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An experimental study of grain-scale microstructure evolution during the olivine–wadsleyite phase transition under nominally "dry" conditions

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ABSTRACT

The grain-scale microstructure evolution during the phase transformation of olivine to wadsleyite was investigated using a multi-anvil high-pressure apparatus. We identified three stages in the microstructure evolution during olivine to wadsleyite phase transformation: (1) nucleation of wadsleyite along potential nucleation sites (e.g., grain boundaries (and grain corners), inside of grains (dislocations, stacking faults)), (2) development of an interconnected network of fine-grained wadsleyite along grain boundaries (3) growth of wadsleyite into olivine. Our observations suggest that at low over pressures (<3 GPa), phase transformation takes place mainly via inter-crystalline mechanism. As the over-pressure increases (>3 GPa), intra-crystalline nucleation makes a significant contribution to the volume fraction transformed.

The rheological properties of a sample undergoing the phase transformation are controlled by the grain-size and the connectivity of newly formed grains. Based on the model of grain-size evolution involving both nucleation and growth, we show that the initial grain-size of wadsleyite is controlled by the competition between nucleation rate and growth rate when phase transformation takes place near the equilibrium boundary (at relatively higher temperatures, >1200 K) but mostly by critical size for nucleation when phase transformation takes place away from the equilibrium boundary (at relatively low temperatures, <1200 K). After the newly formed grains cover the potential sites for nucleation ("site-saturation"), grain-size is governed by grain-growth till ~50% volume fraction is transformed. Due to the temperature sensitivity of initial size and of the grain-growth rate, the size of newly formed grains in subducting slabs is highly sensitive to the temperature at which the phase transformation occurs. At low temperatures, the size of new grains is small due to small initial grain-size and sluggish grain-growth. In contrast, grain-size during phase transformation is large at high temperatures due to the large initial grain-size and subsequent fast grain-growth kinetics. Consequently, substantial reduction of slab strength is expected in the cold regions of a subducting slab but not in the warm regions.

1. Introduction

This paper reports the results of an experimental study on the grain-scale microstructural evolution during the olivine to wadsleyite phase transformation. The grain-scale microstructures have important implications for the rheological properties of a slab particularly in or around the transition zone where a series of phase transformations occur.

Seismological observations suggest that there is strong resistance for subduction in the transition zone globally (e.g., Isacks and Molnar, 1969), but subducted slabs deform intensively in some regions (e.g., Fukao and Obayashi, 2013). Particularly interesting observation is that deformation is extensive especially in the western Pacific where old (cold) slabs subduct (Karato et al., 2001). There have been some modeling studies to understand the nature of deformation of slabs in the transition zone (e.g., Čižková and Bina, 2013; Goes et al., 2017).

A key to understand slab deformation is its rheological properties. Karato et al. (2001) provided a model to explain the regional variation in slab deformation by invoking the temperature sensitivity of grain-size evolution after a phase transformation. However, the study by Karato et al. (2001) was based only on theoretical estimation of grain-size, and the flow law parameters were based on the scaling law because experimental studies on plastic deformation at the transition zone conditions were missing at that time.

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In the mean time, there has been major progress in the experimental studies of plastic deformation of minerals at high-pressures (e.g., Farla et al., 2015; Hustoft et al., 2013; Kawazoe et al., 2010a, 2010b; Nishihara et al., 2008) and of diffusion (e.g., Shimojuku et al., 2004). These studies provide better constraints on the flow laws of olivine, wadsleyite and ringwoodite, and also show that a substantial grain-size reduction would reduce the creep strength of these minerals.

However, the degree of grain-size change and grain-scale microstructure evolution during transformation of olivine to wadsleyite remain poorly constrained from experiments. Grain-size evolution and the spatial distribution of newly formed fine grains during the phase transformation in the mantle transition zone have potentially large influence on the strength of a slab in the transition zone.

We conducted an experimental study on the grain-scale microstructural evolution associated with the olivine to wadsleyite phase transformation for a range of Pressure (P)–Temperature (T)–time (t) paths. The key issues that we investigate include (i) the size of new grains and (ii) spatial distribution (nucleation mechanisms) of new grains. Based on the experimental observations, we develop theoretical models of grain-size evolution and apply them to deformation under the geological time scale.

2. Experimental and analytical procedures

2.1. Synthesis of starting sample

Single crystals of San Carlos olivine free from inclusions were handpicked by visual inspection. These crystals are ground to a fine powder in a ball mill and grain-size was sorted by sedimentation and sieving to ensure a starting grain-size of about 5–10 μm. Olivine powder is then cold pressed and vacuum sintered at 1573 K for 12 h to get dense (greater than 98% density) polycrystalline olivine aggregate.

2.2. Experimental procedure

High-pressure experiments were performed using a KIWI 1000-ton Kawai-type multianvil apparatus installed at Yale University. Pressure was generated using a (6–8) double stage multianvil system. Six outer anvils support the eight inner truncated cubic tungsten carbide anvils. Edge length of the inner anvil is 26 mm and the truncation edge of the inner anvil used was 5 mm. Cr_2O_3-doped MgO octahedron with 10 mm edge length is used as a pressure medium. Rhenium heater within a $LaCrO_3$ thermal insulator sleeve or a $LaCrO_3$ heater was used. Sample enclosed in a Mo foil was separated from the heater using a MgO sleeve. Temperature was measured using a $W_{95}Re_5$–$W_{74}Re_{26}$ (type C) thermocouple; no correction for the possible effect of pressure was applied. Thermocouple was placed in a 4-bore alumina tube, which was placed in MgO sleeve and axially inserted into the furnace.

We notice that temperature within the central region of the sample is substantially higher than the value measured at the thermocouple. Therefore, we map the temperature distribution in the assembly using the enstatite–diopside thermometry at 15 GPa (Gasparik, 1996). These experiments were conducted at a nominal temperature of 1873 K. Temperature variation within the sample is shown in Fig. S1. Temperature at the center of the sample is higher by 100 K than the thermocouple reading. However, temperature along a horizontal line within the sample doesn't vary more than ±30 K. In addition to the enstatite–diopside thermometry, we use 'Cell Assembly' finite element modeling code developed by Hernlund et al. (2006) to further corroborate the temperature distribution. Both the experiments and the finite element code show similar temperature variation within the sample (Fig. S1).

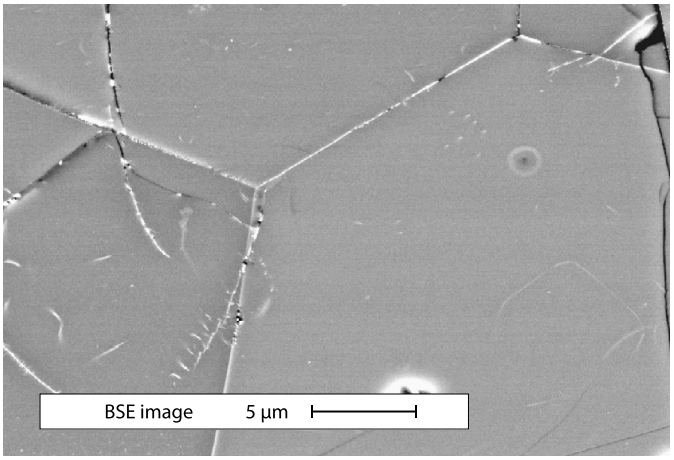

Fig. 1. Back Scatter Electron (BSE) image showing dislocation density in olivine before transformation (K1541, second stage annealing at $P = 14$ GPa, $T = 873$ K for 360 min). Bright spots and lines are dislocations; dislocation density is less than $10^{11}/m^2$. Sample was decorated following method of Karato (1987).

Experimental runs were performed in two stages. A schematic diagram of this process is shown in Fig. S2. In all the experiments, we used the same P–T–t conditions of stage 1 ($P = 11$ GPa, $T = 1773$ K, for 2 h). Stage 1 is necessary to anneal dislocations in olivine that may have been produced during compression.

We realized that the first-stage annealing and hot compression (at 873 K) has an important influence on the efficiency of pressure generation in the second stage. Therefore, we calibrated the pressure at the second stage as a function of hydraulic oil pressure using the experimental results on the olivine–wadsleyite transformation by Katsura et al. (2004) ($P = 14 \pm 0.5$ GPa at $T = 1673$ K), and for pressures higher than 14 GPa, we used the phase relations in the $Mg_4Si_4O_{12}$–$Mg_3Cr_2Si_3O_{12}$ system (Sirotkina et al., 2015).

The starting material for the experiments is a vacuum sintered San-Carlos olivine aggregate (5–10 μm grain-size). After the first stage annealing, the grain-size varied from 10 to 30 μm with a median grain-size of 25 μm. Substantial grain-growth ensures that most of the dislocations are eliminated because migrating grain boundaries consume most of the dislocations (e.g., Karato, 1988). To measure dislocation density in our sample before transformation, we decorated a sample (K1541) following the method of Karato (1987). Dislocation density of the sample after annealing is about $<10^{11}/m^2$; most of the dislocations are near grain boundaries (Fig. 1).

Initial grain-size distribution of olivine prior to transformation in all the experiments is similar. We do not modify the starting sample or the cell assembly dimensions and hence expect that this distribution of grain-size prior to transformation would remain the same for all runs. After annealing a sample at stage 1, we decrease the temperature to 873 K and then compress sample to 14–16 GPa at 873 K over a period of 150–200 min. No transformation was observed in samples when compressed to wadsleyite stability field at 873 K and annealed at 873 K and 973 K (for e.g., K1541 was annealed at second stage at 14 GPa and 873 K for 6 h and K1545 was annealed at second stage at 14 GPa and 973 K for 47 h).

After reaching the desired pressure, temperature was increased to the desired temperature rapidly (>100 K per min). This is to ensure that we have a well-defined time for the beginning of phase transformation. Samples were annealed at the various P–T–t paths in wadsleyite stability field and then the heating power supply was turned off to quench temperature; pressure was then decreased over a period of 12–14 h.