Flow behaviors and AE activities during syndeformational antigorite dehydration at high pressures

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Intermediate-depth earthquakes are seismic activities in Wadati-Benioff zone at depths from 60 km to 300 km, where subducting plates deform plastically rather than brittle failure. To understand the shear instability mechanisms above \(\sim 2\) GPa, there have been some acoustic emission (AE) measurements with multi-anvil apparatus (e.g., Jung et al., 2009; Gasc et al., 2011). However in previous studies, the relationships among dehydration, plastic flow and shear instability were unclear because quantitative flow data, reaction kinetics, and AE activities could not be obtained simultaneously. To conduct quantitative and simultaneous measurements of these processes, we have carried out syndeformational antigorite dehydration experiments at high pressure.

High-pressure deformation experiments were conducted at 1-8 GPa, 300-1050 K, and strain rates of 3.4-7.4 \(\times 10^{-5}\) s\(^{-1}\) in pure shear using a 1500-ton uniaxial press (SPEED Mk. II) with a D-DIA type guide block installed at BL-04B1, SPring-8. 50 keV monochromatic X-ray were used to measure reaction kinetics and stress-strain data. To monitor shear instabilities by detecting AEs, six piezoelectric devices were positioned between first and second stage anvils of MA 6-6 type system. AE waveforms were recorded in trigger mode using six-channel 8-bit digital oscilloscopes at a sampling rate of 50 MHz, and were analyzed to determine 3D AE source location and AE magnitude. We used three kinds of starting materials (1.7 mm in diameter and 2.7 mm in length) of polycrystalline antigorite (Atg), fine-grained forsterite (Fo) polycrystal, and two-phase mixtures of Atg and San Carlos olivine (10%, 30%, and 50%Atg). The starting sample was first compressed at room temperature, then heated at constant load, and finally deformed with constant strain-rate mode. In some experiments, dehydration occurred during heating or deformation. Microstructures of recovered samples were preliminarily observed by optical microscopy.

Many AE events of relatively large amplitude were observed from Fo sample during cold compression stage at lower than 2 GPa. In contrast, almost no AEs were observed from Atg and 10-50% Atg samples during cold compression and heating stages. Optical microscopic observations of recovered Atg samples from each stage revealed that some faults are generated during cold compression stage, and fault slipping occurred during the heating stage. Creep behaviors of Atg samples during the deformation stage indicate that flow stress reached steady state at the sample strain of more than 10%, and no stress drops were observed until the final strain of 30-40%. These flow behaviors and flow strengths are almost similar to the previous study (Hilairet et al., 2007). Few AEs were recorded and additional faults were not observed from recovered sample during the deformation stage. We observed dehydration of Atg and 10-50% Atg samples when increasing temperature from 673K to 1000K during deformation, however we did not observe AEs during the syndeformational dehydration stage. Faults were absent in fully dehydrated samples. Although more detailed microstructural observations are needed, these results suggest that syndeformational dehydration do not enhance shear instability of antigorite in pure shear at high pressure.
Experimental study on polycrystal anelasticity with implications for upper mantle seismic structure

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Rock anelasticity causes dispersion and attenuation of seismic waves. Therefore, for the quantitative interpretation of seismic low velocity and/or low Q regions in the upper mantle, understanding of rock anelasticity is necessary. Recent experimental studies have shown that anelasticity of polycrystalline materials is subject to the Maxwell frequency $f_M$ scaling: $Q^{-1}(f/f_M)$. However, the applicability of this scaling to the seismic waves has not been guaranteed because experimental frequencies normalized to $f_M$ of the laboratory samples are usually much lower than the seismic frequencies normalized to $f_M$ in the upper mantle ($10^6 \leq f/f_M \leq 10^9$). In this study, by using polycrystalline organic borneol as an analogue to mantle rock, we measured anelasticity up to $f/f_M \sim 10^8$ and found that the Maxwell frequency scaling is not fully applicable at $f/f_M > 10^4$. A closer examination of our data showed that each of the relaxation spectra obtained under various temperature, grain size, and chemical composition can be represented by the superposition of a background dissipation which is subject to the Maxwell frequency scaling and a peak dissipation which is always centered at $f/f_M = 10^3$. Significant increases of the peak amplitude and width with increasing temperature, grain size, and impurity (dyphenylamine) content result in failure of the Maxwell frequency scaling at $f/f_M > 10^4$, where the peak dissipation dominates over the background dissipation. To quantitatively estimate the dispersion and attenuation of seismic waves, it is important to understand the behavior of the peak dissipation.

The addition of impurity (diphenylamine) to borneol significantly reduces the melting (solidus) temperature from $T_{melt} = 477$ K to $T_{melt} = 316$ K. Therefore, we have speculated that the observed variation of the peak dissipation with impurity and temperature can be scaled by the normalized temperature $T/T_{melt}$, such that the peak amplitude and width increase with increasing $T/T_{melt}$. The significant broadening of the peak observed near (but below) the solidus temperature ($T/T_{melt} = 0.93$) means that seismic velocity and Q are considerably lowered even without melt and has important implications for upper mantle seismic structure. We further investigated the detailed behavior of the peak dissipation at near solidus temperatures ($0.88 \leq T/T_{melt} \leq 1.01$), and found that the peak amplitude saturates at about $T/T_{melt} = 0.95$, but that the peak width continuously increases up to the supersolidus temperature $T/T_{melt} = 1.01$. The obtained result was formulated in terms of the two nondimensional parameters $f/f_M$ and $T/T_{melt}$ and preliminarily applied to the seismic waves in the upper mantle. The result shows that low V and low Q occur at near solidus temperatures even without melt. At the onset of melting, seismic wave velocity shows a discrete reduction due to the poroelastic effect of melt, but the seismic attenuation does not show a discontinuous change.

Keywords: anelasticity, seismic attenuation, upper mantle
Rheological transition during large strain deformation of melting and crystallizing metapelites

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Torsion experiments (strain-rate = $3 \times 10^{-4}$s$^{-1}$) were performed to investigate the large strain ($\gamma \approx 15$) rheology on quartz-muscovite aggregate as analogue to pelitic rocks undergoing melting and crystallization during deformation at 300 MPa confining pressure and 750°C temperature. Microstructures reveal four distinct but gradational stages of crystal-melt interactions during deformation: (a) solid state deformation, (b) initiation and domination of partial melting, (c) simultaneous partial melting and crystallization, and (d) domination of crystallization. The microstructural stages are linked to the rheology of the deforming samples. Partial melting starts at relatively low finite shear strains ($\gamma = 1$ to 3) showing approximately 60% strain softening. At $\gamma = 4$ to 10 the partially molten bulk material shows a steady state flow at low stress. Further crystallization of new crystals at the expense of melt between $\gamma = 10$ and 15 causes weak strain hardening until the material fails by developing brittle fractures. The stress exponent ($n$), calculated at $\gamma = 1, 5, \text{ and } 10$, increases from $\sim 3$ to $\sim 43$, indicating a transition from power to power law breakdown or exponential flow of the bulk system. Hydrostatic experiments for equivalent times and conditions of the torsion experiments were also conducted to evaluate the reaction kinetics and microstructures under static conditions. The new experimental data establish that partially molten rock does not flow according to a constant strain rate-dependent power law (steady state) rheology. The rheological transition from strain rate sensitive to strain rate insensitive flow is interpreted as a function of melt-crystal ratio, their mutual interactions, and the evolution of microstructures in the partially molten rock. EBSD measurements reveal weak crystal preferred orientations, which are different for each mineral.

Keywords: Rheology, Deformation, Melting, Crystallization, Crystal preferred orientation, Localization
The uppermost mantle structure in the oceanic plate induced by mantle diapir

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This study presents results of microstructural analyses and crystallographic preferred orientations (CPOs) in order to clarify characteristics of harzburgite in the Samail and Hilti mantle sections. The Samail peridotites of seven samples were collected in the Maqsad area across the mantle diapir. The Hilti peridotites of thirteen samples were collected in the zone of the high-T deformation near the paleo-Moho in the mantle section. Thus, we studied twenty samples in Oman ophiolite. Olivine CPOs in the Samail samples are characterized by slightly strong point maximum of [010] and weak girdle distributions of [100] and [001]. Olivine CPOs in the Hilti samples are characterized by two types of the distributions: one type shows slightly strong point maximum of [010] and weak girdle distributions of [100] and [001], and the other type is slightly strong point maximum of [010] and weak point distributions of [100] and [001]. The olivine fabrics have been used to be represented by fabric intencities of each axis such as pfJ so far. However, pfJ does not necessarily reflect olivine fabrics of their whole axes and it strongly depends on a stereographic distribution. Moreover, AG-type has various patterns of distributions in both a-axis and c-axis, so that it is difficult to classify AG-type by any previous methods. In this study, we prefer to classify olivine CPO patterns using Vp-Flinn diagram. It shows that both Samail and Hilti samples are almost identical each other and in the VP-Flinn diagram that olivine fabrics could be classified into three types: 1) A-type like fabric, 2) AG-type like fabric and 3) low Vp anisotropy AG-type like fabric. Grain boundaries have different characteristics between them; the Samail samples showed regular grain boundaries, whereas the Hilti samples showed irregular grain boundaries. It is likely that the temperature in the Oman ophiolite would have been the highest in the mantle diapir beneath the ridge axis and therefore its solidus line could be deeper as moving away from the ridge axis. Therefore, the difference of grain boundary geometries may result from a temperature change in the vicinity of the mantle diapir from vertical flow to subhorizontal flow. The olivine grain sizes are almost constant regardless of their CPO patterns and are in the same range between 0.6 and 2.1 mm in both the Samail and the Hilti samples. It shows that the Hilti samples may have experienced only a small horizontal strain that is not enough to change olivine CPOs except a weak grain size reduction due to dynamic recrystallization. As a consequence, the olivine CPOs could have been formed in the mantle diapir and preserved even in the horizontal structure away from the mantle diapir. The olivine fabric could have been formed by melt effects like MORB in the mantle diapir or diffusion creep.

Keywords: olivine fabrics, lithosphere, uppermost mantle, mantle diapir
Supply rate of continental materials by subduction of island arcs

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Evolution of life on the Earth is strongly related to the oceans and the continents, both of which are unique to the Earth. Continental materials contain a large amount of incompatible and radiogenic elements, which may affect terrestrial thermal history and chemical evolution, as well as the Earth’s surface environment. Geological studies have revealed that continental materials subduct from the Earth’s surface via the following three mechanisms: tectonic erosion, sediment subduction, and direct subduction of immature oceanic arcs, which are found, for example, in the western Pacific. In the first two processes, the continental materials are conveyed through subduction channels of thickness of 2-3km just above the subducting slabs. Here, in order to estimate the supply rate of continental materials of oceanic arcs to the deep mantle, we have conducted numerical simulations of subduction of arcs based on the finite element method, using relevant rheology models. The results show that the subduction rate highly depends on temperature profiles of the subducting slabs and the size of the arcs.

Keywords: continental crust, island arc, mantle, subduction, rheology
Anisotropic viscosity of olivine aggregates: A laboratory, field, and numerical approach

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Shearing of mantle rocks within Earth’s lithosphere and asthenosphere causes mantle minerals, particularly olivine, to develop crystallographic textures that can be detected seismically. In laboratory studies, such crystallographic textures have also been associated with dependence of the viscosity of a mineral on the orientation of an applied stress. This anisotropic viscosity may affect tectonic plate motions and the stability of the lithospheric base, but it is highly dependent on 1) the rate of fabric development and 2) the poorly constrained viscosity tensor, which relates the stress applied to an olivine crystal of known orientation to the resulting strain-rate.

Here we constrain the importance of viscous anisotropy in the upper mantle using deformation experiments conducted in a gas-medium apparatus. Olivine aggregates were deformed at a temperature of 1200 °C and a confining pressure of 300 MPa. One set of samples was initially deformed in torsion and subsequently deformed in tension. A second set of samples was initially deformed in tension and subsequently deformed in torsion. Torsion experiments reached a maximum shear strain of ~20. This combination of strain paths allowed us to quantify the effect of evolving crystallographic texture on multiple components of the viscosity tensor.

We developed a micromechanical model that allows estimation of the complete viscosity tensor based on a measured texture and a single-crystal viscosity tensor. We then used the laboratory-derived mechanical data in conjunction with this model to invert for the values of the single-crystal viscosity tensor. We developed a second, independent model of olivine textural development that takes into account the strength of individual slip systems yet is stable to high strains. We calibrate this texture model through comparison to the strengths and shapes of measured textures in experimental samples. Together, these two calibrated models allow us to constrain both the rate of texture development in an arbitrary deformation geometry and also the resulting macroscopic viscosity tensor. Our results indicate that olivine textural development can yield viscosities that vary by over an order of magnitude depending on the orientation of the applied stress relative to the dominant crystallographic texture.

We tested our mechanical and textural evolution model through comparison to natural peridotite shear zones exposed in the Josephine Peridotite. We find that (1) the natural textural development is well approximated by our numerical model and (2) a significant portion of the strain localization can be attributed to the development of viscous anisotropy.

Keywords: rheology, olivine, crystallographic preferred orientation, viscosity, rock deformation, shear zones
Evolution of the rheological and microstructural properties of olivine aggregates during dislocation creep under hydrous conditions

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Since hydrogen plays an important role in dynamic processes in the mantle, we conducted high-strain torsion experiments on Fe-bearing olivine aggregates under hydrous conditions. Most of the samples deformed homogeneously to total shear strains of up to $\gamma \approx 5$. We deformed samples to high enough strain that a steady-state microstructure was achieved, in order to investigate the evolution of the rheological and microstructural properties. The stress exponent of $n \approx 3$ and the grain size exponent of $p \approx 0$ determined from the fitting of strain rate, stress, and grain size data indicates that these samples deformed by dislocation creep. Fourier transform infrared (FTIR) measurements of an imbedded olivine single crystal demonstrated that our samples were saturated with hydrogen during the deformation experiments. The crystallographic preferred orientation (CPO) of the olivine aggregates changed as a function of strain due to competition among the three slip systems (010)[100], (100)[001], and (001)[100]. The observed strain weakening that occurs early in each experiment is to geometrical softening associated with development of a CPO, which reduces the stress by 36% in constant strain rate experiments. The evolution of the rheological and microstructural properties observed in our experiments is important for understanding dynamic evolution of Earth’s mantle under hydrous conditions.

Keywords: olivine, deformation, high-strain torsion experiment, hydrous conditions, mantle
Synthesis and deformation experiments on polycrystalline olivine

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It is very important to examine the flow properties of the upper mantle for understanding the mantle flow and the origin of asthenosphere. Previous studies on creep properties of polycrystalline olivine prepared from naturally derived olivine have shown the effects of temperature, grain size, stress, and the amount of water and melt, which help to construct an applicable flow law to the natural condition (Karato et al 1986, Hirth and Kohlstedt 1995, Hirth and Kohlstedt 2003). However, Faul and Jackson (2007) showed that olivine aggregates prepared from reagents using sol-gel technique had 1~2 orders of magnitude larger strength compared to the naturally derived olivine aggregates indicating a presence of significant but unknown factor that controls creep rate of olivine aggregate. In this study, we synthesized olivine aggregates by using new technique and conducted high-temperature creep experiment on the synthesized olivine aggregates. Also we introduced small amount of impurities on such aggregates to investigate the effect of impurities on the creep properties of olivine aggregates.

The aggregates were prepared by applying vacuum sintering to nano-sized olivine powder synthesized from highly pure and fine-grained (<100 nm) raw powders (Koizumi et al 2010). An appropriate source of Fe and a method to achieve reductive atmosphere were searched. Olivine aggregates with and without dopants of 0.1% Al$_2$O$_3$, CaO, NiO, TiO$_2$ were prepared. Deformation tests on these samples showed no major difference in their strength under diffusion creep. Further, the strength was essentially identical to the aggregates by Faul and Jackson (2007). Our results indicate that the some chemical species other than those investigated in this study has a significant effect on creep properties of polycrystalline olivine.

Keywords: Synthesis of polycrystalline olivine, High-temperature deformation experiments
In situ deformation of eclogite at high pressure and temperature

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Little is known about the physical behaviour of heterogeneous, rheological contrasting rocks in the Earth’s mantle and their impact on mantle processes. Understanding of the stress/strain partitioning, microstructural evolution, and seismic signatures in bimineralic eclogites provide crucial information for transport modeling of these dense materials into and out of the deep Earth. For example, heterogeneity in the peridotite-dominated mantle may develop via a variety of processes such as entrainment and mixing of discrete eclogite bodies from subducted slabs or from detachment of deep continental roots into the convecting mantle. Exhumed eclogitic bodies are characterized by intense foliation and strain localization and strong shape- and crystallographic preferred orientations. In this preliminary study we used synchrotron X-ray diffraction at the ID06 beamline at ESRF to estimate the stresses \textit{in situ} on a deforming bimineralic eclogite (∼40% garnet and ∼60% clinopyroxene) and a dunite (100% olivine) at 5 GPa and a range of temperatures (1473 - 1673 K), i.e. at subsolidus conditions. The starting material was prepared as a residual eclogite after extraction of ∼46 wt% melt from GA2 eclogite (Spandler et al., 2008, \textit{J Petrology} 49, 771-795) and was pre-sintered at 5 GPa and 1673 K using a multi anvil apparatus. The run products yield well-equilibrated sub-/euhedral crystals of garnet and tabular crystals of clinopyroxene with average grain sizes of <30 µm. Coesite was present only as multiple small, rounded inclusions in garnet. The compressive deformation experiments were carried out at constant strain rates of $10^{-6}$ to $10^{-5}$ s$^{-1}$ to axial strains of 5 to 30% on a stack comprising the pre-sintered bimineralic eclogite sample above a pure San Carlos olivine sample (as a reference). The cylindrical samples have dimensions of 1.2 mm in diameter by 1.5 mm in length and are separated by metal foils (e.g. Re) acting as strain markers visible in radiographic images. The deviatoric stresses are transmitted via hard alumina pistons above and below the sample stack. Furthermore, the samples are surrounded by a boron nitride sleeve and graphite resistance heater and the cubic pressure medium is made of boron epoxy, which is transparent to synchrotron X-rays. We expect results on the strength partitioning between garnet and pyroxene in a residual bimineralic eclogite and between the eclogite and the dunite deformed under conditions applicable to the upper mantle. Complementary information on crystallographic preferred orientation development of garnet and clinopyroxene in eclogite, and of olivine in dunite will give insight into the deformation mechanisms and interpretation of fast seismic wave propagation directions and anisotropy relative to the prevalent stress direction. The data will be compared against natural field and seismic observations and offer insight into deep crustal seismic reflectors seen in regions of intense deformation.

Keywords: two-phase deformation, eclogite rheology, in situ X-ray diffraction, mantle heterogeneity
Grain boundary sliding as the major deformation mechanism of olivine in Earth’s upper mantle

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Anisotropic propagation of seismic waves have been observed in the Earth’s upper mantle, which is attributed to the crystallographic preferred orientation of olivine associated with mantle dynamics. The most likely deformation mechanism of olivine that explains the observed seismic anisotropy has been considered to be dislocation creep (e.g., Karato et al., 1986). Diffusion creep may also partly contribute to the deformation of olivine in the upper mantle, while another possible mechanism, grain boundary sliding, has been thought to play only minor roles in some limited circumstances such as in melt-bearing rocks or fine-grained rocks (Hirth and Kohlstedt, 1995; Hiraga et al., 2010). Hansen et al. (2011) proposed that the dislocation-accommodated grain boundary sliding (DisGBS) may dominate the upper mantle flow, based on deformation experiments for olivine at a pressure of 0.3 GPa and temperatures to 1523 K. Development of crystallographic preferred orientation (CPO) of olivine through DisGBS-controlled creep is so significant (Hansen et al., 2012) that this would be a potentially important deformation mechanism controlling the upper mantle flow. Nevertheless, most of experimental investigations on the deformation mechanism of olivine have been limited to the pressures below 0.5 GPa, which are lower than those in the actual upper mantle.

We conducted uniaxial deformation experiments on olivine aggregates with a composition of Mg$_{1.8}$Fe$_{0.2}$SiO$_4$ at pressures 1.5-6.7 GPa and at temperatures 1273-1473 K with strain rates of 0.3-7.2×10^{-5} s$^{-1}$ using a deformation-DIA apparatus. The averaged values of stress exponent, activation energy, and activation volume were obtained to be 3.0, 423 kJ/mol, and 17.7 cm$^3$/mol, respectively. The obtained parameters supports the deformation of water-poor olivine controlled by DisGBS. A significant water-fugacity dependency of creep strength of olivine on water fugacity was observed, and the water fugacity exponent was obtained to be 1.25. The dependency of creep strength of olivine controlled by DisGBS on pressure is weak due to competing the pressure-hardening effect of activation volume and the pressure-softening effect of water fugacity. Because creep strength of olivine controlled by the grain boundary sliding is insensitive to pressure, the estimated viscosity of water-poor olivine is independent of depth and is in a range of 10$^{20}$-10$^{21.5}$ Pa s throughout the upper mantle, which is consistent with geophysically observed viscosity profiles. Viscosity of the deep upper mantle would be overestimated by ~10-10$^{4}$ times if we assume the conventional dislocation creep mechanism for water-poor olivine.

Keywords: grain boundary sliding, olivine, upper mantle, pressure, water, viscosity
Observations of grain- to multi-grain-scale deformation of mineral aggregates deformed by diffusion creep

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We deformed cylindrical polycrystalline samples of synthesized forsterite + diopside and anorthite mineral aggregates at its diffusion creep regime. We polished the lateral side of the sample where we imposed grooves parallel to the compression axis of the sample using a focused ion beam. These marker lines allow us to observe fine-scale deformation of the sample. After the high temperature one atmosphere compression creep experiment, we observed the sample surface with marker lines under scanning electron microscope.

Strains of bulk sample and of the marker line exhibited the similar values indicating that the similar deformation proceeded both at bulk and surface regions of the samples. Grain rotation, which was identified by misfits of the markers at grain boundaries and rotation of intra-granular markers, were frequently observed in all the samples. No distortion of the markers within the grains was found indicating the absence of intragranular deformation process such as a glide of dislocations; however, in the samples deformed at high stress (~300 MPa), curved intra-granular markers were observed, which is consistent with dislocation activity at high stress condition. The changes in grain configuration were also observed elsewhere in the samples demonstrating significant operation of grain boundary sliding which produced grain switching. Grain rotations were controlled by the orientation of long flat crystallography-controlled grain boundaries with respect to the compressional direction. Such grain rotation resulted in a significant development of crystallographic preferred orientation in the samples.

Keywords: diffusion creep, mineral
Microstructure and growth-kinetics of forsterite reaction rim under high pressure

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Since many transport properties, such as rheology, highly depend on grain-size of the constituent materials, knowledge of grain-growth is important for accurate understanding of dynamics in the Earth’s mantle. Tasaka and Hiraga (2013) showed that grain-growth in forsterite-enstatite two-phase system is rate-limited by growth of secondary phase through Mg-O grain-boundary diffusion in primary phase. In this study, we have experimentally studied growth-kinetics of forsterite (Mg\(_2\)SiO\(_4\)) reaction rim at deep upper mantle conditions which is controlled by Mg-O grain-boundary diffusion in forsterite. Based on the experimental results, depth dependence of grain-growth in the upper mantle is discussed.

Experiments were carried out using multi-anvil apparatus installed at GRC, Ehime University, Japan. The starting materials, MgO (single crystal) and MgSiO\(_3\) (powder or aggregate), were packed in Pt capsule. The annealing experiments were conducted at pressure of 3.0-11.1 GPa and temperature of 1473-1873 K for duration of 0-780 min. Recovered samples were analyzed by SEM or FE-SEM for microstructural observation and by FT-IR to examine water content.

Water content in MgO single crystal in the recovered samples was relatively low and 6.9 wt ppm H\(_2\)O at the maximum. The Pt marker, which was originally placed at MgO-MgSiO\(_3\) boundary, was always on MgO-Mg\(_2\)SiO\(_4\) boundary indicating that Mg-O diffusion in Mg\(_2\)SiO\(_4\) is the rate-limiting process in the rim growth. MgSiO\(_3\) inclusions were found in Mg\(_2\)SiO\(_4\) grains suggesting the grain-boundary diffusion is rate-limiting. Based on the analysis using equation for reaction-rim growth rate-limited by grain-boundary diffusion (Gardes and Heinrich, 2011), the activation energy and the activation volume were determined to be 375 kJ/mol and -2.1 cm\(^3\)/mol, respectively. Although reason for the small negative value of the activation volume is not quite clear, this may be due to successive structural change of grain-boundary. The results suggest that the grain-growth in the Earth’s upper mantle is faster at deeper part.

Keywords: Upper mantle, Element diffusion, Olivine, Forsterite, Grain-growth, Rheology
In situ creep strength measurement on ringwoodite up to 1700 K at 17-18 GPa using a deformation-DIA apparatus

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Deformation experiments on polycrystalline (Mg₀.⁹Fe₀.¹)₂SiO₄ ringwoodite have been conducted in uniaxial geometry up to strains of 15.5 % at 17.3-17.9 GPa, 1500-1700 K and strain rates of 3.46-3.59 × 10⁻⁵ s⁻¹ using a deformation-DIA apparatus at the synchrotron facility SPring-8. Stress magnitude was measured from azimuthal dependence of three diffraction peaks of ringwoodite (220, 311 and 400) by two-dimensional X-ray diffraction. Strain was calculated from X-ray radiographs of strain markers. Stress magnitude of ringwoodite at steady-state deformation, i.e. creep strength, was 400 MPa at 1500 K. The creep strength decreased to 130 MPa with increasing temperature to 1700 K. The creep strengths of this study are lower than those (4-6 GPa) observed at 3-10 GPa and room temperature using a deformation-DIA apparatus (Nishiyama et al., 2005) and those (1.7-2.8 GPa) determined at 21-23 GPa and 1800 K using a rotational Drickamer apparatus (Hustoft et al., 2013; Miyagi et al., 2014). Further deformation experiments and subsequent sample analyses should be performed to determine flow laws of ringwoodite and in turn to discuss viscosity at the lower part of the mantle transition zone.

Keywords: ringwoodite, creep strength, stress, strain, deformation-DIA apparatus, synchrotron X-ray
Si and O self-diffusion in stishovite

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Seismological studies revealed the seismic reflectors in the lower mantle at the depth from 900 to 1850 km (e.g., Kaneshima and Hellfrich, 1999; Niu et al., 2001; Castle and Creager, 1999). These reflectors are interpreted as pieces of subducted ancient oceanic crust (Kaneshima and Hellfrich, 1999), indicating the high viscous oceanic crust to inhibit the deformation during subduction and further mixing with the lower mantle. At lower mantle depth, stishovite is as much as 20% in the basaltic composition and 40% in the sedimental rocks (Irifune and Ringwood, 1993; Ono et al., 2001; Irifune et al., 1994). Therefore, the viscosity of stishovite may be one of the key parameter to constrain the viscosity of the ancient oceanic crust in the lower mantle. Viscosity of solid is thought to be controlled by diffusion of the constituting elements. To know the viscosity of stishovite, thus, diffusivity of Si and O was studied by means of high pressure experiments in this study.

Single crystals of stishovite (~500 µm in size) were synthesized at 12 GPa in a Kawai-type high pressure apparatus by slow-cooling method (Shatskiy et al., 2010). The polished {110} surfaces were coated with ~150 nm 29Si and 18O enriched SiO2 layer in a high-vacuum thermal evaporator. The coated crystals were again compressed at 14-21.5 GPa and 1673-2073 K in the Kawai-type apparatus for diffusion. The recovered samples were measured by secondary ion mass spectroscopy (SIMS) to obtain diffusion profiles by the depth profile method. The obtained profiles were fitted to semi-infinite diffusion model and the fitting results were \( \Delta E \), \( \Delta V \) and log\( D_0 \) to be 178.6±4.4 kJ/mol, 6.0±0.2 cm³/mol and -12.9±0.14 m²/s, respectively, for Si diffusion, and 262.7 kJ/mol, 5.0±1.6 cm³/mol and -10.1 m²/s, respectively, for O diffusion, where \( \Delta E \), \( \Delta V \) and \( D_0 \) are the activation enthalpy, activation volume and pre-exponential factor, respectively.

Our results show that Si diffusion in stishovite is slower than O under mantle conditions and hence the deformation of stishovite is controlled by Si diffusion. The diffusivity of Si in stishovite is ~3 orders of magnitude smaller than that in wadsleyite and garnet (Shimojuku et al., 2009, 2013), ~4 orders of magnitude smaller than that in ringwoodite (Shimojuku et al., 2009) and perovskite (Yamazaki et al., 2000). We can conclude that stishovite is the hardest mineral among the main mantle minerals. The survival of the subducted slab observed as seismic reflector at the lower mantle might be supported by the significantly high viscous stishovite.

Keywords: stishovite, diffusion, viscosity, subducted oceanic crust
Development of a rotational Drickamer apparatus (RDA)

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Many major developments in the study of deformation of Earth materials occurred based on the developments of new techniques. They include the contributions from UCLA lab (Griggs) based on the development of a solid-medium deformation apparatus, and the contributions from ANU lab (Paterson) based on the development of a high-resolution (but low pressure) deformation apparatus. Surprisingly (or embarrassingly), there was a large hiatus in the development of new technology between ~1980 and ~2000 and until very recently reliable experimental results on rheological properties were limited to ~0.3 GPa (~10 km).

Recognizing this, a group of scientists in USA started a serious effort of developing new techniques of quantitative deformation experiments. The developments include the design and operation of new types of deformation apparatus and the development of synchrotron-based techniques of measuring stress and strain. In this presentation, I will review the development of RDA (rotational Drickamer apparatus) and a theory of stress measurements performed in my lab. The advantage of RDA include (i) the capability of conducting high-P (and T) deformation experiments (compared to D-DIA), and (ii) the capability of large-strain deformation experiments. A brief description of RDA deformation experiments and some recent results will be presented including the results of deformation of a mixture of bridgmanite + ferro-periclase at the lower mantle conditions.

Keywords: high pressure, deformation experiments, synchrotron facility, lower mantle, RDA
Dislocation mobility in ringwoodite as a function of temperature and water content

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Ringwoodite is the dominant mineral in the lower part of the Earth’s mantle transition zone from 520 to 670 km depth. Understanding the creep properties of ringwoodite is thus essential to know the geodynamical process at this depth. Dislocation creep, which could cause seismic anisotropy and non-Newtonian viscosity, is an important creep mechanism for high temperature plastic deformation of minerals. Since the dislocation creep rate is controlled by the mobility of dislocations, we measured the dislocation mobility in ringwoodite single crystals as a function of temperature and water content under the mantle transition zone conditions.

The ringwoodite single crystals were synthesized from natural olivine at 1800 K and 22 GPa in a multi-anvil apparatus. The crystals were deformed after synthesis by increasing the load to create high density dislocations. The deformed crystals were surrounded by CsCl and annealed at 22 GPa, 1600-2000 K for dislocation recovery. The water contents in the crystals were measured by Fourier transform infrared, the dislocation densities before and after recovery were determined by transmission electron microscopy, and the dislocation mobility were calculated from the dislocation densities and annealing durations.

The activation enthalpy for dislocation mobility is $360 \pm 90$ kJ/mol. This value is much lower than that for Si diffusion ($480 \pm 90$ kJ/mol), but similar to that for O diffusion ($370 \pm 80$ kJ/mol) (Shimojuku et al., 2009). It is probably because the dislocation movement in ringwoodite is controlled by both Si and O due to their similar diffusion rates (less than 0.5 orders of magnitude difference, Shimojuku et al., 2009). Temporary results suggest that the water content exponent for the dislocation mobility in ringwoodite is about 1.1($\pm$0.9). Namely, in contrast with olivine (Fei et al., 2013; 2014), water may have large effect on ringwoodite rheology. But more experimental data are required for precise determination of the water content exponent.

Shimojuku et al. (2009), EPSL 284, 103-112.
Fei et al. (2014), JGR 119, 7598-7606.

Keywords: ringwoodite, dislocation mobility, recovery, water content
Preliminary deformation experiments for in-situ stress-strain measurements of bridgmanite

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In order to discuss mantle dynamics in the Earth’s interior, knowledge of viscosity of the Earth’s lower mantle, which is the highest of the whole mantle, is important. Viscosity models of the Earth’s lower mantle were reported by geophysical observations. However, observation values of viscosity have large variety (2-3 order magnitude). Although determination of viscosity of lower mantle minerals by high pressure experiments is needed to understand mantle dynamics, stress-strain relationship for bridgmanite, which are principal minerals of the Earth’s lower mantle, are not reported due to difficulty of high pressure deformation experiments. In this study, we tried in-situ stress-strain measurements of Mg-Pv at 27 GPa.

In-situ uniaxial deformation experiments were conducted using MADONNA, which is D-DIA apparatus, as Kawai-type apparatus at SPring-8 BL04B1. Experimental conditions are 27.3 GPa, 1473 K estimated by equation of state on bridgmanite (Katsura et al., 2009). WC anvils with slit or corn (5°) to take tomography and 2D X-ray diffraction, was used along X-ray path. Two-dimensional X-ray diffraction patterns were taken for 300 s using CCD detector. To calculate the stress magnitude from the X-ray diffraction data, we used a model of stress-lattice strain relationship (Singh et al. 1998),

\[
d_{hkl}(\psi) = d_{0hkl} [1+(1-3\cos^2\psi) \sigma/6 G_{hkl}] (1)
\]

where \(d_{hkl}\) is the d-spacing measured as a function of azimuth angle \(\psi\), \(d_{0hkl}\) is the d-spacing under the hydrostatic pressure, \(G_{hkl}\) is the appropriate shear modulus for a given hkl, and \(\sigma\) is the uniaxial stress. Pressure and stress were estimated using bridgmanite (111) diffraction peak at deformation experiments. X-ray radiographies of the strain markers was taken using an imaging system composed of a YAG crystal and a CCD camera with an exposure time of 10 s.

Uniaxial tension stress and strain of Mg-Pv at 27.3 GPa, 1473 K were estimated as \(1.3\) GPa and \(4\%\) during deformation by differential ram. We confirmed deformation experiments at the lower mantle pressure conditions can be conducted by WC anvils. We will perform additional deformation experiments with large strain.

Keywords: Bridgmanite, In-situ measurements, deformation experiments, The Earth’s lower mantle
Iron concentration around dislocation in naturally deformed olivine

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The concentration of specific atom on dislocation core can be caused by pipe diffusion and Cottrell atmosphere. Both the phenomena are important for the property of materials including rocks and minerals. As an example of the former, Fe concentration has been reported in the naturally deformed olivine (e.g. Plumper et al., 2011). Pipe diffusion is important for atomic migration during various reactions in the Earth, such as metasomatism and serpentinization. On the other hand, Kitamura et al. (1986) and Ando et al. (2001) have reported Fe concentration in the mantle-derived olivine caused by Cottrell atmosphere. The Cottrell atmosphere strongly influences plasticity of materials in the low strain rate regime. Therefore, the discovery of Cottrell atmosphere from the mantle-derived olivine indicates that the effect on the plasticity of olivine is important to understand mantle dynamics under very low strain rate condition. However, the possibility of pipe diffusion cannot be neglected completely to explain the observations of Kitamura et al. (1986) and Ando et al. (2001). Here, we carried out more detailed chemical composition analysis of the mantle-derived olivine to assess whether the Fe concentration on dislocation core is a common phenomenon, and to clarify the exact mechanism of the Fe concentration, i.e. Cottrell atmosphere or not.

We studied two types of peridotites, which are xenolith-type in basalt (Takashima, Megata, Kurose and Salt Lake) and alpine-type (Uenzaru and Horoman) by using EPMA and ATEM techniques. EPMA and ATEM analyses show Fe concentration at dislocations in all the studied samples, which suggests that it is a common phenomenon in mantle peridotites. Fe-enrichment at the rim of olivine grains and other major element concentration on dislocations, which are general features of pipe diffusion, cannot be observed. Therefore, the mechanism of Fe concentration on dislocation core in olivine grains is possibly derived by Cottrell atmosphere, not pipe diffusion.


Keywords: Cottrell atmosphere, olivine, dislocation
Fabrication of textured Fe-free and Fe-bearing olivine aggregates using colloidal processing under high magnetic field

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Crystallographic preferred orientation (CPO) of minerals is considered to be widely produced in the Earth’s interior. Due to the presence of anisotropic physical properties such as elasticity, plasticity, electron conductivity and etc... of single crystal minerals, their bulk rock properties can significantly be affected by the presence of CPO. To measure CPO effect on the bulk rock properties by room experiments, it is required to prepare polycrystalline materials with ideally controlled CPO.

Magnetic field was applied to fine-grained (∼120 nm) equigranular Fe-free and Fe-bearing olivine particles, which were dispersed in ethanol (solvent) with dispersant (polyethyleneimine). We expected the particles to align with respect to magnetic direction due to their magnetic anisotropy. The aligned particles were gradually deposited on a solid-liquid separation filter during ethanol drainage. The directions of magnetic field and particle deposition were parallel. The dried particles were then densified isostatically at 200 MPa for 10 min and sintered using the alumina tube furnace with vacuum pump.

Highly dense (density of ≥ 99%) and fine grained (∼1 µm) samples with a-axis alignment for Fe-free and c-axis alignment for Fe-bearing olivine to the magnetic direction were obtained. Such synthesized aggregates will allow us to measure CPO effect on the physical properties of olivine aggregate.

Keywords: crystallographic orientation, olivine
High pressure and high temperature deformation on lawsonite: Implication for low velocity layers in subduction zones

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Low-velocity layers (LVLs) located in the upper portions of subducting slabs, are regions of lower seismic wave velocities than those in the surrounding mantles. LVLs apparently persist to depths of 100-250 km [1,2]. Hydrated mafic rocks provide a plausible explanation for the origin of LVLs and trench-parallel/normal S-wave fast polarization. Lawsonite (CaAl₂Si₂O₇(OH)₂·H₂O), which is stable at depths greater than serpentine minerals, is considered to be one of the prime candidate hydrous minerals that can be present deep in the cold subduction zones [3,4]. Single crystals of lawsonite have a high elastic anisotropy, suggesting that the development of the crystallographic preferred orientations (CPOs) when it deformed might strongly affect the seismic properties [5].

This study reports deformation experiments on lawsonite aggregates that were conducted at high pressure and high temperature corresponding to 150 km depth in the subduction zone, to investigate the development of CPOs and the seismic properties of lawsonite. Experiments were performed using a multi-anvil apparatus with six independently acting rams installed at Bayerisches Geoinstitut, Bayreuth University. The starting material consisted of fine-grained (<25 µm) natural lawsonite powder, which was loaded in a Pt capsule and annealed for >20 h at 5 GPa, between 500 and 800 °C. The samples were then deformed using pure or simple shear geometry at strain rates of 10⁻⁴-10⁻⁶ s⁻¹ and a finite strain of 0.3-1.0. Recovered samples were analyzed using a scanning electron microscope (SEM) coupled with an electron backscatter diffraction (EBSD) detector and a transmission electron microscope (TEM).

The deformed lawsonite aggregates display a porphyroclastic texture characterized by a bimodal grain size distribution. The microstructures with dynamically recrystallized grains imply the evidence for the deformation through grain-boundary sliding accommodated by diffusion creep due to the grain size reduction, whereas the porphyroclasts (20-50 µm in size) have undulose extinction, deformation lamellae, irregular grain boundaries, and many sub-grain boundaries. The porphyroclasts also display a CPO characterized by a girdle distribution of the [100] axes in the shear plane with a maximum concentration close to the shear direction. The [010] axes form a maximum subnormal to the shear plane. The microstructures and the occurrence of a CPO show that the dominant deformation mechanism for the porphyroclasts is dislocation creep. These results of CPOs diverge from those of previous studies of natural lawsonite rocks [e.g., 6], which might result from differences in experimental or natural conditions. TEM images show a variety of dislocations with a high density of {110} wedge-shaped mechanical twins. Lawsonite seems to have numerous potential slip systems with [100][010] appearing to be the most dominant. The calculated anisotropies of the seismic wave velocities (AVp = 2% and AVs = 6%, respectively) are characterized by the fast propagation of P-wave is oriented subnormal to [010] maxima of the deformed lawsonite aggregates and the polarization of the fastest S-wave is perpendicular to the foliation. This indicates that lawsonite can contribute to the LVL observations and trench-normal S-wave splitting observed at depth of >150 km in the cold subducting slab of northeastern Japan [7].


Keywords: lawsonite, deformation, crystallographic preferred orientation, high pressure, subduction zone, low velocity layer
Calibration for stress measurement of Griggs-type high temperature and high pressure deformation apparatus

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1. Introduction

It is necessary to perform deformation experiments in appropriate temperature and pressure conditions equivalent to the inside of the earth to characterize rheological properties of rocks. There are several types of deformation apparatus using different confining media such as gases, liquids or weak solids (e.g., Tullis and Tullis, 1986). Liquid medium apparatus has a disadvantage that it cannot be used for temperatures above 500 °C because of prevention from alteration of oil. Gas medium apparatus has the most accurate stress measurements because of using internal force gauge. However, experiments are typically restricted to confining pressures less than 300 MPa. Solid medium apparatus can provide us high pressure (~2.0 GPa) easily and safely. However, its stress measurements accuracy is low mainly because of frictions between the confining media and samples or loading piston (e.g., Tullis and Tullis, 1986).

Recently, comparison of stress measurements of Griggs-type apparatus with solid salt assemblies (SSA) and gas apparatus provide a calibration law for Griggs apparatus with SSA (Holyoke and Kronenberg, 2010). This calibration law allowed steady-state stresses to be measured accurately to within ±30 MPa. However, it was not able to reproduce elastic, transient and post-yield behaviors because the calibration law was obtained from the comparison of stresses measured only at 5% strain. Moreover, since the calibration was carried out in low confining pressure, influence of confining pressure to stress measurements of Griggs apparatus is not clear. Calibration law for Griggs apparatus in various deformation conditions are required for revealing detailed rheological properties of the lower crust and upper most mantle rocks. In this study, we derived a calibration law for stress measurements of Griggs apparatus by the master curve method.

2. Axial compression experiments and construction of master curves

Axial compression experiments were performed on high-purity metals (nickels and molybdenums) using a Griggs apparatus with SSA. Experiments were performed in several conditions (confining pressures: ~300 MPa, ~1200 MPa, ~1500 MPa, temperatures: 600 °C, 700 °C, 800 °C, strain rates: 2×10^{-4} /s, 2×10^{-5} /s, 2×10^{-6} /s). Measured stresses were consistent with results of the former study (Holyoke and Kronenberg, 2010) within ±30 MPa under the identical confining pressure of ~300 MPa. Measured stresses tended to become higher with confining pressures. Logarithms of steady-state stresses almost linearly increase with confining pressures in the range of this study. Obtained mechanical data were analyzed based on high temperature viscoelastic constitutive law proposed by Shimamoto (1987). Then, a master curve which normalized temperatures strains and confining pressures was constructed. A master curve from mechanical data (Holyoke and Kronenberg, 2010) using gas apparatus was also constructed.

3. Derivation and application of a calibration law

Master curves were constructed from identical materials between Griggs and gas apparatuses under normalized temperatures, strains, and confining pressures. Therefore, it is considered that differences between both master curves are derived from distinction of various rheology components of two apparatuses. A calibration law for Griggs apparatus was derived from differences in both master curves. Applying the calibration law to stress measurements of metals using Griggs apparatus with SSA, it became possible to reproduce gas apparatus/s stresses not only at steady-state but also at elastic, transient and post-yield behaviors within an error of ±30 MPa. Moreover, calibration can be extended to higher confining pressure up to 1500 MPa. When the calibration law was also applied to the stresses of carbonate rock, although an error was at most ~70 MPa, elastic to post-yield behaviors could be reproduced.

Keywords: rheology, rock deformation experiment, solid medium deformation apparatus
Weak-beam dark-field TEM characterization of dislocations in wadsleyite deformed in simple shear at 18 GPa and 1800 K

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Characterization of dislocations in textured wadsleyite is important in understanding crystallographic preferred orientation (CPO) of wadsleyite and in turn seismic anisotropy at the mantle transition zone. A [001](010)-textured wadsleyite was recently obtained by deformation experiments on wadsleyite in simple shear at 15-18 GPa and 1770-1870 K with a deformation-DIA apparatus (Kawazoe et al., 2013; Ohuchi et al., 2014). However, [001] dislocations have been rarely reported in wadsleyite in the literature (cf. Cordier, 2002). To reconcile the wadsleyite CPO pattern with its slip systems, dislocation microstructures of the [001](010)-textured wadsleyite have been investigated in weak-beam dark-field imaging in a transmission electron microscope. 1/2<101> partial dislocations on the (010) plane are characterized with [100] dislocations on the (001) plane and 1/2<111> dislocations forming {011} slip bands. The former partial dislocations are extended on the (010) stacking fault as a glide configuration (i.e. Shockley-type stacking faults with 1/2<101> displacement vector). The [001] slip on the (010) plane occurs by glide of the dissociated dislocations on a sub-oxygen close packing plane, which can play an important role to generate the crystallographic preferred orientation patterns reported in water-poor deformation conditions (e.g., Kawazoe et al. 2013, Ohuchi et al. 2014).

Keywords: wadsleyite, crystallographic preferred orientation, dislocation, seismic anisotropy, transmission electron microscopy, deformation-DIA apparatus