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Thank you for your assistance.
Sound velocities of hydrous ringwoodite to 16 GPa and 673 K

Zhu Mao a,⁎, Jung-Fu Lin a, Steven D. Jacobsen b, Thomas S. Duffy c, Yun-Yuan Chang b, Joseph R. Smyth d, Daniel J. Frost e, Erik Haun f, Vitali B. Prakapenka g

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► We have studied the elasticity of Fe-ringwoodite with 1 wt.% H2O at high P-T. ► Water lowers the elasticity but does not affect the pressure derivatives at 300 K. ► The reduction caused by hydration is enhanced when T is elevated at high P. ► T-variations with ~0.1 wt.% H2O may explain the seismic anomaly at 660-km depth.
Supplementary materials.
Sound velocities of hydrous ringwoodite to 16 GPa and 673 K

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A R T I C L E  I N F O

Article history:
Received 4 November 2011
Received in revised form 10 February 2012
Accepted 2 March 2012
Available online xxx

Editor: L. Stixrude

Keywords:
hydrous ringwoodite
single-crystal elasticity
high pressure-temperature
Brillouin scattering

A B S T R A C T

To understand the effect of hydration on the sound velocities of major mantle minerals and to constrain the mantle’s H2O budget, we have measured the single-crystal elastic moduli of hydrous ringwoodite, (Mg1.633Fe2+0.231Fe3+0.062)Si1.016H0.796O4 with 1.1 wt.% H2O using Brillouin scattering combined with X-ray diffraction in an externally-heated diamond anvil cell up to 16 GPa and 673 K. Up to 12 GPa at 300 K, the presence of 1.1 wt.% H2O lowers the elastic moduli of ringwoodite by 5–9%, but does not affect the pressure derivatives of the elastic moduli compared to anhydrous ringwoodite. The reduction caused by hydration is significantly enhanced when temperatures are elevated at high pressures. At 12 GPa, increasing temperature by ΔT=100 K leads to a 1.3–2.4% reduction in the elastic moduli (C11, C12, and C44). Comparing our results with seismic observations, we have evaluated the potential H2O content in the lower part of the transition zone. Our results indicate that the observed seismic velocity anomalies and related depth depression of the 660-km discontinuity could be attributed to thermal variations together with the presence of ~0.1 wt.% H2O.

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The H₂O content in the Earth's transition zone can be estimated by comparing the sound velocities of candidate hydrous minerals such as wadsleyite and ringwoodite with seismic velocities. Previous experiments at 300 K and first principle studies (at 0 K) have determined the high-pressure elasticity of hydrated transition zone minerals (Avouac et al., 2009; Lin et al., 2009; Mao et al., 2008a,b). Using the reported error is the standard deviation from the mean. Using the eight-position centering method (King and Finger, 1979), which has allowed us to improve the precision of the lattice parameters measured previously (Smyth, 1987), yields a lattice parameter, a = 8.1060 (±0.0007) Å, with a cell volume of 532.62 (±0.14) Å³. The measured lattice parameter between this study and Smyth et al. (2003) is within the experimental errors. The Fe³⁺/ΣFe ratio, measured by Mössbauer spectroscopy, is about 0.10 (Smyth et al., 2003). The water content of the ringwoodite sample was determined on the Cameca nanoSIMS 50 L at the Department of Terrestrial Magnetism, Carnegie Institution of Washington. Using a 1 nA Cs⁺ primary beam, a 10 µm pre-sputter raster was carried out for 60 s. The analysis raster was 7 µm and a beam-blanking mask was used to collect ions from an area of approximately 2.6 × 2.6 mm² in the sample at each probe point. The water content was calibrated against three NIST standard silicate glasses, 519-4-4 with 0.17 wt.% H₂O, and D30-1 with 1.63 wt.% H₂O. High P–T Brillouin scattering and X-ray diffraction experiments were performed at beamline 13BM-D of the GSECARS sector, Advanced Photon Source, Argonne National Laboratory. A single crystal of 30–35 μm thickness was double-side polished and loaded into the externally-heated diamond anvil cell (EHDAC) along with a Ne pressure medium. A Pt foil was placed next to the crystal for in situ pressure measurements (Fei et al., 2007); Ne was also used as a secondary pressure calibrant (Fei et al., 2007). A K-type thermocouple, placed onto the diamond 500 μm away from one of the diamond culets, was used for the temperature measurements. Brillouin spectra of hydrous ringwoodite were collected in a forward scattering geometry with 50° scattering angle at pressures up to 16 GPa and three temperatures (300 K, 550 K and 673 K) (Fig. 1). Pressure stability of the EHDAC was monitored by collecting X-ray diffraction patterns after each pressure or temperature increment such that the measured pressure from the pressure calibrant did not change within experimental uncertainties. Pressures were determined from the X-ray diffraction patterns of the Pt calibrant collected before and after each Brillouin measurement. We also collected the diffraction pattern of ringwoodite before and after each heating cycle to determine the unit cell volume and density of the sample. There was no change in the unit cell volume of ringwoodite before and after the heating within experimental uncertainties, indicating that the water content did change. In addition, Raman spectra were collected from the ringwoodite sample quenched from 673 K and 16 GPa. The vibrational frequencies and spectral intensity in the OH vibrational bands for the quenched sample were similar to those of the starting sample (SFig. 1). We thus conclude that minimum water was lost after the high P–T experiments. This is consistent with previous observations of dehydration of ringwoodite beginning above 720 K (Inoue et al., 2004; Ye et al., 2009). Single-crystal elastic constants, C, of hydrous ringwoodite at each P–T condition were obtained by fitting the measured velocity.
curves using Christoffel’s equation via non-linear least squares methods (Table 1, Figs. 2 and 3) (Every, 1980):

\[ C_{ijkl} \partial_\rho_{ij} \phi = 0, \]  

(1)

where \( C_{ijkl} \) are the elastic constants in full suffix notation, \( \phi \) represents the direction cosines of the phonon propagation direction, \( \rho \) is the measured acoustic velocity, and \( \rho \) is the averaged density determined by X-ray diffraction before and after the Brillouin scattering measurements at each given P-T condition. The direction cosines of the phonon propagation direction, \( \phi \), are described by three Eulerian angles (\( \theta, \chi, \varphi \)). The Eulerian angles relate the laboratory reference to the crystallographic reference frame (Shimizu, 1995), and can be determined from the single-crystal X-ray data.

Using the single-crystal elastic constants, we calculated the Voigt–Reuss–Hill averages for the adiabatic bulk (\( K_s = 175 \pm 1 \) GPa) and shear (\( G = 106 \pm 1 \) GPa) moduli (Fig. 4 and Table 1). These results are similar to results from GHz-ultrasonic measurements on similar material, with \( K_s = 177 \pm 2 \) GPa and \( G = 103 \pm 2 \) GPa (Jacobsen and Smyth, 2006; Jacobsen et al., 2004). Although the samples from the current Brillouin study (run S20104) and the GHz-ultrasonic study (run S29001 from Smyth et al., 2003) are both similar in bulk composition and water content, ringwoodite in the current study co-existed with wadsleyite and not stishovite, whereas ringwoodite in the current study (run SZ9901 from Smyth et al., 2003) are similar in bulk composition and water content, ringwoodite in the current study co-existed with wadsleyite and not stishovite, whereas ringwoodite in the current Brillouin study (run SZ0104) and the GHz-ultrasonic study were made at slightly higher pressures (20 GPa) and co-existed with stishovite and not wadsleyite. The silica activity is known to affect the hydration mechanisms in olivine (Mosenfelder et al., 2006), and it is also possible that there are differences in the defect structure of ringwoodites from these two runs.

At 300 K, third-order finite Eulerian strain equations were used to obtain the pressure derivatives of the elastic moduli, yielding: \( C_{11}' = 6.9 \pm 0.1 \), \( C_{12}' = 2.4 \pm 0.1 \), \( C_{44}' = 1.0 \pm 0.1 \), \( K_{SO}' = 4.0 \pm 0.1 \), and \( G_0' = 1.6 \pm 0.1 \). However, the longitudinal modulus, \( C_{11}' \), and shear modulus, \( G \), exhibit a pressure-dependent derivative, and hence they were also fitted to the fourth-order finite Euler strain equations (Figs. 3 and 4), yielding \( C_{11}' = 8.7 \pm 0.5 \), \( C_{44}' = -0.38 \pm 0.11 \) GPa\(^{-1} \), \( G_0' = 1.98 \pm 0.2 \), and \( G' = -0.11 \pm 0.05 \) GPa\(^{-1} \). The measurements at high temperatures show a similar curvature with increasing pressure at 550 K and 673 K. We conclude that the pressure derivatives of the elastic moduli are similar at high temperatures (Figs. 3 and 4). The temperature derivatives of the bulk modulus, (\( \partial K_s / \partial T \))\(_P\), and shear modulus, (\( \partial G / \partial T \))\(_P\), at constant pressure were evaluated using a linear equation (Fig. 5). Since (\( \partial K_s / \partial T \))\(_P\) and (\( \partial G / \partial T \))\(_P\) are only slightly affected by pressure, the average value for (\( \partial K_s / \partial T \))\(_P\) and (\( \partial G / \partial T \))\(_P\) over pressure are \(-0.038\) GPa\(^{-1}\).

Table 1

<table>
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<tr>
<th>Temperature</th>
<th>P (GPa)</th>
<th>( \rho ) (g/cm(^3))</th>
<th>( C_{11} ) (GPa)</th>
<th>( C_{12} ) (GPa)</th>
<th>( C_{44} ) (GPa)</th>
<th>( K_s ) (GPa)</th>
<th>( G ) (GPa)</th>
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<td>299.7 (1.7)</td>
<td>112.7 (1.8)</td>
<td>115.3 (0.5)</td>
<td>175.2 (1.3)</td>
<td>106 (1.0)</td>
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<tr>
<td>3.7</td>
<td>3.721 (0.003)</td>
<td>328.0 (2.0)</td>
<td>122.5 (2.5)</td>
<td>118.5 (1.2)</td>
<td>197 (1.7)</td>
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<tr>
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<td>124 (2.6)</td>
<td>120.3 (0.7)</td>
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<td>7.4</td>
<td>3.792 (0.003)</td>
<td>354.5 (2.3)</td>
<td>129 (2.4)</td>
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<tr>
<td>9.0</td>
<td>3.828 (0.003)</td>
<td>362.6 (2.5)</td>
<td>133 (2.6)</td>
<td>123 (2.8)</td>
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<tr>
<td>10.9</td>
<td>3.855 (0.003)</td>
<td>372 (2.9)</td>
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Fig. 2. Example of the measured acoustic velocities compared with fitting results. Black and red circles: measured \( V_P \) and \( V_S \), respectively; black and red lines: fitting results. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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\[
(\pm 0.007) \text{GPa/K and } (\partial G/\partial T)_P = -0.017 (\pm 0.004) \text{GPa/K}, \text{ respectively (Fig. 6).}
\]

4. Discussion

The single-crystal elastic constants (\(C_{ij}\)) and the aggregate bulk (\(K_s\)) and shear moduli (\(G\)) of iron-bearing hydrous ringwoodite from analyses of the in situ Brillouin and X-ray diffraction measurements show two distinct features: (1) reduced elastic moduli by hydration at 300 K comparable to previous GHz-ultrasonic measurements (Jacobson et al., 2004), and (2) at high pressure, further reduction of elastic moduli at high-temperature (Figs. 3 and 4). As shown in previous studies, hydration reduces the elasticity of olivine, wadsleyite and ringwoodite at ambient conditions with the influence of water increasing from olivine to wadsleyite and ringwoodite. In forsterite with about 1 wt.% of water, the pressure derivatives of \(K\) and \(G\) (4.5 and 1.75, respectively) are higher than anhydrous forsterite (4.2–4.4 and 1.4–1.5 from Abramson et al., 1997 and Zha et al., 1996), causing a potential velocity crossover above about 3 GPa at 300 K (Mao et al., 2010). In Fe-free wadsleyite, the pressure derivatives of hydrous wadsleyite are similar to anhydrous wadsleyite (Mao et al., 2008b) (Table 1). In contrast to a previous high-pressure, GHz-ultrasonic study of hydrous Fe-bearing ringwoodite, which showed an increase in the pressure derivatives of both \(K\) (5.3) and \(G\) (2.0) (Jacobson and Smyth, 2006), here we find that the \(K'\) and \(G'\) of Fe-bearing hydrous ringwoodite are similar to anhydrous Fe-bearing ringwoodite. The discrepancy between the current pressure derivatives of Fe-bearing hydrous ringwoodite from the high-pressure GHz-ultrasonic study of Jacobson and Smyth (2006) is not yet understood, but it could possibly result from different hydration mechanisms (Panero, 2010) between the two samples synthesized under different conditions (run S20401 and run S29901 of Smyth et al., 2003) or it is possible that the silica gel introduced into the GHz-ultrasonic study hardened at high pressure causing a uniaxial stress on the sample in the direction of the acoustic wave propagation. We note that the static compression study of Fe-bearing hydrous ringwoodite of Manghnani et al. (2005) reported a similarly high derivative of the bulk modulus with \(K' = 6.2\). However, if the pressure derivatives of the moduli in hydrous phases are similar to anhydrous phases, as evidenced from the current study, the same amount of water in Fe-bearing ringwoodite can produce two to three times reductions in the elastic moduli compared to

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those of wadsleyite and olivine at high pressures because the initial (low pressure) moduli are more reduced in ringwoodite than in wadsleyite or olivine. Our experiments showed that all of the elastic moduli of hydrous ringwoodite exhibit similar pressure derivatives as compared to the anhydrous counterparts, but the addition of 1.1 wt.% H2O lower these constants by 5%–9% at high pressures and high temperatures (Figs. 3 and 4).

High temperatures further reduce the elastic moduli of hydrous ringwoodite at high pressures (compared with anhydrous ringwoodite) but does not have a noticeable effect on their pressure derivatives (Figs. 3 and 4). Specifically, increasing temperature by 100 K would decrease the elastic moduli of hydrous ringwoodite by 1.3–2.4% at 12 GPa, much greater than the observed 0.5–1.2% reduction for anhydrous ringwoodite (Figs. 3 and 4) (Sinogeikin et al., 2003). The bulk modulus is more sensitive to temperature than the shear modulus; the temperature derivative of $K_b$ is $-0.038$ ($\pm 0.007$) GPa/K, much greater than that of anhydrous ringwoodite ($-0.021$ ($\pm 0.002$) GPa/K) (Figs. 3, 4, and 6) (Higo et al., 2008; Mayama et al., 2005; Sinogeikin et al., 2003). The strong reduction in the elastic moduli of hydrous ringwoodite reflects a net effect of temperature, iron content, and hydration mechanism in our samples. 

Next we modeled the sound velocity of ringwoodite using a thermal Birch–Murnaghan equation of state and our experimental results (Figs. 7 and 8) (Duffy and Anderson, 1989). Based on these calculations, the presence of 1 wt.% H2O in ringwoodite causes an approximate 2.5 (±0.4)% reduction in the compressional ($V_P$) and shear ($V_S$) velocities at 300 K at a depth between 520 and 660 km. The velocity reduction is approximately two times more than that in Fe-wadsleyite with 2 wt.% H2O (Fig. 8) (Mao et al., 2011). Since the wadsleyite-to-ringwoodite transition is expected to occur at approximately 520-km depth, our findings indicate that seismic wave velocities at 520 to 660-km depth may be more sensitive to hydration than the layer above (Jacobsen et al., 2008; Mao et al., 2008a,b, 2010, 2011). Considering the overall effect of iron content, hydration and temperature, our model further shows that the $V_P$ and $V_S$ of hydrous ringwoodite along a mantle geotherm are $7\%$ (±0.5)% and $4.5$ (±0.7)% lower than those of the anhydrous ringwoodite (Fig. 8); these differences could be further enhanced by anelasticity effects (Karato, 1995), which need to be taken into account in future studies.

Quantifying the amount of H2O in the Earth’s mantle has been a long-standing challenge in deep-Earth research integrating seismology, mineral physics, and geodynamics. Using the measured sound velocities of hydrous ringwoodite at simultaneous high P–T, our results apply to interpreting seismic signatures in the potential water-rich region of the transition zone where ringwoodite is believed to be the

![Fig. 5. Effect of temperature on the bulk, $K_b$, and shear moduli, $G$, of hydrous ringwoodite at given pressure. A linear fit was applied to obtain the temperature derivative of $K_b$ and $G$. Vertical lines represent the propagated uncertainties (±1σ).](image)

![Fig. 6. Temperature derivative of the bulk (black) and shear moduli (red). Solid circles: hydrous ringwoodite, this study; open circles: anhydrous ringwoodite (Sinogeikin et al., 2003). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)](image)

![Fig. 7. Modeled $V_p$ and $V_s$ of ringwoodite in the transition zone. Blue dashed line: anhydrous ringwoodite (wadsleyite) at 300 K (Sinogeikin et al., 2003; Liu et al., 2009); solid line: ringwoodite with 1.1 wt.% H2O at 300 K (blue), 673 K (orange) and along the 1400 °C mantle geotherm (red) and wadsleyite with 2 mol% H2O (blue) (Mao et al., 2011). Vertical lines represent the propagated uncertainties (±1σ). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)](image)
Most abundant phase. Seismic velocity anomalies, together with the depth depression of the 660-km discontinuity at the bottom part of the transition zone lie in regions near subducting slabs and the surrounding mantle under Japan, Philippine Sea, Caribbean–South American Plate boundary, and western United States (Cao and Levandier, 2010; Flanagan and Shearer, 1998; Fukao et al., 2001, 2009; Grand et al., 2002; Houser et al., 2008; Mégnin and Romanowicz, 2000; Niu et al., 2005; Suetsugu et al., 2006, 2010; Tono et al., 2005; van der Lee et al., 2008; Widiyantoro et al., 1999). Using Fig. 9, we estimate the water content and temperature anomalies at the bottom of the transition zone near subducted slabs (Fig. 9). Since the P-wave travel-time spectra have higher resolution to image the stagnant slabs than the S-wave spectra, we focused on using the P-wave data to estimate the water content in the region. Thus, water content estimated from the VP anomaly and the depth depression of the 660-km discontinuity is less than 0.1 (±0.2) wt.% in the related regions, where estimated temperature anomalies are approximately 200–500 K (Fig. 9). The amount of water estimated here is three to five times less than that predicted in previous work, without considering the net effect of water and temperature (Cao and Levandier, 2010; Houser et al., 2008; Suetsugu et al., 2010). The water content estimated here for the lowest portion of the Earth’s transition zone is consistent with recent studies using seismicity (Green et al., 2010), electromagnetic induction (Kelbert et al., 2009), and electronic conductivity of wadsleyite and ringwoodite (Yoshino et al., 2008).

Our results here confirm the idea that the presence of water can lower the elasticity and sound velocities of mantle minerals.
However, the magnitude of the reduction by a given amount of water is much greater than previously thought, when the combined effect of hydration, temperature, and pressure is taken into consideration. Specifically, hydration strongly increases the magnitude of the temperature derivative of the bulk modulus. Compared to previous ultrasonic and static compression (X-ray diffraction) studies, the increased temperature derivatives lead to an enhanced reduction in the sound velocity by hydration at high pressures. Ignoring the combined effect of hydration, temperature, and pressure can thus lead to an overestimation of the mantle water budget. In addition, the effect of hydration on the elasticity of other major mantle phases, including garnet and pyroxene, also needs to be taken into account in future studies evaluating the potential mantle H2O content.

Acknowledgments

We thank I. Kantor and K. K. Zhuravlev for experimental assistance and A. Wheat for manuscript editing. Experiments performed at GeoSoilEnviroCARS (Sector 13), Advanced Photon Source (APS), Argonne National Laboratory, was supported by the National Science Foundation (NSF) through EAR-0622171 and U.S. Department of Energy (DE-FG02-94ER14466). Use of the Advanced Photon Source was supported by the DOE, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357. Work and GECARS was partially supported by COMPR, the Consortium for Materials Properties Research in Earth Sciences under NSF Cooperative Agreement EAR 10-43050. This research was supported by the NSF grants EAR-0838221 and EAR-1056760 to J.F. Lin, EAR-0748707 to S.D. Jacobsen, EAR-0738510 to T.S. Duffy, and EAR07-11150 and 11-13369 to J.R. Smyth. Additional support was provided by the Carnegie/DOE Alliance Center (CDAC) and Energy Frontier Research in Extreme Environments (Effree) to J.F. Lin, and by the David and Lucile Packard Foundation to S.D. Jacobsen.

Appendix A. Supplementary data

Supplementary data to this article can be found online at doi:10.1016/j.epsl.2012.03.001.

References


doi:10.1016/j.epsl.2012.03.001


Please cite this article as: Mao, Z., et al., Sound velocities of hydrous ringwoodite to 16 GPa and 673 K, Earth Planet. Sci. Lett. (2012), doi:10.1016/j.epsl.2012.03.001