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# In situ observation of crystallographic preferred orientation of deforming olivine at high pressure and high temperature



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## ABSTRACT

Simple-shear deformation experiments on polycrystalline olivine and olivine single-crystal were conducted at pressures of 1.3-3.8 GPa and temperatures of 1223-1573 K to understand the achievement of steady-state fabric strength and the process of dynamic recrystallization. Development of crystallographic preferred orientation (CPO) of olivine was evaluated from two-dimensional X-ray diffraction patterns, and shear strain was measured from X-ray radiographs. The steady-state fabric strength of the A-type fabric was achieved within total shear strain of  $\gamma$  = 2. At strains higher than  $\gamma$  = 1, an increase in concentration of the [010] axes mainly contributes to an increase in fabric strength. At strains higher than  $\gamma$  = 2, the magnitude of  $V_{SH}/V_{SV}$  (i.e., ratio of horizontally and vertically polarized shear wave velocities) scarcely increased in most of the runs. The  $V_{SH}/V_{SV}$  of peridotite (70 vol.% olivine + 30 vol.% minor phases) having the steady-state A-type olivine fabric coincides with that of recent global one-dimensional models under the assumption of horizontal flow, suggesting that the seismic anisotropy observed in the shallow upper mantle is mostly explained by the development of A-type olivine fabric. Experimental results on the deformation of single-crystal olivine showed that the CPO of olivine is influenced by the initial orientation of the starting single crystal because strain is concentrated in the recrystallized areas and the relic of the starting single crystal remains. In the upper mantle, the old CPO of olivine developed in the past may affect the olivine CPO developed in the present.

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# 1. Introduction

Olivine is the main constituent mineral in Earth's upper mantle, and its crystallographic preferred orientation (CPO) controls the seismic anisotropy in the upper mantle. Dislocation creep is one of the dominant deformation mechanisms of olivine in the upper mantle (Karato et al., 1986), and the CPO of olivine develops through the dislocation creep-controlled flow. Recent studies have revealed that olivine fabrics can be also formed by dislocation- or diffusion-accommodated grain boundary sliding (Hansen et al., 2012; Miyazaki et al., 2014). The seismic anisotropy signatures, such as the direction of shear wave splitting and the  $V_{SH}/V_{SV}$  (i.e., ratio of horizontally and vertically polarized shear wave velocities), are affected not only by flow direction but also by the olivine CPO. It has been well investigated that the pattern of olivine CPO changes depending on dissolved water content, differential stress, temperature, and pressure (Jung and Karato, 2001; Katayama et al., 2004; Ohuchi et al., 2011). Change of the dominant deformation mechanism and partial melting of peridotites may also affect the pattern of olivine CPO (Holtzman et al., 2003; Karato, 1992). In the case of dislocation creep-controlled flow, development of

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olivine CPO is controlled by the easiest slip system (e.g., Jung et al., 2006). Many experimental studies have verified that A-type fabric (developed by the (010)[100] slip system), B-type (by the (010)[001] slip system), C-type (by the (100)[001] slip system), and E-type fabrics (by the (001)[100] slip system) are formed under the upper mantle conditions.

Dynamic recrystallization inevitably occurs through dislocation-creep-controlled creep of olivine, and thus, the fabric strength reaches to a steady state value with increase in strain due to the weakening of fabric strength caused by dynamic recrystallization (Lee et al., 2002). Because the relationship between fabric strength and seismic anisotropy shows an exponential form (Ismail and Mainprice, 1998), seismic anisotropy in the asthenospheric upper mantle is expected to have an upperlimit value. To evaluate the fabric strength quantitatively, the J-index (Bunge, 1982: Mainprice and Silver, 1993) and M-index (Skemer and Karato, 2008) have been used. Recently, the strain dependency on fabric evolution of olivine has been evaluated from microstructures of the quenched samples deformed at 0.3 GPa (Hansen et al., 2012, 2014) and 1 GPa (Boneh and Skemer, 2014). Hansen et al. (2012, 2014) demonstrated that a steady-state fabric is not reached until a very large shear strain ( $\gamma > 10$ ) and fabric strength of olivine increases up to the J-index of 10-30 (M-index of 0.4-0.7). Wide ranges of values of the J-index and M-index of natural peridotites that experienced deformation have been reported as 3-15 and 0.03-0.34, respectively (Ismail and Mainprice, 1998; Muramoto et al., 2011), implying that olivine CPOs in some of natural peridotites did not reached to steady state. Evolution of CPO in deforming olivine polycrystals has been simulated using various types of approaches (see a review by Tommasi et al., 2000). Tommasi et al. (2000) simulated the development of olivine CPO using an anisotropic viscoplastic self-consistent model and showed that seismic anisotropy almost reaches a steady-state value at shear strain  $\gamma \sim 8$ . In their calculation, however, the effect of dynamic recrystallization on the development of olivine CPO was not considered. Kaminski et al. (2004) showed that dynamic recrystallization prevents the formation of a singular CPO even at high strains (in their model, no rotation of grains and grain boundary sliding-controlled flow were assumed for recrystallized grains).

Even though fabric strength correlates with the magnitude of seismic anisotropy (e.g., Ismail and Mainprice, 1998), the strain-dependency of the fabric strength of olivine has not yet been fully investigated at asthenospheric upper mantle pressures (2-13 GPa). It has been reported that the relative activity of each slip system in olivine changes depending on pressure due to the difference of activation volume among the slip systems (Raterron et al., 2007, 2012). Ohuchi et al. (2011) showed that the fabric strength of A-type olivine decreases with increase in pressure, and they discussed that the weakening of fabric strength was caused by a decrease of the strength contrast between the (010)[100] and the (010)[001] slip systems with pressure increase. Therefore, the strain dependency on the fabric strength of olivine needs to be evaluated under the upper mantle conditions. One of the most effective methods used to evaluate the evolution of CPO in a deforming sample is "in-situ" observation of two-dimensional diffraction patterns. Orientation contrast in a two-dimensional diffraction pattern represents the CPO development. Wenk et al. (2004) performed "in-situ" measurements of CPOs of minerals at high pressures using a technique combined with two-dimensional diffraction patterns and the Rietveld method. Recently, similar techniques have been applied to uniaxial deformation experiments of forsterite under upper mantle conditions (Bollinger et al., 2012). However, flow direction is not unique in the uniaxial deformation geometry and thus the development of olivine CPO needs to be evaluated in the simple-shear geometry. In this study, therefore, we experimentally evaluate the strain dependency of fabric strength of olivine in simple-shear geometry under upper mantle conditions through "in-situ" observations of two-dimensional diffraction patterns.

## 2. Experimental procedure

#### 2.1. Starting materials

The starting materials for deformation experiments were prepared from a sample 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 and porosity of the hot-pressed sample were 15.3 µm and 0.1 vol.%, respectively. The water content dissolved in the hot-pressed sample was 4015 ppm H/Si. Oriented single crystals of olivine (Fo<sub>90</sub>) from San Carlos was used as a starting material of the M1373 and M1441 runs. The hot-pressed sample and the single crystal olivine were core-drilled with a diameter of 1.5 mm, and then sectioned to have a thickness between 300 and 450 µm. In order to remove water dissolved in the olivine samples, all of the sectioned parts of the sintered sample and the olivine single crystal sample were fired at 0.1 MPa and 1170 K under reducing conditions ( $f_{02} \sim 10^{-16}$  bars) for 6 h. Water content in the fired samples is less than 40 ppm H/Si.

#### 2.2. Deformation experiments and "in-situ" observations

We conducted simple-shear deformation experiments on olivine samples at pressures of 1.3-3.8 GPa, temperatures of 1223-1573 K. and strain rates of  $9.7 \times 10^{-5} - 7.5 \times 10^{-4} \text{ s}^{-1}$  using a deformation-DIA apparatus at the BL04B1 beamline of the SPring-8 (M-series runs in Table 1) or at the NE7A beamline of the Photon Factory (TO-series). Two of the four sliding blocks on the down-stream side have a conical X-ray path (maximum  $2\theta$ angle  $\sim 10^{\circ}$ ). The MA-6-6 system, which consists of six second-stage anvils with truncated edge lengths of 5 mm, an anvil guide, and cell assembly, was adopted for the experiments (Nishiyama et al., 2008). Two X-ray transparent anvils, which were made from cubic boron nitride (cBN), were used for the second-stage anvils on the lower-stream side. The anvil guide was made of engineering plastic (columns along X-ray path) and stainless steel (other parts) (Kawazoe et al., 2011). A sketch of the cell assembly used for the deformation experiments is shown in Fig. 1. The design of the cell assembly was based on Ohuchi et al. (2010, 2012). A semi-sintered cobalt-doped magnesia ((Mg, Co)O) cube with an edge length of 7 mm was used as the pressure medium. A graphite heater was located at the inner bore of a tubular LaCrO<sub>3</sub> thermal insulator. Copper and molybdenum electrodes, hard alumina pistons, and machinable alumina rods were placed along the direction of the uniaxial compression. Two X-ray transparent rods, which were made from magnesia (MgO), were placed along the X-ray path in the pressure medium. A sectioned olivine sample was placed into a nickel capsule and then sandwiched between two alumina pistons that were coated with platinum (note that the platinum layer with  $\sim 1 \,\mu m$  thickness prevents the chemical reaction between olivine and alumina pistons). Occurrence of iron-loss from olivine was limited to olivine grains which were next to the platinum layer, the platinum strain marker, and the nickel capsule (e.g., Ohuchi and Irifune, 2013). The nickel capsule was surrounded by a hexagonal boron nitride (hBN) sleeve. Download English Version:

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