Comparative experimental study on several methods for measuring elastic wave velocities in rocks at high pressure

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Abstract To measure elastic wave velocities in rocks at high temperature and high pressure is an important way to acquire the mechanics and thermodynamics data of rocks in the earth's interior and also a substantial approach to studying the structure and composition of materials there. In recent years, a rapid progress has been made in methodology pertaining to the measurements of elastic wave velocities in rocks at high temperature and high pressure with solids as the pressure-transfer media. However, no strict comparisons have been made of the elastic wave velocity data of rocks measured at high temperature and high pressure by various laboratories. In order to compare the experimental results from various laboratories, we have conducted a comparative experimental study on three measuring methods and made a strict comparison with the results obtained by using the transmission method with fluid as the pressure-transfer medium. Our experimental results have shown that the measurements obtained by the three methods are comparable in the pressure ranges of their application. The cubic sample pulse transmission method used by Kern is applicable to measuring elastic wave velocities in crustal rocks at lower temperature and lower pressure. The prism sample pulse reflection-transmission method has some advantages in pressure range, heating temperature and measuring precision. Although the measurements obtained under relatively low pressure conditions by the prism sample pulse transmission method are relatively low in precision, the samples are large in length and their assemblage is simple. So this method is suitable to the experiments that require large quantities of samples and higher pressures. Therefore, in practical application the latter two methods are usually recommended because their measurements can be mutually corrected and supplemented.

Keywords: elastic wave velocity, rock, high temperature and high pressure, measuring method.

Laboratory measurement of elastic wave velocities in rocks at high temperature and high pressure is an important approach to acquiring a lot of mechanics and thermodynamics data of rocks in the earth's interior. Comparisons of the experimental results with the seismic wave observations are of great significance in the exploration of the internal structure and composition of the earth. For this reason, a number of laboratories pertaining to high-pressure in the geological field attach great importance to the development of methods for measuring elastic wave velocities in rocks at high temperature and high pressure[1—4]. At present, there are many methods for measuring elastic wave velocities in rocks at high temperature and high pressure. According to the

pressure-transfer media, two broad categories of measuring methods can be classified: the methods with fluids as the pressure-transfer media and those with solids as the pressure-transfer media. The former can guarantee a smooth measuring of elastic wave velocities in rock samples under strictly controlled hydrostatic pressures. However, this category of methods have not been adopted by most laboratories because of lower pressure and temperature realized (at present time the maximum pressure only reaches 3.0 GPa; the maximum temperature, 300℃) and complex sample assemblage. To raise the experimental pressure and temperature, in recent years, the measuring methods with solids as the pressure-transfer media have witnessed a rapid development and thereafter a great wealth of experimental data on elastic wave velocities in rocks have been acquired using these methods. For example, Song et al. have studied the elastic wave velocities in alkaline-olivine basalt collected from Inner Mongolia and in olivine and clinopyroxene of the upper mantle xenolith[5,6]; Yang et al. have measured the elastic wave velocities in granite collected from South China[7]; Gao et al. and Kern et al. have measured the elastic wave velocities in eclogite from the Dabie ultrahigh-pressure metamorphic belt, Central China[8,9]. In order to open up a possibility to share and comprehensively compare the data obtained by different measuring methods, a comparative study has been conducted on the three methods of measuring elastic wave velocities in rocks with solids as the pressure-transfer media at high temperature and high pressure. Moreover, the results have also been attested by using the measuring methods with fluids as the pressure-transfer media so as to make a comprehensive evaluation of the three methods.

1 Experimental method

In this paper a comparative study has been conducted on the three methods for measuring elastic wave velocities in rocks at high temperature and high pressure with solids as the pressure-transfer media.

1.1 Prism sample pulse transmission method (simplified as method 1 below)

This measuring method was developed and established on the YJ-3000 wedge-type cubic-anvil press by Prof. Xie Hongsen et al. in the Institute of Geochemistry, Chinese Academy of Sciences[10]. The experimental sample was prepared as a cylindrical prism measuring 12 mm in diameter and 32.5 mm in height. The prismatic specimen was wrapped by a stainless steel heater and assembled in the central hole of the pressure-transfer medium——cubic pyrophyllite (its side length is 32.5 mm). The transducer was put in the low-temperature zone on the far terminal of a tungsten carbide anvil from the specimen. During heating, cooling water was let to pass through the steel ring sleeving the anvil to keep the transducer at a constant temperature.

The pressure in the sample chamber was calibrated by using quartz-coesite phase transition and the high-pressure melting curve of copper, with the error involved in pressure measurement being less than 2%. The temperature in the sample chamber was calibrated by means of Pt-$Pt_{90}Rh_{10}$ thermocouple, with the error involved in temperature measurement being less than

10℃. The variation of sample's length was worked out in terms of piston shift of the press. The time of the ultrasonic waves through the sample was figured out by subtracting the time through the upper and lower pressure anvils from that through the anvil / sample / anvil. On this basis the elastic wave velocities could be calculated. The pressure and temperature that this method can realize are 0.1—6.0 GPa and up to 1500℃, respectively.

1.2 Prism sample pulse reflection-transmission method (simplified as method 2 below)

There is a major problem with method 1. Cracking happened when pressure was applied on the sample. With increasing pressure, the sample would be compacted under the action of pressure, and therefore the elastic wave velocity data acquired in the low-pressure range would be slightly lower[11]. In addition, a relatively large temperature gradient would exist since the prism sample was large in length. In order to reduce the temperature and pressure gradients occurring when the sample was heated at high pressure and enhance the precision of measurements, Liu et al. have made some improvements on sample assemblage and adopted the pulse reflection-transmission method to measure elastic wave velocities[12]. The height of the prism sample was shortened to be 6.0 mm and the buffer rods of the same diameter were assembled on both ends of the sample so as to make the sample lie in the optimal isothermal and isobaric region. The buffer rods were made of the adaptable materials chosen through experiments to acquire ideal acoustic impedance matching (fig. 1).

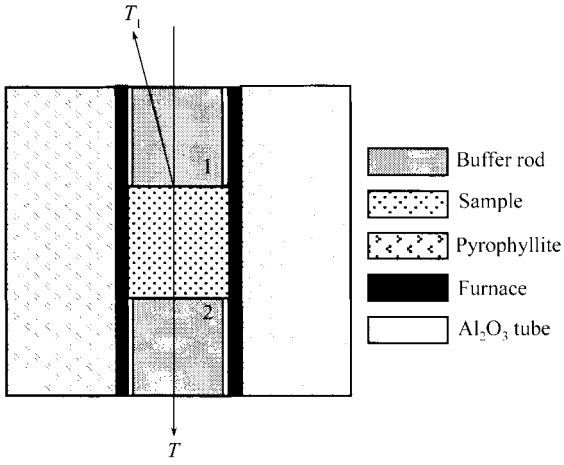

Fig. 1. Sketch map of the sample assemblage.

A CTS-200A type ultrasonic flaw detector manufactured by the Shantou Ultrasonic Electronic Instruments Factory was used as the ultrasonic emission source and the ultrasonic emission and acception could be effected in one channel. In order to improve time-measurement precision, a TDS784A digital oscilloscope made by the Tek Co. was used in parallel connection. In the experiment the routine transmission method was used to measure the total time, T, the transmission taken between the upper and lower transducers, and then the reflection method was used to measure the to-and-for time, T_1, between the upper transducer and the interface of the upper buffer rod and the sample, and the to-and-for time, T_2, between the lower transducer and the interface of the lower buffer rod and the sample. The traveling time of ultrasonic waves in the sample was calculated with the formula $t_S = T - (T_1 + T_2) / 2$. The length of the sample was corrected using the formula $L = L_0 \times (1 - p/3k_0)$, where L and L_0 represent the length of the sample at high pressure and that at normal pressure, respectively, p is the pressure, and k_0 is the bulk modulus. Finally the wave velocity was calculated : $V = L / t_S$. Ex-

perimental results showed that the relative error involved in wave velocity measurements is less than 1.5%. The temperature and pressure calibration by this method is the same as that by method 1. The realized pressure and temperature ranges have come up to the levels of method 1; the measurement precision, however, is higher than that obtained by the former.

1.3 Cubic sample pulse transmission method (simplified as method 3 below)

This measuring method was established on the multi-anvil apparatus at the University of Kiel by Kern[13—16]. The experimental sample was cubic in form, with the side length of 43 cm. The sample itself was used as the pressure-transfer medium. The six anvils moved forwards simultaneously and applied pressure on the rock sample in three directions so as to make the internal pressure more homogenized. Heating furnaces were mounted on the near-sample ends of the six anvils so as to make the sample heated in all directions. It was reported that when the temperature reached 700℃, the temperature gradient of the sample chamber was lower than 5℃[13].

The transducers were placed in the low temperature zones at the far-from-the sample ends of the anvils. In terms of the position shift of the piston one can work out the variation of experimental sample's length under high pressure conditions. The time of ultrasonic waves passing through the sample can be worked out by subtracting the time of ultrasonic waves passing through the upper and lower anvils from that of ultrasonic waves passing through the anvil-sample-anvil. From this one can figure out the elastic velocities. The pressure and temperature ranges that this method can realize are below 0.7 GPa and 750℃, respectively.

2 Experimental comparison of the three measuring methods at high pressure and normal temperature

2.1 Comparative experiment on the applied pressure ranges of the three measuring methods

Plagioclase amphibolite was sampled from the Fuping Group of the North China platform and the P-wave velocities of the samples were measured using the three methods mentioned above. The experimental results are shown in fig. 2. It can be seen from the figure that the maximum pressure acquired by method 3 is 0.7 GPa. That is because in the experiment by method 3 the sample was drastically cracked when the pressure was higher than 0.7 GPa, while accompanied with a big sound. Meanwhile, the wave velocity went down. When the pressure was applied again, the wave velocity varied irregularly, higher one movement, lower the next. Under such circumstances we could do nothing but stop our experiment. It can be seen clearly that the pressure range of application for method 3 is below 0.7 GPa. As can be seen from the figure, in the pressure range of 0.7 GPa the measurements obtained by methods 3 and 2 are well consistent with each other. As compared with the results of measurement obtained by the other two methods (2 and 3), the P-wave velocities measured by method 1 at lower pressure are relatively low. Till the pressure reached 2.5—3.0 GPa or higher, the results of measurement obtained by method 1 would be consistent with those by method 2. The causes leading to these results were introduced in the section

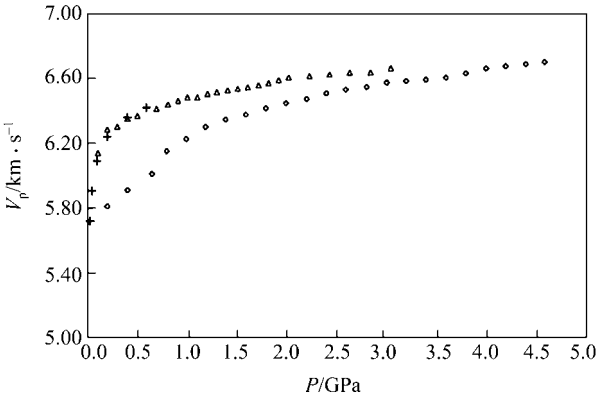

Fig. 2. Comparison of the elastic wave velocity measurements by the three methods on plagioclase amphibolite samples from the Fu- ping Group of the North China platform. ◇, Method 1; △, method 2; +, method 3.

on experimental methods. From the above discussion, it can be seen that the pressure range of application for method 1 is above 2.5—3.0 GPa.

In order to further understand why the applied pressure of method 3 is relatively low, we chose single crystalline calcite as the sample in consideration of its characteristic phase transition at normal temperature and high pressure, and used methods 2 and 3 to measure its P-wave velocities at high pressure. It is found from the experimental results that the two methods yielded quite different results (fig. 3). The results obtained by method 2 are the same as those obtained by other methods used by previous authors[17—20], and also consistent with the experimental results obtained by method 1. The variation curve of elastic wave velocity in calcite with pressure possesses the following characteristics: At the beginning of the experiment the P-wave velocity tends to increase with increasing pressure. When the pressure reaches 1.3—1.5 GPa or so, the P-wave velocity goes down rapidly to its minimum value because of phase transition in the calcite. Then, the P-wave velocity will increase with the continuous rise of pressure. However, for the results obtained by method 3, when the pressure is lower than 0.45 GPa, the results of measurement are consistent with those obtained by method 2; when the pressure is higher than 0.45 GPa, the wave velocity continues to drop because of the cracking of the sample, even when the pressure is higher than 1.3—1.5 GPa, still the wave velocity will not increase with increasing pressure. This result indicates that since no other kind of materials was used as pressure-transfer media for method 3, a considerably large pressure gradient would be produced in the interior of the sample once the sample was cracked. The measurement data obtained at > 0.45 GPa are of a mixed result: in the pressure range of 0.45—(1.3—1.5) GPa the drop of wave velocity is attributed mainly to the fragmentation of the sample; in the pressure

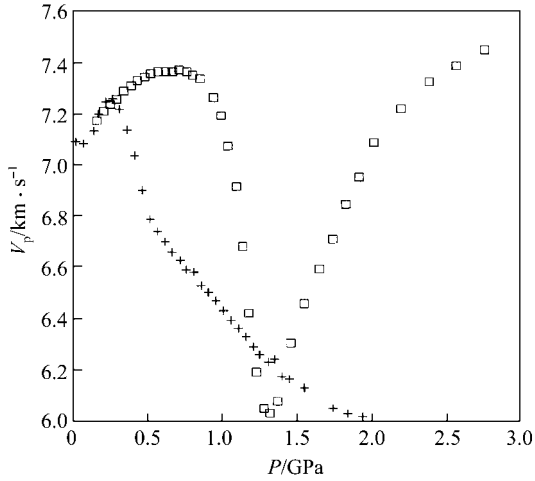

Fig. 3. The pressure-elastic wave velocity relationship diagram of single crystalline calcite established on the results obtained by the two measuring methods. +, Method 3; □, method 2.

range of 1.3—1.5 GPa the wave velocity continues decreasing, but the magnitude is much lower than that obtained by method 2, indicating that the results are a combination of wave velocity in the fraction in which phase transition took place and that in the fraction in which no phase transition occurred; in the pressure range of > 1.3—1.5 GPa the wave velocity will continue dropping, indicating that a part of the sample is still in the state of phase transition. The result is a combination of the wave velocity in the high-pressure calcite already experienced phase transition and that in the fraction still under phase transition. Through this comparative experiment, we think that method 3 would have a narrower pressure range of application when applied to brittle samples.

2.2 Comparative experiment on the measuring precision of methods 2 and 3

In order to attest the measuring accuracy of methods 2 and 3 in the pressure ranges of application, we chose the granulite samples collected from the Jining Group of the North China platform and measured their P-wave velocities in different directions using the two methods at high pressure and normal temperature. The results are shown in fig. 4. It can be seen from this figure that in the pressure range of 0.7 GPa the results of measurement obtained by the two methods in three directions of the samples are perfectly consistent.

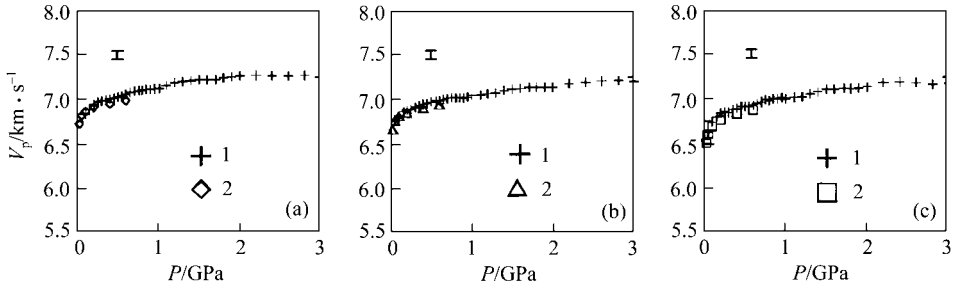

Fig. 4. Comparison of the P-wave velocities in the Jining Group granulite (JN11) samples from the North China platform. (a) Measurement in the direction parallel to the foliation and lineation. (b) Measurement in the direction parallel to the foliation and perpendicular to lineation. (c) Measurement in the direction perpendicular to the foliation. 1 stands for the data obtained by the reflection-transmission method, and 2 represents the data obtained by Kern[21]. The linear scale indicates the errors involved in measurements.

On the basis of the above experiments, using method 2 as a correction standard for the other measuring methods with solids as the pressure-transfer media, we have experimentally compared the data obtained by method 2 with those obtained by the methods with fluids as the pressure-transfer media to confirm the reliability of precision of this method. Shown in fig. 5 are the P-wave velocities measured at high pressure and norm temperature by method 2 at our laboratory on the hornblende eclogite samples collected from the Dabie Mountains. Also presented in the figure are the data of the same rock samples measured at high pressure and normal temperature using the pulse transmission method with fluid as the pressure-transfer medium at the Lab. of the Department of Geology and Geophysics, Wisconsin University of the United States[22]. From the comparison we can see that the results obtained by both methods in the three directions of the samples are consistent with each other.

The above experimental results indicate that of the three methods with solids as the pressure-transfer media, the prism sample pulse reflection-transmission method has a higher precision than the other two methods. In addition, its pressure range of application is also wider than that of the other two methods.

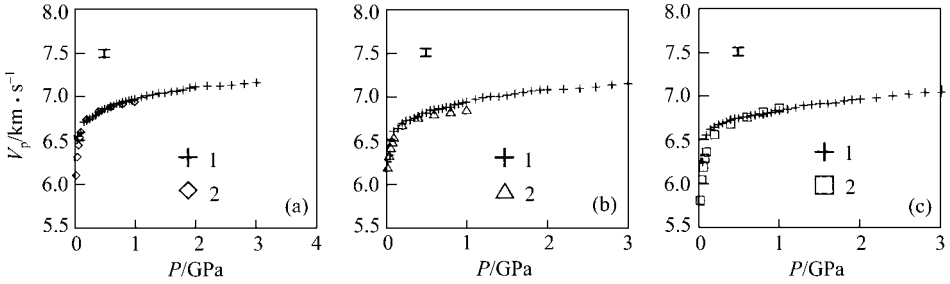

Fig. 5. Comparison of the high-pressure elastic wave velocities in hornblende-bearing eclogite sample (HL969) from Hualiangting of the Dabie Mountains measured by method 2 and Christensen[22]. (a) Measurement in the direction parallel to the foliation and lineation. (b) Measurement in the direction parallel to the foliation and perpendicular to lineation. (c) Measurement in the direction perpendicular to the foliation. 1 stands for the data obtained by using method 2, and 2 represents the data obtained by Christensen[22] using the pulse transmission method with fluid as the pressure-transfer medium.

3 A comprehensive assessment on the three methods for measuring elastic wave velocities in rocks at high pressure and high temperature

On the basis of the above comparative experiments on pressure ranges of application and measuring precision, the characteristics of the three measuring methods with solids as pressure-transfer media are summarized in table 1.

As can be seen from table 1, method 3 is particularly applicable to the crustal rocks whose elastic wave velocities can be measured at relatively low pressure and temperature. As this method can be used not only to measure the P- and S-wave velocities simultaneously, but also to measure the wave velocities of the sample in different directions simultaneously, a large wealth of elastic wave velocity data are available for rocks[13—16]. Nevertheless, due to relatively low applied pressure and temperature, the method could not be applied to crustal rocks in the whole crust. Especially, when the method is applied to those brittle rocks, the pressure range to be realized would be lower.

Method 2 is a kind of measuring method developed on the basis of method 1. Method 2 has the advantage over method 1 that the rock samples will not be cracked in the process of applying pressure on them. So the pressure range of application has been enlarged and the measuring precision has been enhanced. But method 2 requires length-small samples and therefore is not so applicable to coarse-grained rock samples. Moreover, as compared with method 2, method 1 is rather simple in sample assemblage and data processing. So method 1 is applicable to measuring elastic wave velocities in rock samples which require relatively high pressure and high temperature.

Table 1 Characteristics of three methods for measurement of elastic wave velocity in rock

Measuring method	Prism sample pulse transmission method (Method 1)	Prism sample pulse reflection-transmission method (Method 2)	Cubic sample pulse transmission method (Method 3)
Pressure range of application/GPa	(2.5—3.0)—6.0	0.1—0.6	< 0.7
Temperature range/℃	room temperature—1500	room temperature—1500	< 700
Method for measuring pressure	calibrated by copper melting curve and quartz-coesite phase transition method	calibrated by copper melting curve and quartz-coesite phase transition method	calculated from the piston pressure and the pressure area of the sample
Method for measuring temperature	thermocouple	thermocouple	thermocouple
Pressure-transfer medium	Prophyllite is used as the pressure-transfer medium, which is relatively stable in pressure-transfer performance.	Prophyllite is used as the pressure-transfer medium, which is relatively stable in pressure-transfer performance.	The sample itself is used as the pressure-transfer medium; variation is often observed in pressure-transfer performance for different samples.
Sample character	long-prismatic in shape, 32.5 in length, 12 mm in diameter	short-prismatic in shape, 6 mm in length, 12 mm in diameter	cubic in shape, with a side length of 43 mm
Sample variation in the process of applying pressure	At the beginning of applying pressure, the sample was cracked; with the rise of pressure the sample was compacted.	uneasy to crack during pressure application	no cracking within the pressure range of application
Whether or not the P- and S-wave velocities can be measured simultaneously	Impossible; the transducers must be replaced.	Impossible; the transducers must be replaced.	P- and S-wave velocities in the same rock sample can be measured simultaneously.
Whether or not the wave velocities of the same rock sample in different directions can be measured simultaneously	Impossible; different-direction samples must be prepared.	Impossible; different-direction samples must be prepared.	The wave velocities of the same sample in different directions can be measured simultaneously.

From the above analysis, we can see clearly that how to choose a measuring method in accordance with the characteristics of rock samples and the pressure and temperature ranges required by the experiment is of great importance in the research on elastic wave velocities in rocks at high temperature and high pressure. In addition, using the different measuring methods to make corrections for the results of measurement is beneficial to the enhancement of experimental research level.

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