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## Earth and Planetary Science Letters

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# Crystallographic preferred orientation of wadsleyite and ringwoodite: Effects of phase transformation and water on seismic anisotropy in the mantle transition zone



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#### ARTICLE INFO

# Article history: Received 9 January 2014 Received in revised form 29 March 2014 Accepted 31 March 2014 Available online 13 May 2014 Editor: I. Brodholt

Keywords:
wadsleyite
ringwoodite
crystallographic preferred orientation
water
mantle transition zone
seismic anisotropy

#### ABSTRACT

Simple-shear deformation experiments on wadsleyite and ringwoodite aggregates were performed at 15–18 GPa and 1473–1873 K to investigate the effect of water on the development of the crystallographic preferred orientation (CPO) of wadsleyite and ringwoodite. The [001] axes of wadsleyite are preferentially sub-parallel to the shear direction and the [010] axes of wadsleyite concentrate in the direction of the shear-plane normal for water content less than 9000 ppm H/Si (i.e.,  $\sim$ 540 wt.ppm) in wadsleyite. At higher water content in wadsleyite ( $\geq$ 9000 ppm H/Si), the concentration of the [100] axes of wadsleyite becomes stronger than that of the [010] axes in the direction of the shear-plane normal. The fabric strength of wadsleyite having low water content (<3000 ppm H/Si) was much stronger than that having water content higher than 3000 ppm H/Si. The magnitude of  $V_{SH}/V_{SV}$  (the ratio of horizontally and vertically polarized shear wave velocities) in the upper transition zone is well explained by the flow of wadsleyite aggregates having water content higher than 3000 ppm H/Si. The back transformation from ringwoodite to wadsleyite may help to suppress the increase in fabric strength of wadsleyite during the deformation. In contrast to wadsleyite, the fabric strength of ringwoodite CPOs was not sufficient to cause robust seismic anisotropy even though the deformation of ringwoodite was controlled by dislocation creep. Thus, the lower transition zone is expected to be largely isotropic.

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#### 1. Introduction

The seismic anisotropy in the mantle transition zone (upper part: 410 to 520 km depth; lower part: 520 to 660 km depth) is mainly controlled by the crystallographic preferred orientation (CPO) of wadsleyite and ringwoodite, and the wadsleyite and ringwoodite CPOs are formed through the mantle flow in the mantle transition zone. Robust anisotropic structures in the upper transition zone have been reported by seismological observations (e.g., Panning and Romanowicz, 2006), though some studies pointed out that the lower transition zone is largely isotropic (Fouch and Fischer, 1996). It has been recognized that magnitude of the ratio of horizontally polarized shear wave velocity ( $V_{SH}/V_{SV}$ ) in the transition zone is much weaker than that in the upper mantle, though the both positive and negative values of  $V_{SH}-V_{SV}$  in the upper transition zone have

been reported (positive: e.g., Panning and Romanowicz, 2006, negative: e.g., Visser et al., 2008). The observed seismic anisotropy signatures, such as the direction of shear wave splitting and the  $V_{SH}/V_{SV}$  are controlled by the flow geometry and the characteristics of the CPO of the minerals in the upper transition zone.

In order to understand the relationship between flow geometry and wadsleyite CPO, slip systems controlling the deformation have been identified in many experimental studies. Sharp et al. (1994) conducted deformation experiments on wadsleyite at 14 GPa and  $\geq$ 1723 K using stress-relaxation techniques, and they reported that the dominant slip systems were [100](010) and [001](010). Similar deformation experiments have been conducted under the conditions of the mantle transition zones (14–19 GPa and 1573–2273 K), and a variety of slip systems in wadsleyite were identified ( $\frac{1}{2}$ (111){101}, [100](010), [100](001), [100](011], [100]{021}, [010](001), [010]{101}, and (101)(010): Thurel and Cordier, 2003; Thurel et al., 2003). Based on the results from stress-relaxation tests on wadsleyite, Tommasi et al. (2004) proposed a mineralogical model in which the polarization direction of the fast S-wave is parallel to the shear direction and the  $V_{SH}/V_{SV}$ 

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is higher than 1 in the case of horizontal flow. Demouchy et al. (2011) conducted stress-relaxation tests on wadsleyite in a simpleshear geometry at 16 GPa and 1673 K, and they reported that concentrations of the [100] and [001] axes in the shear direction under the conditions of water content is lower and higher than  $\sim$ 6000 ppm H/Si, respectively. However, the exact values of pressure, temperature, and stress at which much of the CPO develops are unknown in stress-relaxation tests (e.g., Karato et al., 2008). Not only stress-relaxation tests but also constant strain-rate deformation experiments have been conducted. Xu et al. (2005) performed deformation experiments on wadsleyite in a simpleshear geometry at 15 GPa and 1600-1700 K using a rotational Drickamer apparatus, and they reported weak alignments of the [010] and [001] axes in the shear direction and shear plane normal, respectively. More recently, Kawazoe et al. (2013) performed simple-shear deformation experiments on wadsleyite at 17.6 GPa and 1800-1900 K by using a deformation-DIA apparatus, and developments of a fabric showing strong concentrations of the [001] and [010] axes in the shear direction and the shear plane normal, respectively, were observed. However, Kawazoe et al. (2013) reported that the magnitude of  $V_{SH}/V_{SV}$  calculated from the observed wadsleyite CPO exceeds that in the mantle transition zone reported from geophysical observations (0.992-0.996: Visser et al., 2008) even at a shear strain of  $\gamma = 0.4$ .

Some slip systems in ringwoodite have been reported in experimental studies. Karato et al. (1998) conducted stress-relaxation tests of ringwoodite (Mg# = 60) at 16 GPa and 1600 K, and they reported activation of two slip systems  $(\frac{1}{2}\langle 110\rangle \{111\})$  and  $\frac{1}{2}\langle 110\rangle \{100\}$ ). Slip on  $\frac{1}{2}\langle 110\rangle \{110\}$  is also reported at 22 GPa and 1673–1773 K (Thurel, 2001). A series of deformation experiments using a deformation-DIA apparatus combined with the in-situ CPO measurement technique showed that activation of the  $\frac{1}{2}\langle 110\rangle \{111\}$ slip system was dominant at 6–10 GPa and room temperature (Wenk et al., 2005). More recently, activation of the  $\frac{1}{2}\langle 110\rangle \{111\}$ slip system has been reported at 23 GPa and 1800 K in shear deformation experiments using a rotational Drickamer apparatus combined with the in-situ CPO measurement technique (Miyagi et al., 2013). Using the ab initio total-energy calculation technique, Carrez et al. (2006) showed that  $\frac{1}{2}\langle 110\rangle \{110\}$  and  $\frac{1}{2}\langle 110\rangle \{111\}$  are the easiest slip systems in ringwoodite at 20 GPa and 0 K.

The seismic anisotropy in the upper mantle is interpreted in terms of olivine fabrics, and many experimental studies have revealed that the development of olivine fabrics are controlled by pressure, stress, and the amount of water dissolved in olivine (e.g., Jung and Karato, 2001; Katayama et al., 2004; Ohuchi et al., 2011). In contrast to olivine, the effects of the physical and chemical environments on the CPOs of wadsleyite and ringwoodite have not been fully investigated yet. Thus, further extensive studies are needed to clarify the variations of the CPO of wadsleyite and ringwoodite.

#### 2. Experimental procedure

The starting materials for wadsleyite and ringwoodite aggregates were prepared from samples of San Carlos olivine (Fo<sub>90</sub>). Inclusion-free crystals of olivine were carefully selected and crushed down to several micrometers using an agate mortar. The fine-grained olivine powders were dried at 573 K for  $\sim\!\!3$  h in a furnace and then stored at 383 K for  $\sim\!\!12$  h in a vacuum oven. The dried fine-grained powders of olivine were placed into a nickel capsule and were sintered at 4.0 GPa and 1223 K for 1.5 h using a Kawai-type multi-anvil apparatus at Ehime University. The average grain size of the hot-pressed sample was 15.3 µm, and the water content dissolved in the hot-pressed sample was 4015 ppm H/Si. An oriented single crystal olivine (Fo<sub>90</sub>) from San Carlos was used as starting material for the M0327 and 0329 runs. The hot-pressed

sample and the single crystal olivine were core-drilled with a diameter of 0.8 mm, and then sectioned to have a thickness between 200 and 250  $\mu$ m. In order to remove water dissolved in the olivine samples, some sectioned parts of the OT922 sample (OT922F) and all of the sections of olivine single crystal were fired at 0.1 MPa and 1170 K under reducing conditions (log  $f_{02} \sim -16$  bar) for 6 h.

Simple-shear deformation experiments on wadsleyite aggregates at pressures P = 15-18 GPa, temperatures T = 1473-1873 K, and shear strain rates of  $3.6 \times 10^{-5}$  –  $2.5 \times 10^{-4}$  s<sup>-1</sup> were performed using a deformation-DIA apparatus installed at Ehime University. Experimental conditions and results are summarized in Table 1. A deformation experiment (Y0313) was conducted using a deformation-DIA apparatus at SPring-8. The MA6-6 system (Nishiyama et al., 2008) with a truncated edge length of the second-stage tungsten carbide anvils of 3 mm was used for the experiments. The 5.5-3 type cell assembly designed by Ohuchi and Irifune (2014) was adopted for the present experiments. A sectioned olivine sample was placed into a platinum capsule and then sandwiched between two alumina pistons (tungsten pistons were used for the M0310, M0315, M0318, and M0320 runs). About 10 wt.% of distilled water was added to the platinum capsule using a microsyringe and then the capsule was sealed with a platinum lid in the M0310, M0315, M0318, and M0320 runs (Table 1). Distilled water was not added to the platinum capsule in other runs.

Main-ram load was first raised to the desired value (1 MN) at a rate of 0.5 MN/h, and then temperature was increased at a rate of  $\sim$ 25 K/min. The generated pressure was estimated from the relationship between the sample pressure and the press load reported. We reevaluated the relationship between the sample pressure and the press load for the 5.5-3 type cell assembly in the deformation-DIA apparatus installed at Ehime University from the phase transitions of (Mg,Fe)<sub>2</sub>SiO<sub>4</sub> ( $\alpha$ - $\beta$ - $\gamma$  transformations: Frost, 2003; Kerschhofer et al., 1998) (Fig. S1). The uncertainty in pressure was  $\pm 1.3$  GPa. Temperature was monitored by a W<sub>97</sub>Re<sub>3</sub>-W<sub>75</sub>Re<sub>25</sub> thermocouple placed along one of the diagonal directions of the cubic (Mg,Co)O pressure media. The thermocouple wires were connected to each other via two tungsten rings that were placed on the outside of the platinum capsule. The temperature gradient between the central part and the edge of the sample was less than 50 K (Ohuchi and Irifune, 2014). After the temperature reached the desired value, the sample was annealed for 20 min. Then the upper and lower anvils were advanced at a constant rate by operating the deformation rams. In order to evaluate the microstructures of samples just before the deformation process, five samples (M0313, M0315, M0317, M0320, and M0321) were quenched after keeping a constant temperature (1673-1773 K) for 20 min without the deformation process. Shear strain was measured by the rotation of a platinum strain-marker, which was initially placed perpendicular to the shear direction. Strain rate was calculated under the assumption that the strain-marker rotated at a constant rate during the deformation experiment. The uncertainty in the strain rate was within 15%.

The recovered samples were cut with a low-speed saw. These were then impregnated with epoxy under a vacuum and polished using alumina powder (down to 1.0 µm) followed by 0.06 µm colloidal silica suspension. The recovered phases in the samples were identified by Raman spectroscopy. The CPOs of wadsleyite and ringwoodite grains were evaluated by the indexation of the electron backscattered diffraction (EBSD) patterns using a JEOL JSM-7000F FE-SEM at Ehime University. EBSD patterns were generated via the interaction of a vertical incident electron beam with a polished sample inclined at 70° with respect to the horizontal. The EBSD patterns were indexed using the CHANNEL5 software from HKL Technology. The EBSD pattern of each grain was obtained at 20 kV acceleration voltage and 7.5 nA probe current. Measurements were performed on a grain-by-grain basis and in

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