – original draft, Supervision, Funding acquisition.

1. Introduction

Aluminum is one of the most important metals and for its exceptional properties in mechanics, thermology, electricity and so on, aluminum and its alloy have become the cornerstone material of modern industry. There are considerable studies on its behavior at high pressure, including X-ray diffraction [1–3], neutron diffraction [4], shock-wave experiments [5–8], volumetrically [9,10], measurements of elastic constants [11–26], melting curve determinations [27–29] and theoretical calculations [30–38]. Note that, in some high-pressure X-ray/neutron diffraction studies, it is a routine procedure to determine pressure by measuring the lattice parameters of a 'marker' (such as NaCl, Ag, MgO, etc.) together with those of the sample and aluminum is also chosen as a calibrant.

Elastic bulk (K_S) and shear (G) moduli and their pressure derivatives are important parameters in understanding the structural behavior and physical properties of materials under compression. Previously, the ultrasonic elasticity of aluminum was mainly measured using its single crystal and the pressure was limited to below 1 GPa. For polycrystalline aluminum, probably limited by experimental techniques or/and unavailability of fully dense polycrystalline specimens, there are relatively few experimental studies on its elasticity and so effective polycrystalline elastic constants usually be calculated from single crystal data by theoretical expressions [30], deriving equation to estimate [31] or averaging procedure (e.g., Voigt-Reuss-Hill averages) [32]. Although the accuracy of averaging procedure is normally higher than that of the first two processes, as Guinan and Steinberg [31] have pointed out, it's fairly confusing during the converting because of the existence of several nomenclatures and the tedious numerical manipulation. On the other hand, ultrasonic measurements on polycrystalline specimens are simpler in principle and of more practical use than those on single crystal specimens [30]. Moreover, in the literature, data on the compressional (V_p) and shear wave (V_s) velocities of polycrystalline aluminum under high pressure is surprisingly scarce. At present, the ultrasonic velocity of material in multi-anvil apparatus (MAA) is normally measured using solid inner pressure medium (e.g. NaCl, BN) to afford quasi-hydrostatic pressure environment. When it comes to the specimens that with small

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elastic moduli, the X-ray source is essential, since it can obtain the change of specimen length under high pressure (X-radiography). Recently, the ultrasonic techniques developed in our laboratory made it's realistic to measure the V_p, V_s simultaneously and hence the elastic properties of material under hydrostatic pressure in MAA.

In this paper, we present the measured results of V_p and V_s of polycrystalline aluminum and its elastic properties up to 4.1 GPa. Meanwhile, the sample pressure in this study was determined based on a new Z-cut α-quartz calibrant, thereby its validity can be tested by the ultrasonic results.

2. Experiment method

The ultrasonic experiments are performed in a cubic-type MAA (CS3600t), in which the six anvils can be driven separately by a multi-axial system of hydraulic rams. This MAA is capable of generating oil pressures up to 90 MPa (about 4 GPa), installed at the Key Laboratory for High-Temperature and High-Pressure Study of the Earth's Interior, Institute of Geochemistry, Chinese Academy of Sciences, China. The schematic diagram of the sample assembly is shown in Fig. 1.

The polycrystalline aluminum sample (99.99% purity) with a diameter of 5.0 mm and length of 2.61 mm was used in this investigation. Using the Archimedes' method, a bulk density of 2.70 g/cm^3 was obtained. Another sample, Z-cut quartz single crystal ($\rho_0 = 2.65$ g/cm^3) with a diameter of 5.0 mm and length of 1.81 mm was used to pressure calibration. Sample is affixed to the polycrystalline alumina ceramics buffer rod (8 mm in diameter, 12 mm in length) by inorganic glue at the side. The surrounding of sample is filled with silicon oil to provide hydrostatic pressure environment and after measurements, the shape of recovered sample remained the same. To minimize the acoustic energy loss, all contact interfaces include the WC anvil, buffer rod, and solid sample are well polished before the measurements and this is also critical to ensure no material, neither surrounding silicon oil nor glue, will be squeezed into the contact interfaces between the buffer rod and solid sample under pressure.

The ultrasonic travel times are measured with the classical pulse-echo method by using a digital oscilloscope (Tektronix DPO2024B, USA), a CTS-8077PR ultrasonic pulse generator/receiver unit (Guangdong Goworld Co., Ltd., Shantou, China) and a dual-mode ultrasonic transducer (PANAMETRICS-NDT X1013, USA) which can produce 10 MHz longitudinal wave and 5 MHz shear wave simultaneously.

The sample pressure in this study was determined based on a new Z-cut α-quartz calibrant [39], as briefly presented below. For Z-cut α-quartz, we can get the following equation:

$$v_{33} = \sqrt{\frac{c_{33}}{\rho}} \tag{1}$$

$$c_{33} = c_{33}^0 + c_{33}^{'} P \tag{2}$$

$$t = \frac{2l}{v_{33}} \tag{3}$$

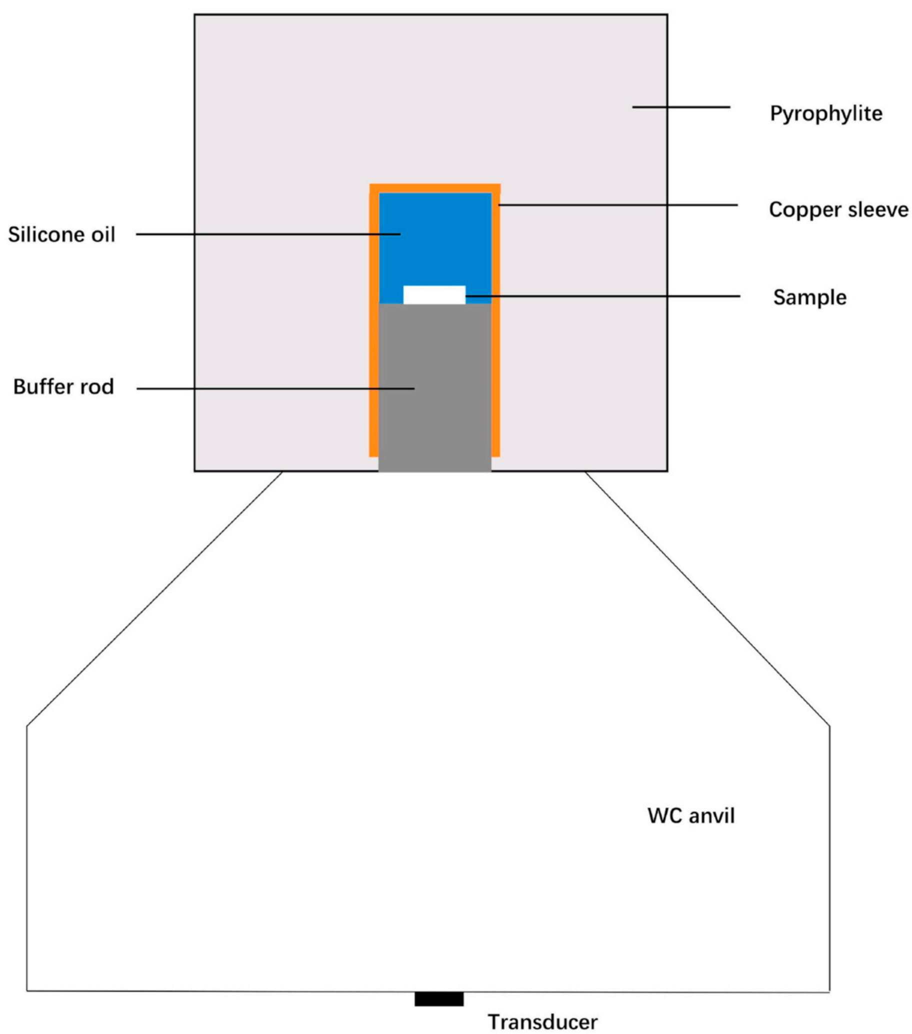

Fig. 1. The schematic diagram of the sample assembly used for ultrasonic measurements in this work.

$$\frac{\rho}{\rho_0}=\frac{V_0}{V} \tag{4}$$

where v_{33} is compressional wave sound velocity in Z direction, ρ is density, V is volume, subscript '0' represents values at zero pressure, c_{33} is the elastic modulus in Z direction, c_{33}^0 and $c_{33}^{'}$ are elastic modulus at zero pressure and its pressure derivative, both are available from McSkimin et al. [40], P is pressure, t is the travel time in quartz, l is the length of quartz under high pressure, which can be obtained by polynomial fitting data of Angel et al. [41] on the unit-cell parameters of quartz:

$$\frac{l}{l_0}=1-0.00729P+0.00068P^2-0.000005P^3+0.00000014537P^4 \tag{5}$$

and the relation between V/V_0 and l/l_0 also can be obtained:

$$\frac{V}{V_0}=-20.013+38.544\left(\frac{l}{l_0}\right)-17.531\left(\frac{l}{l_0}\right)^2 \tag{6}$$

Then using equations (1)–(4), we will find:

$$t=\frac{2l}{\sqrt{\frac{c_{33}^0+c_{33}^{'}P}{\rho_0\left(\frac{V}{V_0}\right)^{-1}}}} \tag{7}$$

as l_0, ρ_0, c_{33}^0 ,$c_{33}^{'}$ are known, using equations (5)–(7), we can calculate the travel time t by increasing pressure P from zero to high pressure, with a 0.01 GPa interval. When the calculated travel time matches precisely (accurate to 0.1 ns) the measured travel time at a specific oil pressure in ultrasonic experiment, the sample pressure can be calibrated and the results is shown in Fig. 2. After polynomial fitting, the pressure calibration curve is expressed as $P(GPa) = -0.2605 + 0.0893P_{oil}(MPa) - 0.0004P_{oil}^2(MPa)$, where P_{oil} is oil pressure.

3. Results and discussion

While ultrasonic experiments can obtain precise measurements of travel times, the length of the sample is needed for the calculation of wave velocities at high pressure. With the measured P and S wave travel times in aluminum, the length change of aluminum sample at high pressures can be obtained by an approach known as the Cook's method [42,43]:

$$\frac{l_0}{l}=1+\frac{1+\alpha\gamma T}{12\rho_0 l_0^2}\int_0^P\frac{dP}{\left(\frac{1}{t_p^2}-\frac{4}{3t_s^2}\right)} \tag{8}$$

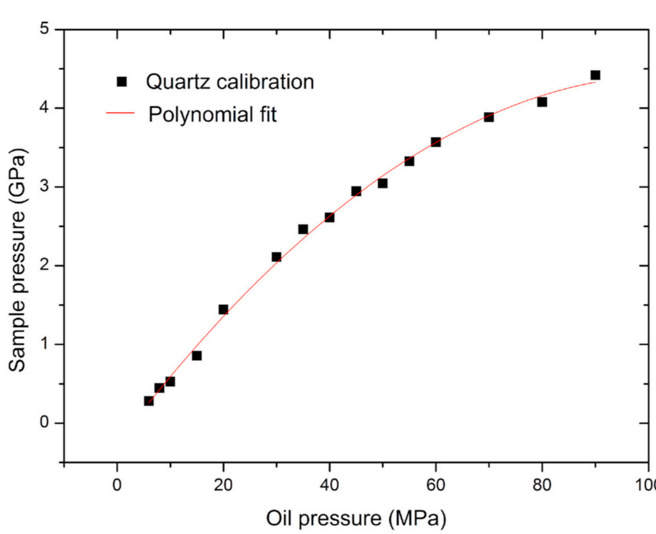

Fig. 2. Sample pressure determined by quartz calibration.

where P is pressure, t_p and t_s are the compressional wave and shear wave travel times in sample, l_0 and l is the length of sample under zero pressure and high pressure, ρ_0 is density under zero pressure, α is the thermal expansion coefficient, γ is the Grüneisen parameter, T is absolute temperature. When T = 295 K, as α and γ are available from ref. [1], we can get $1+\alpha\gamma T\approx 0.04$. Using the length l from equation (8) and the measured travel times, the V_p, V_s, ρ, K_s and G can be calculated ($V_p = 2l/t_p V_s = 2l/t_s$, $\rho = \rho_0(V_0/V) = \rho_0(l_0/l)^3$, $K_s = \rho(V_p^2 - 4V_s^2/3)$, $G = \rho V_s^2$) at all pressures (Table 1). As shown in Fig. 3, the V_p and V_s linearly increase with pressure at room temperature. After linear fitting, the measured results of V_p and V_s can be expressed as $V_p(km/s) = 0.164P(GPa) + 6.41$ and $V_s(km/s) = 0.096P(GPa) + 3.14$, respectively.

Meanwhile, the change of volume at high pressure also can be obtained and the comparisons with data from static compression experiments [1,9,10] and shock-wave experiments [5] are shown in Fig. 4. Agreement among these data is excellent, except Vaidya and Kennedy's data [10], which are systematically slightly higher.

To obtain the zero-pressure adiabatic bulk and shear moduli as well as their pressure derivatives, we can use the third-order finite strain (FS) approach, which is fitting the velocity and density data simultaneously to the following three equations [43]:

$$\rho V_p^2=(1-2\varepsilon)^{5/2}(L_1+L_2\varepsilon) \tag{9}$$

$$\rho V_s^2=(1-2\varepsilon)^{5/2}(M_1+M_2\varepsilon) \tag{10}$$

$$P=-3K_{t0}(1-2\varepsilon)^{5/2}(1+3(4-K_{t0}^{'})\varepsilon/2)\varepsilon \tag{11}$$

where the stain ε is defined as $\varepsilon = (1-(\rho/\rho_0)^{2/3})/2$, and the fitted coefficients L_1, L_2, M_1, and M_2 can be expressed with the elastic constants as:$L_1 = K_{so} + 4G_0/3$,$L_2 = 5(K_{s0} + 4G_0/3) - 3K_{s0}(K_{s0}^{'} + 4G_0^{'}/3)$, $M_1 = G_0$, $M_2 = 5G_0 - 3K_{so}G_0^{'}$. Then the elastic constants can be calculated. The zero pressure adiabatic bulk and shear moduli and their pressure derivatives are determined to be$K_{s0} = 75.0$GPa,$G_0 = 26.5$GPa, $K_{s0}^{'} = 4.87$,$G_0^{'} = 2.16$. The zero pressure isothermal bulk moduli and its pressure derivative are determined to be $K_{t0} = 72.3$ GPa, $K_{t0}^{'} = 4.67$.

Another method to obtain elastic moduli and their pressure derivatives using ultrasonic data was introduced by Anderson [44,45], which is expressed as:

$$K_{s0}=\rho_0(V_{p0}^2-4V_{s0}^2/3) \tag{12}$$

$$G_0=\rho_0 V_{s0}^2 \tag{13}$$

$$K_{t0}=K_{s0}(1+\alpha\gamma T)^{-1} \tag{14}$$

$$K_{s0}^{'}=2\rho_0(V_{p0}^{'}V_{P0}-4V_{s0}^{'}V_{s0}/3)+1+\alpha\gamma T \tag{15}$$

$$G_0^{'}=2\rho_0 V_{s0}V_{s0}^{'}+G_0/K_{t0} \tag{16}$$

$$K_{t0}^{'}=K_{s0}^{'}+\alpha\gamma T\left(\frac{K_{t0}}{K_{s0}}\right)\left[1-\frac{2}{\alpha K_{t0}}\left(\frac{\partial K_{t0}}{\partial T}\right)_p-2K_{s0}^{'}\right] + \left[\alpha\gamma T\left(\frac{K_{t0}}{K_{s0}}\right)\right]^2\left[K_{s0}^{'}-1-\frac{1}{\alpha^2}\left(\frac{\partial\alpha}{\partial T}\right)_p\right] \tag{17}$$

Where V_{p0}, V_{s0}, $V_{p0}^{'}$ and $V_{s0}^{'}$ is zero pressure compressional velocity, shear velocity and their pressure derivatives, which can be obtained from linear fitting of velocities data (Fig. 2). As $\left(\frac{\partial K_{t0}}{\partial T}\right)_p$ and $\left(\frac{\partial\alpha}{\partial T}\right)_p$ are available from literature [44], the elasticity can be calculated. Using this method, The zero pressure adiabatic bulk and shear moduli and their pressure derivatives are determined to be$K_{s0} = 75.4$GPa,$G_0 = 26.6$GPa, $K_{s0}^{'} = 4.57$,$G_0^{'} = 1.99$. The zero pressure isothermal bulk moduli and its

Table 1
Experimental data and calculated ultrasonic results of aluminum. The travel time error is 1 ns, and the relative uncertainty is less than 0.2%. The uncertainty is about 0.5% for V_p, V_s and 1.5% for elastic moduli.

P (GPa)	*l* (mm)	*tp* (μs)	*ts* (μs)	*Vp* (km/s)	*Vs* (km/s)	*ρ* (g/cm3)	*Ks* (GPa)	*G* (GPa)
0.26	2.6169	0.8110	1.6630	6.453	3.147	2.710	77.1	26.8
0.59	2.6130	0.8045	1.6310	6.496	3.204	2.722	77.6	27.9
0.99	2.6084	0.7955	1.6105	6.558	3.239	2.736	79.4	28.7
1.29	2.6051	0.7855	1.5890	6.633	3.279	2.747	81.5	29.5
1.72	2.6003	0.7755	1.5665	6.706	3.320	2.762	83.6	30.4
2.06	2.5968	0.7705	1.5490	6.740	3.353	2.773	84.4	31.2
2.38	2.5934	0.7620	1.5365	6.807	3.376	2.784	86.7	31.7
2.67	2.5903	0.7545	1.5225	6.866	3.403	2.794	88.6	32.3
2.95	2.5876	0.7500	1.5100	6.900	3.427	2.803	89.6	32.9
3.20	2.5850	0.7445	1.5000	6.944	3.447	2.811	91.0	33.4
3.44	2.5827	0.7405	1.4905	6.976	3.466	2.819	92.0	33.9
3.66	2.5806	0.7375	1.4800	6.998	3.487	2.826	92.6	34.4
4.03	2.5770	0.7285	1.4635	7.075	3.522	2.837	95.1	35.2
4.19	2.5756	0.7270	1.4495	7.085	3.554	2.842	94.8	35.9

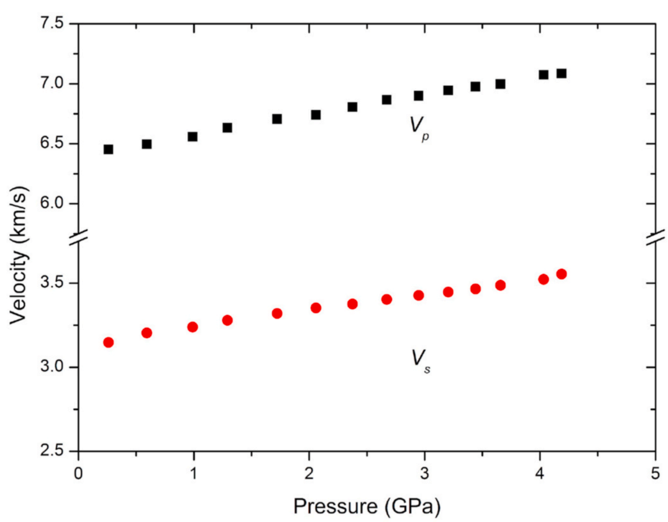

Fig. 3. Compressional and shear wave velocity of aluminum as a function of pressure at room temperature.

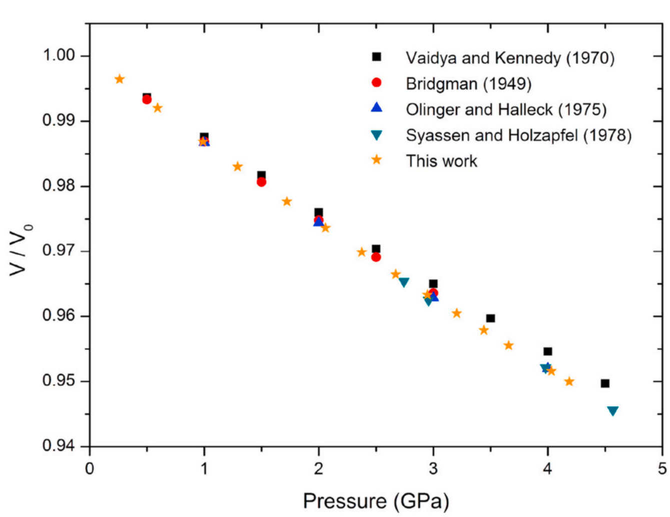

Fig. 4. Volume change of aluminum as a function of pressure.

pressure derivative are determined to be $K_{t0} = 72.5$ GPa, $K_{t0}^{'} = 5.09$. Those results are compared with previous results in Table 2.

As compared in Table 2, in general, our results of elastic moduli from two data processing methods, third-order finite strain and the Anderson's method, are in good agreement with the previous values, within their respective error range (about 1%–2%). The pressure derivatives of bulk moduli from previous results differ mainly in a small range, that is 4.4–5.19 for $K_{s0}^{'}$ and 4.3–5.3 for $K_{t0}^{'}$. Since the pressure derivative of elasticity is sensitive to pressure and velocities, this dispersion is common, even in the same type of experiments. Our calculated values 4.57 and 4.87 for $K_{s0}^{'}$, 4.67 and 5.09 for $K_{t0}^{'}$ are located in the corresponding range. In terms of pressure derivative of shear moduli, our experiment results 1.99 and 2.16 for $G_0^{'}$, are in good agreement with corresponding value of 2.0 from Voronov and Vereshchagin [15] and 2.07 from Trappeniers et al. [20] and 2.17 from Witczak et al. [24] and slightly higher than the corresponding value of 1.75 from Guinan and Steinberg [31], which was obtained by equation estimate using ultrasonic data from Thomas [17]. However, if we use our two sets of ultrasonic data, including K_{s0}, G_0, K_{t0} and $K_{s0}^{'}$, to perform the estimate in their equation, we can obtain $G_0^{'} = 1.73$and $G_0^{'} = 1.76$, respectively, which are very close to their assessed value. The reason for this abnormity is not clear, but the large error range (>10%) in their estimate process may be a possible explanation.

The Debye temperature is an important physical constant of matter, we also calculated the Debye temperature of aluminum using the equation:

$$\theta_D = \frac{h}{k}\left(\frac{3N}{4\pi}\right)^{1/3}\left(\frac{\rho}{M/q}\right)^{1/3}\left(\frac{2}{3V_s^3}+\frac{1}{3V_p^3}\right)^{-1/3} \quad (18)$$

where *h*, *k* and *N* are Planck constant, Boltzmann constant and Avogadro number; *M* is the molecular mass, *q* is the number of atoms in the molecular formula; V_p and V_s are the compressional and shear velocity. θ_{D0} at room temperature is determined to be 412 K (the V_{p0}, V_{s0} values were obtained by linear fitting the sound velocities under high pressure) and it is in good agreement with the corresponding value of 410 K and 408 K, calculated by Flinn and McManus [46] and Witczak et al. [24], respectively.

4. Conclusion

The compressional and shear wave velocities of polycrystalline aluminum under hydrostatic pressure up to 4.1 GPa were measured at room temperature. Using two types of data processing methods, three-order finite strain method and the Anderson's method, the ultrasonic elasticity of polycrystalline aluminum was obtained and it is in good agreement with previous results from single crystal or polycrystalline aluminum experiments. Accordingly, it demonstrates that (1) the accuracy of our experimental methods, especially the validity of the Z-cut quartz calibrant for pressure determination used in this work (2) the elastic moduli determined on polycrystalline aluminum from this work

Table 2
Elastic moduli and their pressure derivatives of aluminum. The unit of elastic moduli is in GPa.

Refs.	K_{s0}	G_0	K'_{s0}	G'_0	K_{t0}	K'_{t0}	Notes
1					72.7	4.30	X-ray diffraction, to 12 GPa
2					71.7		X-ray diffraction, to 20 GPa, combine ultrasonic data
4					72.8		Neutron diffraction, to 5.7 GPa
11					74.5	4.04	Single crystal, ultrasonic, to 1 GPa
12	75.3				72.6		Calculation
13	75.7	26.5	4.0	2.6			Polycrystal, ultrasonic, to 0.9 GPa
14	76.4		5.19		72.7	5.31	Single crystal, ultrasonic, to 0.65 GPa
15	76.9	25.0	4.75	2.0			Polycrystal, ultrasonic, to 1 GPa
17	75.9				72.7	4.54	Single crystal, ultrasonic
18	76		5.11		72.9	5.15	Single crystal, ultrasonic, to 0.4 GPa
20		26.4		2.07			Single crystal, Resonance, to 0.25 GPa
22	76.6		4.85		73.0	5.01	Single crystal, Resonance, to 0.25 GPa
23	76				72.4		Single crystal, composite oscillator
24		26.0		2.17	75.0	4.50	Polycrystal, ultrasonic, to 1 GPa
26					72.6		Lattice vibrational method
30				2.01		5.22	Calculation using ultrasonic data
31				1.75			Equation to estimate, combine ultrasonic data
34					70.2		Calculation
This work	75.0	26.5	4.87	2.16	72.3	4.67	Polycrystal, ultrasonic, 3rd finite strain, to 4.1 GPa
This work	75.4	26.6	4.57	1.99	72.5	5.09	Polycrystal, ultrasonic, Anderson's method, to 4.1 GPa

are in good agreement with the corresponding values measured on single crystals, but our method is more convenient and ready for use and should be valuable for the measurements of elasticity of other material, especially those experiments using compressible specimens which have small elastic moduli and lacking of X-ray source (3) the three-order finite strain method seems to be more convenient than the Anderson's method, since the latter needs more parameters to calculate elastic moduli and their pressure derivatives.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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