



# New constraints on upper mantle creep mechanism inferred from silicon grain-boundary diffusion rates



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## ABSTRACT

The creep in the Earth's interior is dominated either by diffusion creep which causes Newtonian mantle flow, or by dislocation creep which results in non-Newtonian mantle flow. Although previous deformation studies on olivine claimed a transition from dislocation creep to diffusion creep with depth in the upper mantle, they might misunderstand the creep rates due to experimental difficulties. Since creep in olivine is controlled by silicon diffusion, we measured the silicon grain-boundary diffusion coefficient in well-sintered iron-free olivine aggregates as a function of temperature, pressure, and water content, showing activation energy, activation volume, and water content exponent of  $220 \pm 30$  kJ/mol,  $4.0 \pm 0.7$  cm<sup>3</sup>/mol, and  $0.26 \pm 0.07$ , respectively. Our results based on Si diffusion in forsterite predict that diffusion creep dominates at low pressures and low temperatures, whereas dislocation creep dominates under high pressure and high temperature conditions. Water has negligible effects on both diffusion and dislocation creep. There is a transition from diffusion creep in the shallow upper mantle to dislocation creep in deeper regions. This explains the seismic anisotropy increases at the Gutenberg discontinuity beneath oceans and at the mid-lithosphere discontinuity beneath continents.

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## 1. Introduction

Diffusion and dislocation creep are considered to be the two main mechanisms of plastic deformation in the Earth's interior. An important question in geodynamics at present is which creep mechanism dominates in the upper mantle. If it is dislocation creep, the strain rate will be proportional to stress to the power of 3.0–3.5, leading to non-Newtonian viscosity. The dominant slip system in dislocation creep will produce lattice-preferred orientation in olivine, which causes seismic anisotropy. In contrast, if diffusion creep dominates, both lattice-preferred orientation as demonstrated by a recent study (Miyazaki et al., 2013), and shape-preferred orientation, will be produced in olivine and strain rate proportional to stress will result in Newtonian viscosity.

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The large water and pressure dependences of olivine creep rates suggested by experimental deformation studies indicate that the dominant creep mechanisms in the upper mantle transition from dislocation creep in the shallow lithosphere – to diffusion creep in the asthenosphere with a transition depth of 200–250 km (Hirth and Kohlstedt, 2003; Karato and Wu, 1993). However, based on silicon and oxygen diffusion experiments, Fei et al. (2012, 2013, 2014) suggest that the aforementioned deformation studies have overestimated the water and pressure effects on creep rates owing to experimental limitations. Demouchy et al. (2012) also suggest that the creep rates in deformation experiments on aggregate samples might be largely affected by grain boundary water. Furthermore, dislocation creep in olivine at high pressures dominated by [001] slip produces little lattice-preferred orientation compared with that at low pressures dominated by [100] slip, indicating that it is not essential to invoke diffusion creep in explaining the reduction in seismic anisotropy noted in the region deeper than 250 km during seismic observations (Mainprice et al., 2005). All of this experimental evidence casts doubt on the theory that there is a transition from dislocation creep in the shallow lithosphere to diffusion

**Table 1**  
Concentration of trace elements measured by X-ray fluorescence (XRF).

Major (wt.%)		Minor (wt.ppm)	
SiO <sub>2</sub>	43.56	V	4.995
MgO	56.46	Cr	5.133
Al <sub>2</sub> O <sub>3</sub>	0.010	Co	0.999
FeO	0.026	Ni	6.130
MnO	0.001	Cu	13.251
TiO <sub>2</sub>	0.008	Zn	16.861
Na <sub>2</sub> O	0.088	Ga	0.945
K <sub>2</sub> O	0.001	Rb	1.439
P <sub>2</sub> O <sub>5</sub>	0.005	Sr	3.497
Total	100.16	Zr	5.843
		Pb	2.291

creep in the asthenosphere. It is therefore necessary to systematically re-examine the creep mechanisms in the upper mantle.

Both diffusion creep (includes Coble creep and Nabarro–Herring creep) and dislocation creep are believed to be driven by diffusion of the slowest element, i.e., Si in silicate minerals, on grain boundaries (for Coble creep) and within grain interior (for Nabarro–Herring and dislocation creep) (Frost and Ashby, 1982; Weertman, 1999). Thus, the silicon grain-boundary and lattice diffusion coefficients ( $D_{\text{Si}}^{\text{gb}}$  and  $D_{\text{Si}}^{\text{lat}}$ , respectively) are essential for simulation of dislocation and diffusion creep. We have already measured  $D_{\text{Si}}^{\text{lat}}$  in forsterite for a wide range of pressure ( $P$ ), temperature ( $T$ ), and water content ( $C_{\text{H}_2\text{O}}$ ) conditions by our advanced technique (Fei et al., 2012, 2013). By applying this procedure to grain-boundary diffusion,  $D_{\text{Si}}^{\text{gb}}$  can be obtained for a much wider range of  $P$ – $T$ – $C_{\text{H}_2\text{O}}$  conditions than is possible through deformation experiments. Thus, in this study, we systematically measured  $D_{\text{Si}}^{\text{gb}}$  in forsterite aggregates as a function of pressure, temperature, and water content, and assert that Coble creep rate in the upper mantle is estimated.

## 2. Experimental and analytical procedure

### 2.1. Starting material

Forsterite (Mg<sub>2</sub>SiO<sub>4</sub>) aggregates were synthesized from a mixture of SiO<sub>2</sub> and Mg(OH)<sub>2</sub> (with particle sizes of 30 nm and 50 nm, respectively). The SiO<sub>2</sub> and Mg(OH)<sub>2</sub> mixture was analyzed via thermogravimetry and differential thermal analyses to 1273 K to estimate the water content on the powder surfaces, which was extremely high (~20 wt.%) owing to the large surface area per unit volume. The two powders were well mixed, calcined at 1273 K to synthesize forsterite fine powder, then formed into pellets (~5 mm in diameter and ~4 mm thick) at 200 MPa in a cold isostatic press. The pellets were sintered at 1630 K for about 3 hrs to sinter the aggregates in a vacuum furnace (~1 × 10<sup>-3</sup> Pa) at The University of Tokyo (Koizumi et al., 2010).

Fine grained aggregates with average grain size of ~0.6 μm and homogeneous grain-size distributions confirmed by scanning electron microscopy (SEM) images (Fig. 1A) were obtained. The chemical composition of calcined powders was measured by x-ray fluorescence (XRF, ZSX PrimusII RIGAKU) at the University of Tokyo as shown in Table 1. The  $C_{\text{H}_2\text{O}}^{\text{bulk}}$  was <1 wt.ppm determined by Fourier transform infrared (FT-IR). Forsterite aggregates (~0.7 × 0.7 × 0.6 mm<sup>3</sup>) cored from the synthesized pellets were used for the diffusion experiments in this study. Several pieces of aggregates were further heated at 1700 K for 20 hrs to produce average grain size of ~2 μm.

### 2.2. Pre-annealing experiments

To obtain samples with various  $C_{\text{H}_2\text{O}}^{\text{bulk}}$ , aggregates were pre-annealed at 8 GPa, 1200–1600 K with talc and brucite (with a

weight ratio of 4:1) as the water source. Each sample was loaded into a one-side-sealed Pt capsule with the talc/brucite mixture at the bottom (Fei et al., 2013). The space between the sample and capsule wall was filled with graphite and enstatite (MgSiO<sub>3</sub>) powders (~20:1 volume ratio) to prevent mechanical damage to the sample and to buffer the silicon activity, respectively. The Pt capsules were sealed by arc welding in liquid nitrogen. The bulk water in the capsule was controlled by the ratio of talc + brucite:graphite + enstatite (Fei et al., 2013), as well as the oxygen fugacity ( $f_{\text{O}_2}$ ) at the graphite stable field, which is about one log unit below the fayalite–magnetite–quartz (FMQ) buffer (Stagno et al., 2013).

High-pressure experiments were performed using the Kawai-type multi-anvil apparatuses at the University of Bayreuth and Okayama University. For each experimental run, the Pt capsule was placed in an MgO cylinder in a stepped graphite heater with a ZrO<sub>2</sub> thermal insulator. The temperature was measured by W97%Re3%–W75%Re25% thermocouple (Fig. 2). The pressure media were MgO (with 5 wt.% Cr<sub>2</sub>O<sub>3</sub>) octahedrons with edge lengths of 14 mm. High pressure was generated by eight tungsten carbide cubes with edge lengths of 32 mm and truncation edge lengths of 8 mm. Each assembly was compressed to 8 GPa at room temperature, heated to the target temperature (1200–1600 K, same temperature condition corresponding to the further diffusion annealing experiment for each sample) at a rate of 50 K/min, kept at the target temperature for 4–10 hrs which was sufficient to equilibrate the water in the sample (Demouchy, 2010), quenched by switching off the heating power, then decompressed to ambient pressure.

For dry condition experiments, samples were pre-annealed at 1–13 GPa and 1200–1600 K for defect equilibrium following the same method described above, but without the water source. Additionally, the capsules with samples and graphite/enstatite powders were dried in a vacuum furnace ( $P < 30$  mbar) at 473 K for >24 hrs and sealed on a hotplate to minimize moisture (Fei et al., 2012).

For ambient pressure experiments, samples with the graphite/enstatite buffers were loaded in Pt capsules without sealing and annealed in a gas mixing furnace (H<sub>2</sub>/CO<sub>2</sub>) at 1200–1600 K for ~12 hrs. The  $f_{\text{O}_2}$  was controlled at near  $\log f_{\text{O}_2} = (\text{FMQ}) - 1$ .

### 2.3. Deposition and diffusion annealing

After pre-annealing, the samples were finely polished using 0.25 μm diamond powder and an alkaline colloidal silica solution. The roughness of the sample surfaces after polishing was less than 10 nm, including the grain boundaries, as confirmed by a 3D-confocal microscope at the University of Bayreuth. The samples were heated up to 420 K for 10–15 min in the vacuum chamber of the pulsed laser deposition system at Ruhr-University of Bochum to remove any surface water absorbed during polish, and deposited with a 600–1000 nm thick <sup>29</sup>Si-enriched Mg<sub>2</sub>SiO<sub>4</sub>-forsterite film. An additional thin ZrO<sub>2</sub> film (~100-nm thick) was deposited to protect the <sup>29</sup>Si enriched forsterite film; we have confirmed in previous studies that this does not affect the silicon diffusion rate (Fei et al., 2012, 2013).

Each thin-film coated sample was annealed once again for diffusion using exactly the same experimental set up,  $P$ – $T$  condition, and chemical environment, and the same talc + brucite and graphite + enstatite ratio used in the corresponding pre-annealing experiment. No free water could present near the sample during annealing because the water source and forsterite aggregates were separated by graphite, which makes unsaturated condition. The  $C_{\text{H}_2\text{O}}^{\text{bulk}}$  in the samples did not change during diffusion (FT-IR analysis section). That is because the  $C_{\text{H}_2\text{O}}^{\text{bulk}}$  in the sample is equilibrated

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