Modern techniques in measuring elasticity of Earth materials at high pressure and high temperature using ultrasonic interferometry in conjunction with synchrotron X-radiation in multi-anvil apparatus

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Abstract

In the past 6 years, state-of-the-art techniques have been developed for the studies of elastic properties of polycrystalline and single-crystal materials using simultaneous ultrasonic and X-ray diffraction techniques at high pressures and temperatures in multi-anvil apparatus. The capability of these techniques is illustrated using recent data for mantle phases such as forsterite, wadsleyite, CaSiO3 perovskite, and (MgFe)O ferripericlase. Adaptation of the X-radiography technique enables direct measurements of sample length at high pressures and temperatures, enabling studies of unquenchable phases, liquids, and multi-phase aggregates. The use of dual-mode transducers produces P and S wave data at the same pressure and temperature conditions in a single acoustic experiment, thereby eliminating the uncertainties caused by the need for interpolating travel-time data to calculate the elastic wave velocities, and bulk and shear moduli at high pressure and temperature. Implementation of the transfer function method for travel-time measurements opens new opportunities for studying time-dependent processes using ultrasonic measurements, such as phase transformations and plastic deformation.

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1. Introduction

Elastic properties of Earth materials at high pressure and high temperature are crucial for understanding the composition, structure and dynamics of the Earth. Seismological investigations provide the primary source of information about the properties and processes of the Earth’s interior from density and velocity profiles as a function of depth. Seismic tomography on a global scale reveals lateral variations of compressional (P) and shear (S) wave velocities and densities at various depths of the Earth’s interior, reflecting differences of chemical composition, mineralogy, and/or temperature. Successful interpretation of these seismic models in terms of these variables requires experimental and theoretical information on the elasticity of deep Earth materials under elevated conditions of pressure and temperature.

Ultrasonic techniques (e.g., McSkimin, 1950) have been used extensively in the last four decades to...
obtain the elastic properties of Earth materials at high pressure and/or high temperature, from early experiments at \( P < 1 \text{ GPa} \) \( (e.g., \text{Gieske and Barsch, 1966; Schreiber and Anderson, 1966; Kumazawa and Anderson, 1969}) \) to subsequent work at pressures up to \( 3 \text{ GPa} \) \( (e.g., \text{Jackson and Niesler, 1982; Rigden et al., 1988, 1992; Gwanmesia et al., 1990; Webb, 1989}) \), and to recent experiments at \( P > 10 \text{ GPa} \) \( (e.g., \text{Li et al., 1998a; Knoche et al., 1998}) \). Ultrasonic interferometry at simultaneous high pressures and temperatures has been made at gigahertz frequencies in diamond-anvil cells \( (e.g., \text{Bassett et al., 2000; Jacobsen et al., 2002; Spetzler et al., 1993, 1996; Reichmann et al., 1998}) \). Although the GHz technique has the advantage of requiring only very small specimens, the maximum pressure and temperature range achieved to date are still limited. At the same time, there has been continuous effort to develop a “transducer and sample friendly” design using large-volume, high-pressure apparatus in a number of Japanese laboratories \( (e.g., \text{Yoneda, 1990; Yoneda and Morioka, 1992; Fujisawa and Ito, 1985; Fujisawa, 1998}) \). However, these experiments shared a common limitation, i.e., the piezoelectric transducers were all subjected to high pressure/stress, causing these measurements to have low signal to noise ratios, and lead eventually to the failure of the experiment due to the failure of the piezoelectric transducer at high pressure. Our innovations in sample cell design and the “stress-free” transducer environment have resulted in great improvements in the adaptation of ultrasonic interferometry techniques for use in multi-anvil high-pressure apparatus \( (\text{Li et al., 1998a}) \). The “sample friendly” cell assembly enables us to perform reproducible measurements on a single sample. In the few years since the successful implementation of ultrasonic interferometry in our laboratory, acoustic velocities for many mantle phases have been measured to mantle pressures using both polycrystalline and single-crystal samples \( (\text{Chen et al., 1996; Flesch et al., 1998; Li et al., 1996; Liebermann and Li, 1998; Gwanmesia et al., 1998; Chen et al., 1999; Liu et al., 2000; Li and Liebermann, 2000}) \).

Earlier ultrasonic experiments at simultaneous high-pressure and high-temperature conditions of the Earth’s mantle demonstrated the feasibility of performing measurements of both P and S wave velocities to pressures above \( 10 \text{ GPa} \) and simultaneous temperatures above \( 1300 \text{ K} \) using large-volume, high-pressure apparatus \( (e.g., \text{Knoche et al., 1998; Li et al., 1998a}) \). However, two major difficulties in these experiments remained to be overcome: (1) the conventional method for determining pressure \( (i.e., \text{a calibration curve fitted through a few points calibrated using known phase transformations}) \) is not precise enough for the determination of the pressure and temperature dependencies of the elastic moduli and cannot readily be applied at high temperatures; (2) the estimation of the sample length \( (\text{which required to convert travel times to velocities at high pressure and high temperature}) \) using previous equation-of-state \( (\text{EOS}) \) data is not reliable, especially when the sample environment deviates from hydrostatic conditions and plastic deformation occurs at high temperatures.

In the last 5 years, we have successfully overcome these challenges by adapting the multi-anvil ultrasonic techniques for use in a DIA-type, cubic-anvil apparatus \( (\text{SAM 85}) \) installed on the superconducting wiggler beamline \( (\text{X-17B1, now X-17B2}) \) at the National Synchrotron Light Source \( (\text{NSLS}) \) of the Brookhaven National Laboratory \( (\text{BNL}) \) \( (e.g., \text{Liebermann and Li, 1998}) \). These developments have enabled P and S wave velocities and X-ray measurements to be performed simultaneously to pressures of \( 8 \text{ GPa} \) and temperatures of \( 1600 \text{ K} \) on both single-crystal \( (\text{Chen et al., 1998; Decremps et al., 2001}) \) and polycrystalline specimens \( (\text{Li et al., 1998b, 2001; Sinelnikov et al., 1998; Kung et al., 2002}) \). Recent implementation of X-radiography to the existing setup allows us to monitor sample lengths directly at elevated pressure and temperature \( (\text{Li and Weidner, 1999; Kung et al., 2002; Li et al., 2004}) \). In this paper, we will describe our state-of-the-art techniques for experimental studies of the elasticity of deep Earth materials at high pressure and high temperature using combined ultrasonic interferometry, X-ray diffraction, and X-radiography in large-volume apparatus.

2. P–V–T measurements using simultaneous ultrasonics and X-ray diffraction

2.1. Ultrasonic interferometry in SAM 85

The ultrasonic techniques described above have been adapted for use in SAM 85 \( (\text{Weidner et al., 1999; Kung et al., 2002; Li et al., 2004}) \).
In Fig. 1a, we show the key elements of the experimental setup on X-17B1 (currently reconstructed as X-17B2) at NSLS/BNL in schematic form: (a) incident X-ray beam with slits and monochromator (optional); (b) the multi-anvil press (SAM 85); (c) a solid-state detector (SSD) or a charge-coupled device (CCD) detecting system; (d) a YAG crystal and a CCD camera to record the X-radiographic images; (e) the ultrasonic system. The ultrasonic piezoelectric transducer-tungsten carbide anvil arrangement and the high-temperature cell assembly used in SAM 85 are shown in Fig. 1a and b; in this configuration, the LiNbO₃ transducer is mounted onto the backside of the WC anvil (Toshiba Grade F) and connected to the ultrasonic interferometer by coaxial cables (Fig. 1a). The WC anvil serves as an acoustic buffer rod to transmit the high-frequency signals (20–70 MHz) into the cell assembly. The acoustic waves pass through the bottom anvil and propagate into another buffer rod (fused silica glass, alumina...
or other material with suitable acoustic impedance) and then into the sample (Fig. 1b). The observed acoustic signals are a series of reflections from the interfaces perpendicular to the wave propagation path, including those from the anvil/buffer rod, buffer rod/sample, and sample/backing material (e.g., NaCl) interfaces.

The phase-comparison method of ultrasonic interferometry, using the Australian Scientific Instruments Ultrasonic Interferometer (ANUTECH), has been employed in our laboratory for a number of years (see details in Niesler and Jackson (1989) and Rigden et al. (1992)). The output from a continuous wave source is gated to produce a pair of phase-coherent, high-frequency pulses which serve as input signals to excite the transducer. The elastic waves generated by each pulse are reflected and transmitted at the buffer rod/sample interface, and the transmitted portion reverberates inside the sample, resulting in a series of ‘sample’ echoes following the buffer rod echo. If the applied pulses are separated by the apparent two-way travel time through the sample, the first buffer echo (B) from the second applied source pulse will superimpose with the first sample echo (S) from the first source pulse. As the carrier frequency is varied, alternate constructive and destructive interference between the superimposed buffer rod and sample signals will occur, resulting in a series of maxima and minima in the amplitude spectrum and sample signals will occur, resulting in a series of 'sample' echoes following the buffer rod echo. If the applied pulses are separated by the apparent two-way travel time through the sample, the first buffer echo (B) from the second applied source pulse will superimpose with the first sample echo (S) from the first source pulse. As the carrier frequency is varied, alternate constructive and destructive interference between the superimposed buffer rod and sample signals will occur, resulting in a series of maxima and minima in the amplitude spectrum.

The pressure is normally increased to a designated peak pressure at room temperature and then the temperature is increased, with a consequent decrease of pressure due to relaxation and flow of the cell materials (see Fig. 2a in Li et al. (2003)). We also experimented with P-T paths that involve heating the cell assembly at moderate temperatures (≈673 K) during the pressurization to relax the stress on the buffer rod and sample, as shown by the dashed lines in Fig. 2; this type of P-T path is very effective in improving signal-to-noise ratio when glass buffer rods are used (see discussion in Li et al. (2001)). After reaching the peak P and T conditions, the experiments were continued via a series of cooling/heating cycles at various pressures; after each cooling stage, the pressure is released slowly and then another heating stage is commenced. In the forsterite experiments, a maximum
temperature of 1273 K was reached in both the P and S wave experiments. However, the sample recovered from the first experiment (P wave) had been shortened about 1.6%, presumably when the temperature was at the maximum of 1273 K. In this case, we used the recovered sample length to deduce the velocities on heating/cooling up to 1073 K during decompression, whereas the original sample length was used to reduce the velocity data prior to reaching 1273 K.

In the experiments on wadsleyite, the experimental range of $P$ and $T$ was limited by the thermodynamic stability of this high-pressure polymorph (which is metastable below pressures of $\sim$13 GPa). Consequently, the peak temperatures were limited to 873 K to prevent back-transformation to the olivine phase. The wadsleyite sample recovered from these experiments was examined in detail: no changes in dimension were observed (which would have reflected permanent plastic deformation) nor were any macroscopic cracks evident; X-ray spectra indicated that the specimen was still pure wadsleyite phase.

In Fig. 3, we plot the unit cell volumes at high pressure and temperature for forsterite up to 6.7 GPa and 1273 K from the P wave experiment. These data can be analyzed independently to obtain the parameters of the $P-V-T$ equation-of-state; they also provide a measurement of the length change of the polycrystalline sample under the assumption of isotropic compression and expansion (in which case $l/l_0 = (V/V_0)^{1/3}$, where $l$ is the sample length and $V$ the unit cell volume; subscript zero represents value at ambient conditions). If the bulk modulus and its pressure derivative are constrained to be $K_0 = 125$ GPa and $K_0'$ = 4.5 from our acoustic data obtained in current experiments, a fit to the data in Fig. 3 using a third-order Eulerian EOS (Birch-Murnaghan) yields the temperature derivative of bulk modulus, $(dK_T/dT)_P = -0.022(2)$ GPa/K, linear thermal expansion ($a = a + bT$) coefficients, $a = 2.4 \times 10^{-5}$ K$^{-1}$ and $b = 3.0 \times 10^{-8}$, and the unit cell volume at ambient conditions $V_0 = 289.80$ Å$^3$. These values are compatible with previous data at high
Fig. 3. Unit cell volumes of forsterite Mg$_2$SiO$_4$ as a function of pressure and temperature obtained in the P wave experiment: (■) measured values; (○) values resulting from fitting the measured data to a modified third-order Eulerian finite strain equation of state.

Travel-time data for the P and S wave velocities in forsterite are plotted in Fig. 4a and b with maximum pressures of 6.7 and 9.2 GPa, respectively. These data are converted to velocities and plotted in Fig. 5a and b using the length determinations described above. With these P–V–P–V–T data, we have obtained direct measurements of the adiabatic bulk ($K_S$) and shear (G) moduli and their first pressure and temperature derivatives for forsterite phase of Mg$_2$SiO$_4$ following the same procedure described for wadsleyite (Li et al., 2001). The inferred parameters, $K_S = 125(2)$ GPa, $G = 81(1)$ GPa, $(\partial K_S/\partial P)_T = 4.5(2)$, $(\partial G/\partial P)_T = 1.4(1)$, $(\partial K_S/\partial T)_P = -0.014(1)$ GPa/K and $(\partial G/\partial T)_P = -0.017(1)$ GPa/K, are compatible with previous data obtained at high pressures and/or high temperatures (e.g., Isaak et al., 1989; Duffy et al., 1995; Meng et al., 1993; Li et al., 1996) (Table 1). The combined velocity and unit cell volume data also provide the opportunity to evaluate the reliability of the Decker NaCl pressure scale (e.g., Liu, 2000), and determine

Table 1

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<td>$K_S$ (GPa)</td>
<td>125(2)</td>
<td>128.8(5)</td>
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<td>129</td>
<td>129</td>
<td>127.4</td>
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<td>$(\partial K_S/\partial P)_T$</td>
<td>4.5(2)</td>
<td>–</td>
<td>4.46(2)</td>
<td>4.1</td>
<td>4.2</td>
<td>(4.8)</td>
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<tr>
<td>$(\partial K_S/\partial T)_P$</td>
<td>–0.014(1)</td>
<td>–0.0157(4)</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–0.021(2)</td>
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<tr>
<td>$G$ (GPa)</td>
<td>81(1)</td>
<td>81.8(2)</td>
<td>80</td>
<td>82</td>
<td>81</td>
<td>–</td>
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<td>–</td>
<td>1.32(3)</td>
<td>1.6</td>
<td>1.4</td>
<td>–</td>
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<tr>
<td>$(\partial G/\partial T)_P$</td>
<td>–0.017(1)</td>
<td>–0.0135(2)</td>
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pressure derivatives without using secondary pressure standard (e.g., Kung et al., 2002).

2.4. Dual-mode transducers

In the experiments on forsterite and wadsleyite, the P and S wave travel times were measured separately using pure-mode transducers; one difficulty in this approach is that it is not simple, in practice, to ensure that the P and S travel times are measured at the same cell pressures in separate experimental runs. Recently, we have used dual-mode LiNbO$_3$ transducers (10$^\circ$Y-cut) in our experiments; these transducers generate both longitudinal and transverse acoustic signals with unspecified polarization direction of the transverse signal. The size of the tungsten carbide cube...
Fig. 5. (a) P wave velocities in Mg$_2$SiO$_4$ forsterite as a function of pressure to 6.7 GPa and temperature to 1273 K. The dashed lines represent data for different isotherms. (b) S wave velocity in Mg$_2$SiO$_4$ forsterite as a function of pressure to 9 GPa and temperature to 1273 K. The dashed lines represent data for different isotherms.

A buffer rod (~35 mm) provides a long delay line, so that the P and S wave echo patterns can be clearly separated in time (Fig. 6a). Travel times of a polycrystalline alumina sample (2.460 mm in length) were measured under ambient conditions using a dual-mode LiNbO$_3$ transducer, showing groups of both P and S wave echoes from the buffer rod (B) and the samples (S1 and S2). (b) Details of the buffer rod and sample echoes in the first P wave group.

Steel buffer rod. The travel-time determinations from the pure-mode P and S wave transducers and those from dual-mode transducer are consistent within 0.1%, which is within the system uncertainty. Other examples of the use of the dual-mode transducers in our laboratory may be found in Kung et al. (2002), Darling et al. (2004), and Sinelnikov et al. (2004).

3. X-radiography in DIA apparatus

3.1. X-ray imaging

X-radiography has been used in the study of viscosity of the molten materials at high pressure and high temperature using a falling sphere method (e.g., Mattiscek and Sobczak, 1994). We have successfully incorporated this technique for use in our simultaneous ultrasonics and X-ray diffraction experiments (e.g., Kung et al., 2002). Briefly, the imaging system consists of a YAG crystal, a mirror, and a CCD camera.
Fig. 7. Example of the X-radiographic images recorded in the high P-T experiments as seen between the gap in the WC anvils (on right). Image file read as gray scale (on left); the two peaks in the darkness scale correspond to the gold foils at the top and bottom of the sample.

On these X-radiographic images, the brightness contrast from region to region results from the difference in the X-ray absorption coefficients of the various materials in the high-pressure cell assembly. If the absorption coefficient for the sample is sufficiently different from those of the adjacent materials (buffer rod or NaCl), the sample region can be clearly distinguished from the rest of the cell assembly. For cases in which the sample and the other parts of the cell assembly have similar absorption coefficients, metal foils with high X-ray absorption (e.g., gold) are inserted at both the top and bottom surfaces of the sample. For our ultrasonic measurements, such gold foils (0.002 mm thick) are routinely inserted at the sample/buffer rod interface to enhance the mechanical coupling between the buffer rod and sample; these also serve well in delineating the sample boundaries, as seen by the dark horizontal bands in Fig. 7.

For the calibration of sample length with respect to pixel number, one can obtain the calibration factor by comparing the pixel numbers from the last image (after the experiment has reached zero pressure) with the length of the recovered sample measured using a precise micrometer. This last image can then be used as the starting image to retrieve the relative change in pixels between neighboring P-T conditions by cross-correlation of the intensity profiles at sample boundaries. Typically, a change of 0.25 pixels or less can be resolved over a sample dimension of about 1000 pixels. If the sample length is directly measured from an image, it has a total uncertainty about 2–4 pixels, which gives 0.2–0.4% in precision. As seen in Fig. 8, the lengths of a polycrystalline sample of (Mg$_{0.83}$Fe$_{0.17}$)O at high pressures obtained from the two different methods described above (one from direct X-ray imaging measurements and the other derived from X-ray diffraction determinations of sample volumes) exhibit remarkable agreement (see also Fig. 4 of Kung et al., 2002).
to conduct precise elasticity measurements on a variety of materials and processes, including materials undergoing mode softening/phase transformations (e.g., Decremps et al., 2000, 2001) or plastic deformation (e.g., Darling et al., 2004), as well as melts or multi-phase aggregates (e.g., Mistler et al., 2002).


The elastic properties of CaSiO$_3$ perovskite under mantle conditions are very important in understanding its possible contribution to the 520 km seismic discontinuity as well as its visibility in the velocity structure of the lower mantle (e.g., Wang et al., 1996; Hama and Suito, 1998; Karki and Crain, 1998; Mao et al., 1989; Tarrida and Richet, 1989). Since this high-pressure phase is unquenchable (i.e., cannot be recovered in its high-pressure form at ambient conditions), measurements of its properties have to be conducted after in situ synthesizing in its stability field (see phase diagram in Gasparik et al. (1994)). Another complication is that the low-pressure wollastonite phase of CaSiO$_3$ undergoes a process of conversion to an amorphous state with increasing pressure, making the estimation of the sample length for the CaSiO$_3$ perovskite phase impossible without direct measurement.

We have successfully conducted P and S wave velocity measurements on the cubic-structure phase of CaSiO$_3$ perovskite within its stability field at 13.5 GPa and 1123 K (see Li et al., 2004). As demonstrated in Li et al. (2004), the starting material, a semi-sintered low-pressure sample of CaSiO$_3$ wollastonite (triclinic, $\rho = 2.97 \text{g/cm}^3$), become progressively amorphous with increasing pressure; an image of the sample at 9.6 GPa and 300 K and the corresponding X-ray diffraction pattern are shown in Fig. 9a and c. The formation and growth of the perovskite phase during heating was monitored by real-time X-ray diffraction; after 1.5 h at 13.5 GPa and 1123 K, the starting material was completely transformed into the cubic-structured perovskite as shown in the X-ray diffraction pattern and the sample image (Fig. 9b and d). The substantial length reduction is obvious in the images before and after the phase transformation, as a consequence of the high density of the new phase ($\rho = 4.26 \text{g/cm}^3$).

From the direct measurements of length and travel times as described above, P and S wave velocities for CaSiO$_3$ perovskite were obtained for pressures between 8 and 11.5 GPa at room temperature (Fig. 10). When the pressure was decreased below 8 GPa, the WC anvil on which the transducer was mounted broke and no further acoustic data could be recorded. Comparing with our experimental data for the P and S wave velocities with those calculated by Karki and Crain (1998) from first-principles methods in Fig. 10, we see that the theoretical values are higher about 10%, presumably because the theoretical calculations are for 0 K and ignore the zero-point energy. Measurements at a wider range of pressure and temperature are necessary in order to provide more robust determinations of the pressure and temperature derivatives.

4. Sound velocity measurement using transfer function method

Ultrasonic interferometry has been used widely in the study of elastic properties of solid and liquid materials since the 1950s. Over the years, different methods have been developed for the measurement of the time of wave propagation, such as the pulse echo overlap (PEO), phase comparison (PC), and pulse superposition (PSIP) methods (see reviews in Truell et al. (1969)). In these methods, a radio-frequency (rf) pulse is transmitted and received by a piezoelectric...
Fig. 9. X-radiographic images for CaSiO$_3$ at $\sim$10 GPa and 300 K (a) and 13.5 GPa and 1123 K (b). The diffraction pattern taken at $\sim$10 GPa and 300 K (c) indicates remnant diffraction peaks of the wollastonite starting material and an amorphous phase produced on compression at room temperature. The X-ray diffraction pattern taken at 13.5 GPa and 1123 K (d) indicates that most of the sample was transformed to the perovskite phase; $hkl$ labels are the diffraction peaks for the cubic perovskite phase of CaSiO$_3$.

Recently, the development of fast analogue/digital acquisition cards has made it possible to use transfer function methods based on digital signal processing (such as cross-correlation function (CCF), Hilbert transform (HT), and continuous wavelet transform (CWT) (Moreno et al., 1999)) rather than relying on the classical pulse superposition or phase comparison methods. In this section, we describe sound velocity measurements using the transfer function method.

The transfer function depicts the response of the system as a function of frequency (e.g., piezoelectric transducers) (Huemer et al., 1999). After a wavelet $x(t)$ is sent to the buffer rod and sample, the received signal $y(t)$ is the convolution of the input signal $x(t)$ and the system response $h(t)$ along the wave propagation path, i.e., $y(t) = x(t) * h(t)$. Fast Fourier transform (FFT) of the convoluted signal yields $Y(f) = X(f)H(f)$. The transfer function of the piezoelectric transducer, buffer rod, and sample ensemble can thus be obtained, $H(f) = Y(f)/X(f)$. Subsequently, convoluting the transfer function with another input signal yields the response of the sample system to the input pulse for frequencies inside the bandwidth.

A typical received waveform for an alumina polycrystal on a tungsten carbide buffer rod is shown in Fig. 11. To simulate the PEO measurement of phase velocity, a 45 MHz sine wave toneburst (8 cycles) was convoluted with the transfer function; thus, the system response to such 45 MHz input signal is...
Fig. 11. Received ultrasonic signals for P and S waves using the transfer function method for a polycrystalline Al₂O₃ sample mounted on a tungsten carbide buffer rod. Reproduced (Fig. 6a). Fig. 6b shows the expanded view of the buffer rod echo and two sample echoes. The time delay is obtained by measuring the time shift of the second copy of the echo pattern relative to the original one until the buffer rod and sample echoes overlap.

As demonstrated in Li et al. (2002), the phase comparison measurements can also be reproduced using the recorded transfer function by sweeping through the frequency range and recording the interference amplitude between the buffer rod and sample (e.g., Fig. 12a). Moreover, using the amplitudes of the buffer rod (A_B), sample (A_S), and their interference (A_I), the effect of the transducer response envelope can be removed from the recorded interference spectrum using the relationship (e.g., Spetzler et al., 1993)

\[ \cos \theta = \frac{A_I^2 - A_B^2 - A_S^2}{2A_B A_S} \]

with \( \theta = \theta_s + \theta_b \)

where \( \theta \) is the phase difference between the buffer rod and sample echoes caused by the additional travel inside the sample (\( \theta_s \)) and the effect of the bonding material (\( \theta_b \)). In Fig. 12b, we show the results after the removal of the transducer response from the interference pattern in Fig. 12a. Fitting the spectrum in Fig. 12b with a cosine function yields precise determination of travel time and the phase shift of the bond. The travel times obtained from both methods are compared in Fig. 12c. Using the transfer function method, such simulations can be conducted multiple times for optimized time measurements by adjusting the data collection window and step size of the sweeping frequency.

We also compared travel times obtained using the transfer function method with those obtained from the
phase comparison method using the ANUTECH interferometer. In Fig. 13, we plot the travel times for a polycrystalline (Mg$_{0.83}$Fe$_{0.17}$)O at high pressures measured using two different ultrasonic techniques (from Kung et al., 2002). The agreement is within 0.25%, comparable to the uncertainty of the individual techniques. However, compared with real-time measurements, the transfer function method reduces data collection times from minutes to seconds, which is a very valuable attribute in conducting experiments at elevated temperatures and/or pressures. The transfer function method also has the advantage of recording the system response to all frequencies inside the pass-band at the same time, in contrast to sweeping through frequencies in the pulse echo overlap and phase comparison methods; this is especially important when travel times are being used to study time-dependent processes, such as plastic deformation of materials (e.g., Li and Weidner, 1999) or phase transformations (Decremps et al., 2000, 2001).

5. Conclusion

We have progressively developed state-of-the-art techniques for the studies of elastic properties of polycrystalline and single-crystal materials using simultaneous ultrasonic and X-ray diffraction techniques at high pressures and temperatures in multi-anvil apparatus, in conjunction with synchrotron X-radiation. The capability of these techniques has been illustrated using our data for mantle minerals such as the forsterite and wadsleyite phases of Mg$_2$SiO$_4$. Incorporation of X-radiography techniques enables us to directly measure sample length with a precision better than 0.5% and the power to resolve relative length changes at $2.5 \times 10^{-4}$ using current setup, regardless of the sample state at high pressure and high temperature.

The implementation of the transfer function method for sound wave velocity measurements opens new opportunities for studying time-dependent processes using ultrasonic measurements, such as phase transformations and plastic deformation. Real-time signals can be reproduced off-line for travel-time measurement using the pulse echo overlap and phase comparison methods over a wide frequency range. The use of dual-mode transducers produces P and S wave data at the same pressure and temperature conditions in a single acoustic experiment, eliminating the uncertainties caused by the need for interpolating travel-time data to calculate the elastic wave velocities and bulk and shear moduli at high pressure and temperature (as was necessary in our earlier experiments without X-rays). Successful application of these techniques to the study of the behavior and physical properties of Earth materials, including unquenchable mantle phases, liquids, and multi-phase aggregates, will greatly improve our interpretation of seismic observations and our understanding about the
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