

# Effect of water on olivine-wadsleyite phase boundary in the $(\text{Mg, Fe})_2\text{SiO}_4$ system

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[1] The phase boundary between San Carlos olivine and its high pressure phase, wadsleyite, have been studied comparatively in dry and water saturated condition. By comparing saturated and dry samples from the same experimental run, we determine that both boundaries of the two-phase coexisting loop shift towards lower pressure or lower iron content, and the pressure width of the loop decreases to 0.3 GPa under saturated conditions at 1473 K. Thus, the presence of water in the Earth's mantle will sharpen the 410-kilometer seismic discontinuity, shallow the depth of the discontinuity, and reduce the velocity jump across the discontinuity. **INDEX TERMS:** 3630 Mineralogy and Petrology: Experimental mineralogy and petrology; 3670 Mineralogy and Petrology: Minor and trace element composition; 3924 Mineral Physics: High-pressure behavior; 3939 Mineral Physics: Physical thermodynamics; 1025 Geochemistry: Composition of the mantle. **Citation:** Chen, J., T. Inoue, H. Yurimoto, and D. J. Weidner, Effect of water on olivine-wadsleyite phase boundary in the  $(\text{Mg, Fe})_2\text{SiO}_4$  system, *Geophys. Res. Lett.*, 29(18), 1875, doi:10.1029/2001GL014429, 2002.

## 1. Introduction

[2] The pressure-induced  $\alpha$ (olivine)- $\beta$ (wadsleyite) phase transformation in the  $(\text{Mg, Fe})_2\text{SiO}_4$  system is generally considered to be responsible for the 410-km seismic discontinuity [Ringwood, 1975; Katsura and Ito, 1989]. Yet, several details remain that question whether the olivine transformation in a mantle with a pyrolite composition can predict the observed discontinuity. Previous phase relation studies on a simple  $(\text{Mg, Fe})_2\text{SiO}_4$  system show that the transformation from  $\alpha$  to  $\beta$  phase occurs in a divariant loop with a pressure interval of  $\sim 0.6$  GPa over which both  $\alpha$  and  $\beta$  phase coexist [Akaogi *et al.*, 1989; Katsura and Ito, 1989]. This mineral physics result yields a wider discontinuity than some seismic observations at 410-km with a width less than 10 km ( $\sim 0.3$  GPa) [Benz and Vidale, 1993; Neele, 1996; Vidale *et al.*, 1995] although

some other studies indicate a reduced discrepancy [Irifune and Isshiki, 1998; Stixrude, 1997]. The velocity jump across the discontinuity has often been determined, with large uncertainty [Nolet *et al.*, 1994; Shearer and Flanagan, 1999], to be smaller than expected from this transformation in a pyrolite mantle [Duffy and Anderson, 1989].

[3] All above mentioned shortcomings of the standard model suggest that other parameters need to be considered. We experimentally define the role of a volatile chemical component that may be either ubiquitous or may vary widely in abundance, namely water. The occurrence of water in mantle-derived magmas and the occurrence of hydrous minerals such as mica and amphibole in samples of the mantle brought up as xenoliths in volcanic eruptions testify to the presence of water in the upper mantle [Bell and Rossman, 1992]. Since the  $\alpha$  and  $\beta$  phases take very different amounts of water in their structures [Inoue *et al.*, 1995; Kohlstedt *et al.*, 1996], presence of water can also notably effect the phase boundary between these two phases. Here we report our experimental result demonstrating that presence of water in the Earth's mantle will sharpen the 410-kilometer seismic discontinuity, shallow the depth of the discontinuity, and reduce the velocity jump across the discontinuity.

## 2. Experimental

[4] The starting material in our experiments is powdered San Carlos olivine with a composition of  $(\text{Mg}_{1-x}\text{Fe}_x)_2\text{SiO}_4$  ( $x \sim 9\%$ ). Hydrous samples are prepared by adding distilled water into the olivine powder. Both dry and wet samples are enclosed into a welded Au75Pd25 alloy capsule separately before loaded in a high pressure cell. A water bath is used during welding the capsule of the wet sample to prevent the water in the capsule from vaporization. Observation of bubbles (caused by an excess of water, but not melt) in the recovered sample indicates that the amount of water during the annealing is saturated in the wet samples. In all the experiments, the dry and wet capsules are loaded at symmetrical positions in one high pressure cell to ensure an identical pressure and temperature condition for both samples during annealing. End member samples ( $x = 0$ ) are also prepared from the starting material of MgO and  $\text{SiO}_2$

mixture.  $\text{Mg}(\text{OH})_2$  is added into the starting material as water source for the hydrous sample with the equivalent of 11.3 wt% of  $\text{H}_2\text{O}$ .

[5] High pressure experiments were carried out using a uniaxial split-sphere apparatus (USSA2000) [Liebermann and Wang, 1992] at SUNY at Stony Brook. The edge length of the truncation on the second-stage cubic-anvil corner is 8.0 mm in the experiments of the  $(\text{Mg}_{0.91}\text{Fe}_{0.09})_2\text{SiO}_4$  system and 5.0 mm in those of the  $\text{Mg}_2\text{SiO}_4$  system. The pressure medium is octahedral semi-sintered magnesia with the edge length of 14 mm in the  $(\text{Mg}_{0.91}\text{Fe}_{0.09})_2\text{SiO}_4$  experiments and 10 mm in the  $\text{Mg}_2\text{SiO}_4$  experiments, respectively. Pressure was calibrated using phase transformations in some reference materials [Liebermann and Wang, 1992], including  $\alpha - \beta$  transition [Akaogi et al., 1989] in our anhydrous samples. The relative precision of two different experiments is estimated about 0.05 GPa while the absolute value of pressure may have an uncertainty of 0.5 GPa. Temperature is monitored using a W97Re3–W75Re25 thermocouple. The sample is first compressed then heated by an internal cylindrical furnace made of graphite or  $\text{LaCrO}_3$ . All annealings are done at a temperature of 1473 K. Annealed samples are quenched by turning off the electric heating power. Recovered samples are examined by micro-focus x-ray diffractometer and electron probe microanalyzer for phase identification and composition determination. The water content in all the samples are measured by using secondary ion mass spectroscopy with a natural amphibole crystal as a standard [Miyagi and Yurimoto, 1995] (1.66 wt% of  $\text{H}_2\text{O}$ ). The accuracy of the measurements mainly depends on the scattering of the standard calibration. In the present analysis, the uncertainty is estimated to be less than 10%.

### 3. Result and Discussion

[6] The experimental conditions and results are summarized in Table 1. The experiments are conducted from 12.6 to 14.7 GPa. In the  $\text{Mg}_2\text{SiO}_4$  system, no difference in the phase boundary between dry and wet condition is observed within the pressure interval of 0.5 GPa, indicating the phase boundary change caused by water (if any) is not greater than 0.5 GPa. In the  $(\text{Mg}_{0.91}\text{Fe}_{0.09})_2\text{SiO}_4$  system, however, a significant change in the  $\alpha - \beta$  coexisting loop boundary is observed. Coexistence of  $\alpha$  and  $\beta$  phases does not appear until the pressure increases up to 13.1 GPa in dry sample whereas  $\alpha$  and  $\beta$  phases start to coexist in wet sample at a pressure of 13.0 GPa. The single  $\beta$  phase appears at 13.25 GPa in wet sample and 13.6 GPa in dry sample. Figure 1 shows Fe/Mg partitioning in the sample at different pressures and reconstructed phase diagrams for  $\alpha - \beta$  transformation under dry and wet conditions from our experimental data. Both upper ( $\alpha + \beta$  to  $\beta$ ) and lower ( $\alpha$  to  $\alpha + \beta$ ) boundaries of the  $\alpha - \beta$  coexisting loop shift to lower iron content under wet condition. The amount of shift of the upper boundary increases notably with pressure, resulting an upward-concave divariant loop. Consequently the depth of the transition becomes shallower and the depth interval of the  $\alpha - \beta$  coexisting region becomes narrower under wet condition than those under dry condition at the experimental temperature of 1473 K. The observation of decrease in the depth interval caused by water is contrary to

**Table 1.** Experimental Conditions and Results

Run No.	Pressure (GPa)	Time (min)	Dry	Phase recovered					
				Fe/(Mg+Fe)			H <sub>2</sub> O wt%		
				$\alpha$	$\beta$	Kd	$\alpha$	$\beta$	Kd'
<b>Mg<sub>2</sub>SiO<sub>4</sub></b>									
2623	13.7	60	Dry	0	–	–	0.21	–	–
			Wet <sup>a</sup>	0	–	–	0.76	–	–
2595	14.2	40	Dry	–	0	–	–	0.2	–
			Wet <sup>a</sup>	–	0	–	–	3.8	–
2577	14.7	45	Dry	–	0	–	–	0.3	–
			Wet <sup>a</sup>	–	0	–	–	3.5	–
<b>(Mg<sub>0.91</sub>Fe<sub>0.09</sub>)<sub>2</sub>SiO<sub>4</sub></b>									
2387	12.6	63	Dry	0.081	–	–	NA	–	–
			Wet	0.073	–	–	NA	–	–
2746	12.75	30	Dry	0.083	–	–	NA	–	–
			Wet	0.078	–	–	NA	–	–
2850	12.95	60	Dry	0.078	–	–	0.10	–	–
			Wet <sup>b</sup>	0.030	–	–	0.29	–	–
3061	13.0	60	Dry	0.084	–	–	NA	–	–
			Wet	0.041	0.135	3.7	0.37	1.9	5.1
2837	13.05	60	Dry <sup>c</sup>	0.081	–	–	0.17	–	–
			Wet	0.037	0.126	3.8	0.71	3.4	4.8
2847	13.1	60	Dry	0.077	0.172	2.5	(0.12)	NA	NA
			Wet <sup>d</sup>	0.032	0.087	2.9	0.64	3.4	5.3
3064	13.1	180	Dry	0.086	0.160	2.0	(0.06)	NA	NA
			Wet <sup>b</sup>	0.029	0.088	3.2	0.55	2.5	4.6
2745	13.25	30	Dry	0.060	0.141	2.6	(0.10)	NA	NA
			Wet	–	0.074	–	–	3.0	–
3066	13.6	60	Dry	–	0.080	–	–	0.07	–
			Wet <sup>d</sup>	–	0.074	–	–	2.6	–
2970	13.7	60	Dry	–	0.079	–	–	0.10	–
			Wet <sup>d</sup>	–	0.078	–	–	2.8	–
2972	13.9	60	Dry	–	0.083	–	–	0.07	–
			Wet <sup>d</sup>	–	0.074	–	–	2.2	–
2717	14.0	34	Dry	–	0.090	–	–	NA	–
			Wet	–	0.088	–	–	NA	–
2729	14.4	33	Dry	–	0.083	–	–	0.10	–
			Wet	–	0.085	–	–	2.6	–

NA, not available due to small grain size and/or minor quantity, or not measured.

<sup>a</sup>the corresponding phase does not exist.

<sup>a</sup>liquid and small amount of clinoenstatite also exist in the recovered sample.

<sup>b</sup>liquid exist in high temperature region.

<sup>c</sup>minor amount of  $\beta$  exists at low temperature region.

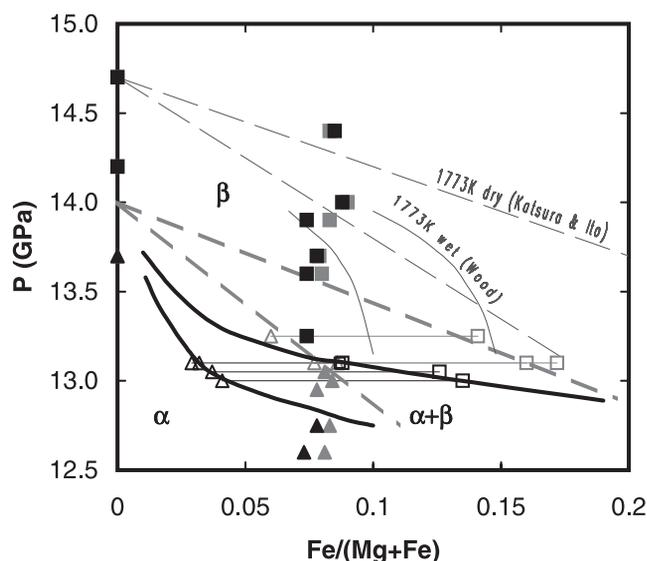
<sup>d</sup>phase E exists in low temperature region.

Parentheses show the results of  $\alpha$  plus  $\beta$ , because the crystal sizes hindered the individual measurements.

Kd =  $(\text{Fe}/\text{Mg})_{\beta}/(\text{Fe}/\text{Mg})_{\alpha}$ .

Kd' =  $\text{Wt}(\text{H}_2\text{O})_{\beta}/\text{Wt}(\text{H}_2\text{O})_{\alpha}$ .

prediction of the theoretical calculation by Wood at 1773 K [Wood, 1995]. As shown in Figure 1, both the experimental result and Wood's calculation indicate a lower pressure phase transition in wet condition, which is in agreement with basic thermodynamics because the greater solubility of water in  $\beta$  than in  $\alpha$  phase implies a lower activity of  $(\text{Mg}, \text{Fe})_2\text{SiO}_4$  in  $\beta$  phase, and therefore implies transition at lower pressure than in the dry case. However, Wood's theoretical model results in a downward-concave divariant loop, which in turn increases the depth interval of the loop (Figure 1). Differences in water concentration between the experiments (water saturated) and theoretical calculation (water undersaturated) may give rise to the discrepancy. Based on the Phase Rule ( $F = C - P + 2$ ), undersaturated water in  $\text{Mg}_2\text{SiO}_4$  system may make the univariant phase boundary between  $\alpha$  and  $\beta$  divariant, and therefore broaden the boundary, whereas existence of fluid phase (saturated



**Figure 1.** The  $\alpha + \beta$  region of phase diagram for  $\text{Mg}_2\text{SiO}_4\text{-Fe}_2\text{SiO}_4$  at 1473 K. Black and red symbols represent the experimental data under wet and dry conditions, respectively. Triangles and squares represent the data of  $\alpha$  and  $\beta$  phases, respectively. Solid symbols indicate a single phase, and open symbols indicate that the  $\alpha$  and  $\beta$  phases (linked by broken lines) coexist. Black solid lines and red dash lines indicate the loop boundaries under wet and dry conditions. For comparison, the calculated loop boundaries under wet condition [Wood, 1995] at 1773 K together with experimental loop boundaries under dry condition [Katsura and Ito, 1989] are also superposed in the figure as blue solid lines and blue dash lines.

water) makes the divariant loop back to univariant. In the  $(\text{Mg, Fe})_2\text{SiO}_4$  system, however, the  $\alpha - \beta$  phase boundary is already divariant in absence of water. Differences between the effects of undersaturated water and saturated water on the width of  $\alpha - \beta$  loop need further experiments to clarify.

[7] Kinetics is an important issue in phase relation study. Within the  $\alpha - \beta$  two-phase loop, iron partitioning between the  $\alpha$  and  $\beta$  phases reaches equilibrium through a diffusion process, during which the experimental partition coefficient,  $K_d (= [\text{Fe}/\text{Mg}]_{\beta}/[\text{Fe}/\text{Mg}]_{\alpha}$ , where  $\text{Fe}/\text{Mg}$  denotes mole ratio), increases from 1 to the equilibrated value. In most of our experiments, samples are annealed for 60 min at 1473 K. A comparative test at 13.1 GPa (run # 2847 and 3064) with different annealing times (180 min versus 60 min) does not yield any time dependence of the experimental  $K_d$ , which indicates that the equilibrated state is reached in the experiments. As listed in Table 1, partition coefficients of the samples after dry annealing range from 2.0 to 2.6, which is consistent with the previous studies [Akaogi et al., 1989; Katsura and Ito, 1989].

[8] In our experiments, annealed  $\beta$  phase under wet condition contains 1.9–3.4 wt%  $\text{H}_2\text{O}$ , being consistent with previous experiments [Kohlstedt et al., 1996]. Water content in wet  $\alpha$  phase reaches a value as high as 0.7 wt%. This is the highest water content in olivine reported in previous studies [Kohlstedt et al., 1996]. The partition coefficient  $K_d' (= [\text{H}_2\text{O}]_{\beta}/[\text{H}_2\text{O}]_{\alpha}$ , where  $[\text{H}_2\text{O}]$  denotes the water content in

wt%) within the  $\alpha - \beta$  coexisting loop is about 5, indicating that  $\beta$  phase can take up 5 times more water than  $\alpha$  phase at the same P-T condition. The  $K_d'$  value is half of that used in Wood's calculation [Wood, 1995]. Some samples annealed under dry condition also contain notable amount of water in our experiments. It is not very clear if this is caused by moisture contained in our fine powdered sample or diffusion of hydrogen during the annealing.

[9] As mineral physics studies and seismic observations become more accurate, the critical tests of the acceptability of the  $\alpha - \beta$  phase transformation being responsible for the 410 km discontinuity become more rigorous. Issues of the sharpness and amplitude of the discontinuity in sound velocity, as well as definition of factors controlling the topography of the discontinuity challenge our chemical and mineralogical models. Seismological studies suggest that the transformation interval should be less than 0.3 GPa and the seismic velocity jump should be 3–5% for P wave and 4–4.6% for S wave [Benz and Vidale, 1993; Neele, 1996; Vidale et al., 1995], although some studies indicate the jump may be larger [Shearer and Flanagan, 1999]. Mineral physics studies indicate that the pressure interval for the dry  $\alpha - \beta$  phase transformation can be as wide as 0.6 GPa in the simple  $(\text{Mg, Fe})_2\text{SiO}_4$  system [Akaogi et al., 1989; Katsura and Ito, 1989]. A recent study by Irifune and Isshiki [1998] shows that the iron content in olivine decreases significantly with increasing pressure due to the formation of a relatively iron-rich majorite in a multi-component system with pyrolite composition. Consequently, the pressure interval for  $\alpha - \beta$  phase transformation can be notably reduced. A nonlinear model for the phase transition in the divariant loop illustrates a reduced apparent pressure interval for  $\alpha - \beta$  phase transformation [Stixrude, 1997]. Our experimental result demonstrates that presence of water can also reduce the pressure interval of  $\alpha - \beta$  phase transformation from 0.6 GPa to 0.3 GPa, which corresponds to a depth of 9 km, very close to the seismic observation. Water content in the mantle is currently not well constrained, however, the presence of water will reduce the pressure interval. Therefore, on account of the iron partitioning among minerals in the mantle, the nonlinear transition effect and the result presented here for the effect of water, the inconsistency between mineral physics studies and seismic observations regarding the sharpness of the 410 km discontinuity is probably negligible.

[10] Presence of water can also affect the amplitude of the velocity discontinuity. Limitation in the resolution and differences in the style of interpretation of seismic data yield a large uncertainty in determining the discontinuity amplitude, especially in longitudinal velocity [Nolet et al., 1994; Shearer and Flanagan, 1999]. Most studies [Duffy et al., 1995; Li et al., 1998] on elastic properties of the  $\alpha$  and  $\beta$  phases indicate that the velocity jump at the  $\alpha - \beta$  phase transition is too high with respect to the observed amplitude, and the discrepancy is more significant in longitudinal velocity than in transverse velocity. One possibility is for the olivine content in the upper mantle to be reduced from ~60 vol.% in pyrolite model down to 30–50 vol.%. A recent study by Yusa and Inoue [1997] shows that the bulk modulus of the hydrous  $\beta$  phase is 5–11% smaller than that of the anhydrous  $\beta$  phase. Consequently the bulk sound velocity of the  $\beta$  phase is reduced by ~3.7% due to

the present of water. If we assume that the velocity reduction by water in the  $\alpha$  phase is not as significant as for the  $\beta$  phase, because the  $\alpha$  phase takes much less water into its structure, the present of water will reduce the amplitude of the 410 km discontinuity. If the water lowers elastic properties for the  $\beta$  phase in a similar way as for the  $\gamma$  phase (ringwoodite) [Inoue *et al.*, 1998], we can expect that the longitudinal velocity of the  $\beta$  phase will be reduced more than the transverse velocity. Therefore, it may be possible to compensate both discrepancies in the longitudinal velocity and the transverse velocity by the water weakening. However, more details about the elastic properties (e.g., temperature and pressure dependence of moduli at the discontinuity) of these phases need to be studied.

[11] Seismic observations on correlation between the depth of 410 and 600 discontinuities are divergent [Helffrich, 2000]. Some studies find the two discontinuities are anticorrelated [Clarke *et al.*, 1995], but some do not [Stammler *et al.*, 1992]. Flanagan and Shearer [1998] and Gu *et al.* [1998] report that the depth of the 410 discontinuity is rather correlated with surface geology. The presence of water will make the 410 discontinuity shallower for the same temperature. Thus, lateral variations in the water content may change the topography of the 410 discontinuity although the maximum 'water-induced' uplift of the '410' topography expected from our experiments is about 15 km whereas seismic observations indicate the topography can be as shallow as 380 km in some exceptional cases [Gu *et al.*, 1998]. Increased water content should sharpen the discontinuity, reduce the magnitude of the jump and shallow the depth of the discontinuity. A seismic discovery of a correlation of these three features will implicate lateral variations in water content in the Earth's mantle. Quantitatively applying our experimental result requires high water concentration in the mantle. While the amount of water in the mantle is still poorly constrained, our result supplies important experimental information for linking seismic observations to the presence of water in the mantle. Studies with controlled undersaturated-water condition and mantle-composition system (e.g., presence of pyroxene) will help to further understand the role of water in the Earth's interior.

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