

## Formation of weak faults during large shear deformation experiments of bimineral mixtures

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Measurements of heat flow, temperature anomaly, and stress orientations along the mature faults affirm that these faults are mechanically weak (e.g., Lachenbruch & Sass, 1980, Zoback, 2000, Kano et al., 2006). One of the explanations for these weak faults is that the presence of weak minerals (fault lubricants) such as phyllosilicates along the fault zones. Frictional experiments on bimineralic mixtures with phyllosilicates are conducted to examine its operation for weakening agent (e.g. Moore & Lockner, 2011). However, these previous studies are conducted with limited shear strain (mostly <10) in spite of significance of fabric development on weakening has been pointed out (Collettini et al., 2009). We thus have performed large strain, friction experiments for two kinds of fault lubricants of graphite and smectite with quartz, to understand how frictional behavior changes with fraction, shear strain, and associated textural maturation. Experiments were done with dry and both dry and water-saturated conditions for graphite and smectite mixtures, respectively. For the graphite-quartz mixtures, friction typically displays strain weakening at any slip rates we tested (150um/s-1.3 m/s). 10-30 vol% mixtures in particular decrease friction coefficient to almost half of the original associated with intense comminution and formation of graphite-connected slip surface within the gouge zone. Hence the relationship between strength versus graphite fraction evolves from gentle to abrupt sigmoidal curve which drops at a fraction of 10-30 vol%, with increasing shear strain. The friction of the smectite-quartz mixtures sheared at 30-150 um/s displays steady-state friction throughout the experiments while strain weakening can be observed for those sheared at faster than 22 mm/s (see Oohashi and Hirose, 2013, this meeting). Strength versus smectite fraction at 30-150 um/s shows sigmoidal curve which drops at a fraction of 30 vol% without visible correlation with shear strain. Textural observations suggest that slip localized within the smectite without visible comminution for the mixtures >30 vol %. These differences on slip localization between two fault lubricants may be ascribed to their physical properties to form foliation within the gouge.

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Keywords: Fault weakening, Graphite, Smectite, Fault gouge, Friction experiment

## Relaxation processes of a thick granular layer at seismic slip rates

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We report on laboratory experiments designed to explore transient responses of a thick granular layer following a step change in slip velocity at seismic slip rates. Experiments were performed at constant normal stresses of 10-30kPa using a ring shear apparatus with inner/outer diameters of 15mm/25mm. We measure the friction coefficient and thickness of glass beads layer at sliding velocities between 0.5 and 3 m/s. Experimental results show that the friction coefficient and layer thickness suddenly increases/decrease as sudden increase/decrease of sliding velocity and then exponentially decay to new steady state with characteristic slip length. We found that characteristic slip length is of the order of 10m when the surface of sliding wall is rough. The response to a velocity step decreases simply symmetric to that to a velocity step increase. In this presentation, we discuss the effect of sliding velocity, normal stress, and surface roughness of the sliding wall on characteristic slip length.

Keywords: high-velocity friction, granular matter, rheology

## Repeated seismic slips recorded in ultracataclastic veins along active faults of the Arima-Takatsuki Tectonic Line

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It is well known that direct evidence of earthquakes within fault zones is limited to the presence of pseudotachylyte (e.g., Lin, 2008). In addition to pseudotachylyte, previous studies have shown that the meso- and microstructural features of cataclastic veins that lack the primary cohesion of the host rocks, including crush-origin pseudotachylyte, fault gouge, fault breccia and some calcite veins, may represent primary evidence of brittle deformation caused by recurrent seismic slip within seismogenic fault zones (e.g., Lin et al., 2012, 2013a,b). It has also been reported that during the 2008 Mw 7.8 Wenchuan earthquake, ultracataclastic veins were produced along the seismic slip plane and injected into fractures within the seismogenic fault zone (Lin, 2011). Therefore, studies on cataclastic veins would provide new insights into the deformation process of seismic slip recorded in seismogenic fault zones.

In this study, we report on the structural mode of typical ultracataclastic veins including crush-origin pseudotachylyte and fault gouge veins that formed repeatedly as simple veins and complex networks within a fault zone along active faults of the Arima-Takatsuki Tectonic Line (ATTL), southwest Japan. We also discuss the formation mechanisms of such veins and their tectonic significance in terms of seismic faulting events.

Field investigations, combined with meso- and microstructural analyses, reveal that numerous ultracataclastic veins are widely developed within a fault zone (<150 m wide) as simple veins, complex lenses, and networks, along active faults of the ATTL, southwest Japan. These veins comprise mainly crush-origin pseudotachylyte vein and weakly consolidated to unconsolidated fault gouge that is black, dark-brown, brown, gray, and brownish-red in color. Meso- and microstructural features show that these pseudotachylyte and fault gouge veins and networks formed during multiple stages, as earlier veins are generally cut and overprinted by younger veins, indicating that the vein-forming events occurred repeatedly and that ultracataclastic material was injected into networks of faults and fractures in the fault zone. The pseudotachylyte and fault gouge veins are characterized by an ultrafine- to fine-grained matrix and angular to subangular fragments of host granitic rocks of various sizes, ranging from sub-micron to millimeters. SEM-EDS and powder X-ray diffraction analyses show that all the ultracataclastic veins are characterized by crystalline materials composed mainly of quartz and feldspar, similar to the host granitic rocks.

The present results support the existing hypothesis that ultrafine- to fine-grained materials formed by comminution can be fluidized and injected rapidly into fracture networks located far from the source fault plane in a solid-fluid-gas system during seismic slip; therefore, such materials provide a record of paleoseismic faulting events that occurred repeatedly within the seismogenic fault zone.

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Keywords: Arima-Takatsuki Tectonic Line active fault zone, ultracataclastic vein, pseudotachylyte, fault gouge, fine-grained material, fluidization

## Friction of granular matter with a wide dispersity

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A simple theory for a constitutive law for steady state dynamic friction in granular matter is presented. Starting from the energy balance equation together with the kinetics of grains, the energy dissipation rate is estimated, which directly leads to a constitutive law. The result indicates that granular matter of lower density is stronger than higher density systems, albeit somewhat counterintuitive. This is a consequence of the fact that the grain rearrangement, which causes energy dissipation, is more frequent in a system of lower density. Thus, the velocity-strengthening nature of granular friction is naturally explained by the negative shear-rate dependence of the density. The present theory also qualitatively explains the experimental observation that a system of smaller layer thickness tends to be velocity-weakening. This theory also applies to systems of fractal size distribution.

Keywords: fault gouge, comminution

## Diffusion creep experiments on polycrystalline anorthite

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It is very important to investigate the rheological properties of the lower crust because it plays a major role on the cause of inland earthquakes. In this study, we selected anorthite as a representative mineral of the lower crust and examined its flow properties.

Previous studies have proposed flow laws of polycrystalline anorthite with different amount of water. Based on these results, the polycrystalline anorthite will deform under diffusion creep on the conditions of the lower crust (temperature 400 to 1000C, grain size of < 100 um, which is often observed in mylonites). The synthetic samples used in previous studies, however, contained a little water and glass phase which are difficult to eliminate with their technique for the sample synthesis .

We prepared polycrystalline anorthite using the technique that does not allow the contamination of water and glass phase. Creep tests were performed at temperatures ranging from 1150 to 1380C, stresses from 10 to 120 MPa, strain rates between  $5 \times 10^{-7}$  and  $2 \times 10^{-4} \text{ s}^{-1}$ , and confining pressure of 0.1 MPa. We read strain rate from the rate becoming independent on time. Arithmetic mean grain size of the specimens was 1 um before and after the creep tests, which were found by the scanning electron microscopy. Log stress and log strain rate showed a linear relationship where its slope gave a stress exponent,  $n$  of 1, indicating that samples were deformed under diffusion creep. We obtain activation energy,  $Q$  of 490(30)  $\text{kJ mol}^{-1}$  and the preexponential factor,  $A$  of  $10^{10.7} \text{ MPa}^{-1} \text{ um}^3 \text{ s}^{-1}$ . Our samples exhibited two orders of magnitude stronger relative to the previous samples. There was no explicit difference in activation energy. The difference in the strength can be attributed to the presence of water and glass phase in the previously studied samples, which can reduce the sample strength.

We applied our obtained flow law to the temperature condition of the lower crust finding that the viscosity of polycrystalline anorthite with grain size of 10 um is harder than that of polycrystalline olivine deformed under dislocation creep at dry condition.

Keywords: polycrystalline anorthite, diffusion creep, the lower crust

## Heterogeneous distribution of water and strain localization of polycrystalline synthetic anorthite

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Recent tomographic investigations for the crust have observed low seismic velocity zones and high electrical conductivity zones especially beneath the active fault zones in the crust. In these zones, it is assumed that aqueous fluids are introduced through fractures and plastic deformation of surrounding rocks has been enhanced (e.g. Wannamaker et al. 2009).

Previous deformation experiments, which focused on the effect of water for feldspar as a dominant constituent in the lower crust, have been performed under equilibrium water conditions within rocks and minerals. These experiments therefore have not evaluated the process for brittle-plastic transition and enhancement of plastic deformation under non-equilibrium water condition which are related to the above tomographic observations.

In this study, we performed shear deformation experiments on initially dry synthetic polycrystalline anorthite under the condition where water was introduced externally and heterogeneously distributed within the sample. Polycrystalline anorthite used in the experiments is composed of the average grain size of 3 micrometers with Si-rich melt of 5 vol%. The sample column of 6.2 mm phi was cut by 45 degree, and 1 mm thickness was obtained. A Ni strain marker was set within the sample in a direction normal to slip orientation. Then, the sample was sandwiched by 45-degree-cut alumina shear pistons. The shear deformation was conducted using a solid pressure medium (Griggs type) deformation apparatus at 900 degree C, confining pressure of 1.0 GPa, and shear strain rate of  $10^{-3.5}$ ,  $10^{-4.0}$ ,  $10^{-4.5}$ /sec up to shear strain of 2. Pyrophyllite powder was added around the ends of two alumina pistons as a source of water by dehydration under the experimental condition.

The stress-strain curves showed significant weakening only for the experiments with 0.5 wt% added water. For example, in an experiment conducted at shear strain rate of  $10^{-4.5}$ /sec with 0.5 wt% water added, the sample was weakened below the differential stress of 50 MPa. On the other hand, in other experiments of the same shear strain rates and added water less than 0.5 wt%, the differential stress reached 1000 MPa and then weakened. Observations of recovered samples under a polarized optical microscope showed that cataclastic flow dominated in the samples of less added water. Plastic deformation dominated in the samples of 0.5 wt% added water experiments, and the strain marker locally shows shear strain of 5.

We measured water distribution in deformed samples by mapping measurements of infrared spectroscopy. The IR spectra of the sample show broad water absorption bands at 2800-3800  $\text{cm}^{-1}$ , indicating that the presence of aqueous fluid at grain boundaries. The maximum water content was 130 ppm  $\text{H}_2\text{O}$  in the samples of 0.1 wt% water added experiments. On the other hand, in the samples with the 0.5 wt% added experiment at the shear strain rate of  $10^{-4.0}$ /sec, the maximum water content was 550 ppm  $\text{H}_2\text{O}$  in strain-localized areas. This sample also includes cataclastically-deformed area, where the water content was c.a. 250 ppm  $\text{H}_2\text{O}$ . Thus, deformation mechanisms and enhancement of plastic deformation greatly depend on heterogeneity of water content. In this study, we also discuss differences of mechanical and microstructural differences which depend on added water content. Then, we discuss relationship between deformation and heterogeneous distribution of water within the samples.

Keywords: Griggs deformation apparatus, Shear deformation experiment, Water introduction, Enhancement of plastic deformation, IR spectroscopy

## Supercooled melt inclusions in lower-crustal granulites and rapid exhumation by channel flow

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We found unexpected felsic (granitic) inclusions with quench textures such as spherulite and dendrite (felsite inclusions), similar to some volcanic rocks, within garnet in presumably slowly cooled lower-crustal granulites of various geologic ages ranging from Early Proterozoic to Middle Paleozoic and wide global distribution (the Limpopo Belt, the Grenville Province, the Lutzow-Holm Complex of East Antarctica, the Highland Complex of Sri Lanka, and the southern Bohemian Massif). The well-preserved textures of felsite inclusions are indicative of melts formed by anatexis during high-pressure and high-temperature metamorphism, crystallization under far-from-equilibrium conditions (at >50 degree undercooling) and subsequent rapid cooling. The occurrence of felsite inclusions in granulites in restricted tectonostratigraphic zones in Sri Lanka, among others examples, may be the first geologic evidence for fast exhumation of lower-crustal rocks to andalusite-stable upper-crustal conditions by channel flow in a continental collision orogen. We hypothesize that granulites ascend episodically along discrete high-strain zones and cool as fast as some felsic magmas. This conclusion sheds new light on the debate regarding the deep crustal processes and necessitates changes to fundamental beliefs about exhumation rates based on rates of plate convergence (1-10 cm/year).

Keywords: felsic melt inclusions, granulite exhumation, supercooling, channel flow

## Interconnection of ferro-periclase reduces viscosity of the subducted slab at the top of lower mantle

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Subduction of cold slab is one of the most important phenomena for dynamics of the Earth and hence many studies have been performed based on geophysical observation, geodynamical simulation and mineral physics. Seismic tomography revealed that the subducting slab classified in two types from the view of shape of slab around 660 km depth discontinuity: one is the continuous penetration into the deep lower mantle and the other is stagnation around 660 km discontinuity forming horizontal layer at this depth (e.g., 1, 2). However, recent tomographic images show the trapped slabs around 1000 km depth, for example, Tonga, Java, Kermadec, Mariana and so on (3, 4). The slab shape around 660 km depth can be explained by the viscosity structure after phase transformation in which relatively low (high) viscosity with colder (warmer) slab because of small (large) grain size (5). However, for understanding the whole mantle convection, the mechanism to trap the subducting slab around 1000 km depth, related to the rheology of lower mantle rock, should be clarified.

The mineral assembly of the subducting slab in the lower mantle is approximately 80 volume % of silicate perovskite and 20 volume % of ferro-periclase. Therefore, we often approximate the bulk rheology of slab by that of silicate perovskite. This approach works well when dominant phase is weaker than secondary phase, for example, a case of the upper mantle (6). However, in the case of the lower mantle, this approximation does not work because silicate perovskite is much stronger than ferro-periclase (7). A presence of ferro-periclase may significantly reduce bulk viscosity when the interconnected layer of ferro-periclase is formed in the bulk rock (8). To estimate the bulk viscosity, we need to understand not only individual viscosities of silicate perovskite and ferro-periclase but also the connectivity of ferro-periclase in the lower mantle rock.

In the present study, we observed the electrical conductivity change of post-spinel phase just after phase transformation from ringwoodite with time at the uppermost lower mantle conditions to detect the interconnectivity of ferro-periclase. The electrical conductivity is very sensitive for the interconnection of high conductive phase of ferro-periclase in mantle composition. Based on the results of the electrical conductivity measurements by means of high pressure experiments, ferro-periclase forms the interconnected layer in the aggregates of silicate perovskite and ferro-periclase. The interconnected microstructure can be maintained for a geological time scale (~10 My) under the condition of the cold subducted slab (~800 °C), indicating the low viscous slab even at lower temperature than the surrounding mantle, because of lower viscosity of ferro-periclase than that of silicate perovskite. The low viscous slab may be prevented the penetration into the deeper lower mantle against the high viscous region at ~1000 km depth, named "viscosity hill" (9, 10), and therefore causes the stagnation at this depth observed in seismic tomography.

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## Synchrotron radiation study on rheological properties of minerals during high-pressure transformations

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Phase transformations of minerals have an important role on the rheology of subducting slabs. Flow properties and the dominant mechanism of deformation are possibly affected through changes of the crystal structure, grain size and polycrystalline texture during the transformation. In the peridotite layer of subducting slabs, the olivine-spinel and the post-spinel transformations are major reactions, which are thought to be origin of the 410 and 660 km seismic discontinuity in the mantle, respectively. In this study, to investigate effects of these transformations on rheological behavior of the subducting slabs, simultaneous deformation and transformation experiments were conducted using analogue reaction systems. Flow stress and transformed fraction were quantitatively obtained by in-situ X-ray observations during the constant strain rate deformation using deformation-DIA (D-DIA) apparatus.

High-pressure transformations of fayalite ( $\text{Fe}_2\text{SiO}_4$ ) and albite ( $\text{NaAlSi}_3\text{O}_8$ ) were used for the analogue of the olivine-spinel and the post-spinel transformation in  $(\text{Mg,Fe})_2\text{SiO}_4$ , respectively. High-pressure deformation experiments were conducted using D-DIA apparatuses at the NE-7 of PF-AR and BL04B1 of SPring-8. The plastic deformation and high-pressure transformation processes were simultaneously observed by time-resolved two-dimensional X-ray diffraction (2DXRD) measurements using monochromatic X-ray (energy 50 keV). 2DXRD patterns were used to obtain the transformed fraction and the differential stress of the sample that was estimated from the distortion of the Debye ring. Plastic strain of the sample was measured from the X-ray radiography images. The microstructure and crystallography of recovered samples were observed using a FE-SEM with an EBSD system.

The olivine-spinel transformation experiments in polycrystalline fayalite were conducted at 973 and 1173 K under quasi-hydrostatic and non-hydrostatic (the samples deformed with the strain rate of  $5 \times 10^{-5} \text{ s}^{-1}$ ) conditions. Overpressure needed for the transformation under non-hydrostatic condition at 973 and 1173 K (2.9 and 0.6 GPa) was smaller than under quasi-hydrostatic condition (3.8 and 1.5 GPa). In deformed sample, creep curves indicated that the sample became harder with increase of the spinel fraction. This observation suggests that the olivine-spinel transformation under relatively small overpressure and high-temperature condition would not cause the slab weakening.

Both the post-spinel transformation and the albite decomposition are eutectoid reactions with having an alternating fine lamellar structure. To investigate the creep behavior during eutectoid transformation, two kinds of polycrystalline starting materials, parental albite and decomposed jadeite + quartz aggregate, were prepared. High-pressure deformation experiments were conducted at 2-4 GPa, 873-1073 K and the strain rate of  $10^{-6}$ - $10^{-5} \text{ s}^{-1}$ . The microstructures of recovered samples as well as the flow and kinetic data suggest sequential variation of the creep mechanism from dislocation creep of the transformed eutectoid colony followed by the grain-size sensitive creep in the degenerated eutectoid structure. This study demonstrated that the creep behavior during the eutectoid transformation involves various processes than previously thought. The slabs may not be weakened promptly after entering into the lower mantle when the size of eutectoid colony is enough large, and keep their strength (or harden) over a period of time depending on the degeneration kinetics of the colony.

## Microstructural development under diffusion creep of olivine-melt system

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Lattice preferred orientation(LPO) in natural rocks is considered to be generated in deformation of the rocks. For example, seismic velocity anisotropy in the asthenosphere is considered to be the result of LPO which is developed under shear deformation of the mantle. Partial melt is likely to be present in the asthenosphere so it is important to understand the rheology and microstructure of the partial molten mantle rock, in order to know the actual state of the asthenosphere. A number of deformation experiments in olivine-melt system have been conducted demonstrating the formation of variety of LPO patterns(Zimmerman et al. 1999, Holtzman et al. 2003).

In this study, we conducted deformation experiments on olivine-melt system to examine the relationship between their flow characteristic and microstructure. We synthesized bulk samples of Fe-free olivine + anorthitic melt(10~20vol%). We used Instron type deformation testing machine equipped with a vertical furnace, and deformed the samples at the strain rate of  $10e^{-5}$ ~ $10e^{-4}$ /s under the temperature 1260 degrees(Celsius) and atmospheric pressure.

Stress and strain rate relationships were obtained from the stress and displacement rate of the samples during the deformation. The stress exponent approximately equals 1, indicating that the samples deformed under diffusion creep. We also measured the orientation of olivine crystals in the deformed samples by EBSD under FE-SEM, finding alignments of b-axes of olivine to the compression direction. Melt distribution changed by the deformation. Large melt pockets(>100 micrometer in diameter) elongated normal to the direction of compression whereas small melt pockets at grain junction align parallel to the compression direction. We consider that the elongated melt pockets were formed by the melt deformation following the bulk sample strain whereas the intergranular melt alignments were generated by an infiltration of the melt to olivine grains which were separated to the tensile direction. LPO of the olivine might be resulted from alignments of short axes of olivine grains, which are parallel to the b-axis of the crystal to the direction of compression.

Keywords: diffusion creep, olivine-melt system, LPO

## Development of olivine LPO under diffusion creep

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Lattice preferred orientation (LPO) of olivine is considered to be a main cause of anisotropic mantle especially of its elasticity, which can tell its dynamic state such as flow direction of the mantle. Olivine LPO is considered to be a consequence of dislocation creep process in the mantle so that intense investigations of the easy slip systems of the mineral under various geological conditions such as temperature, pressure and water fugacity have been conducted. Here we show that synthetic polycrystalline forsterite (+ Ca-bearing enstatite) aggregates demonstrate strong LPO after deformation under diffusion creep where large contribution of grain boundary sliding (GBS) to the sample strain. Combining the LPO patterns developed under tensile and compression tests, our observations correspond to A- and E-type fabrics, previously identified in experimental and natural samples, depending on temperature conditions without the effect of water and pressure on intragranular slip systems. Development of LPO under GBS creep strongly correlates the shape of grains which is crystallographically controlled. Such crystal shape provides grain boundary planes corresponding to crystallographic planes so that GBS and its consequence of grain rotation proceed at specific direction of the crystal resulting in an alignment of specific crystallographic axis to the flow direction forming LPO. Our finding adds new interpretations of the mechanism to form mantle anisotropy.

Keywords: olivine, LPO, diffusion creep

## Cooperation of dislocation gliding and grain boundary sliding in hydrous peridotite

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Deformational behavior of olivine in mantle wedge strongly affects subduction dynamics and geological processes at convergent margins (mantle flow, volcanism, earthquakes and orogeny). Many experimental works are addressed on the deformation under wet conditions. However, there are some difficulties in extrapolating the results to the conditions in subduction zones, especially for temperature conditions. A recent experimental study showed that a mechanism of grain boundary sliding (GBS) can be prevailing in polycrystalline olivine with interstitial hydrous melt, suggesting that a superplastic flow due to GBS of olivine possibly affects on a coupling between mantle wedge and subducting slab. Our EBSD analyses of natural hydrous peridotite revealed transitional structures due to cooperation of GBS and dislocation gliding. Here we present results of microstructural analyses that constrain a GBS flow law under wet conditions.

We used three dunite samples with different proportions of olivine porphyroclasts (SGB) (about 20, 40 and 60%), representing the various degrees of recrystallization. They were exposed at the Gongen outcrop that belongs to the Higashi-akaishi ultramafic body in the Sanbagawa metamorphic belt. All the samples include mm-sized planar grains of amphibole that define the strain geometry of samples. Strain shadows of chlorite and phlogopite around amphibole porphyroclasts indicate water-rich conditions during deformation. Geothermometry for fine Opx and Cpx in the matrix suggests deformational temperature of 700-770 oC. Pressures are inferred to be in a range of 1-2 GPa.

Using well indexed EBSD maps for these samples, olivine grains are separated into two fractions with and without significant internal misorientation (MO): we call them as wSGB and w/oSGB grains respectively. Then, we analyzed grain size, axial ratio and crystallographic preferred orientation (CPO) for each fraction and internal MOs of representative porphyroclasts using MTEX and HKL software.

The olivine CPO of wSGB is stronger in a more recrystallized sample and shows a weak concentration of a-axis parallel to amphibole lineation. On the other hand, the CPO of w/oSGB is weak and independent of the extent of recrystallization. Grains with SGB are elongated (aspect ratio = 2.0) whereas those without SGB are close to equant. Frequency distributions of grain sizes for wSGB and w/oSGB can be approximated as distinctive log-normal distributions and the mean values are  $10^{2.3}$  micron for wSGB and  $10^{2.0}$  micron for w/oSGB.

These observations suggest that larger grains are dominated by intracrystalline deformation with a dislocation mechanism. Sub-grain structures in porphyroclasts are consistent with [100] slip in {0kl} planes. On the other hand, smaller grains have been deformed under a mechanism without CPO strengthening. Almost equant shapes of olivine grains and high frequency of quadruple junctions of grain boundaries are consistent with GBS mechanisms rather than diffusion creep. Recrystallizing porphyroclasts is associated with nucleation of neoblasts in support of grain boundary migration, implying that diffusional processes have accommodated displacements among grains.

We interpreted that the microstructures of the hydrous dunite record a mechanism transition from dislocation gliding to diffusion-accommodated GBS due to grain size reduction. The critical grain size for mechanism transition lies between representative grain sizes of wSGB (200 micron) and w/oSGB (120 micron). Differential stress is estimated as 30-130 MPa based on recrystallized grain size piezometers. These values are, however, inconsistent with an extrapolation of an experimentally determined GBS flow law. This indicates that some refinement of the flow law parameters are required in order to discuss deformational mechanisms in cold thermal conditions expected for subduction zones.

Keywords: olivine, microstructure, rheology, grain boundary sliding, hydrous peridotite, subduction zone

## Microstructural observation on naturally deformed olivine in peridotite

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Deformation microstructures of mantle derived olivine, such as slip system and density of dislocation and recrystallized grain size are strongly influenced by deformation conditions in the mantle. Slip system of dislocation in deformed olivine changes with temperature, water content and stress (Carter and Ave Lallemand, 1970; Jung et al., 2006). And dislocation density and recrystallized grain size also change with stress, which can be used as a geopiezometer (Kohlstedt and Goetze, 1974; Jung and Karato 2001). In this study, we try to estimate the deformation condition and history recorded in the peridotite xenoliths using these microstructures. The samples were collected from 1) Takashima in Saga Pref., Japan 2) Kurose in Fukuoka Pref., Japan 3) Megata in Akita Pref., Japan and 4) Salt Lake in Hawaii, USA.

The slip systems are determined from the patterns of Lattice-Preferred Orientation (LPO), and directly observation of dislocations using Weak-Beam Dark-Field (WBDF) method of TEM. We use SEM with EBSD system and TEM for measurement of LPO, and the dislocation density, respectively. The recrystallized grain size is measured mainly under the optical microscope.

We now obtain the following results. 1) The active slip systems suggested by LPO of Kurose, Megata and Salt Lake are same, which is (010)[100]-A-type, {0kl}[100]-D-type or (001)[100]-E-type. The development of LPO of Takashima is poor, but it probably suggests that it is (010)[001]-B-type or (100)[001]-C. 2) The active slip systems determined by WBDF method are (010)[100]-A-type for Takashima and Salt Lake, and (001)[100]-E-type for Kurose and Megata. These results, except for Takashima, confirm those of LPO measurements. On the basis of the experimental result, we infer that the peridotite xenoliths from Takashima and Salt Lake, and Kurose and Megata were deformed under "low stress and low water content" and "low stress and high water content" conditions, respectively. 3) The stresses estimated from dislocation densities are bigger than those from recrystallized grain size in Megata, Takashima and Salt Lake. It is known that the dislocation density remains constant just after the stress applies, while the recrystallized grain size becomes constant after the strain reaches large. Therefore, these results suggest that the deformation stage of these peridotites were under transient creep, in which the additional stress applied after the steady state creep. On the other hand, the both stresses estimated from dislocation density and recrystallized grain size in Kurose is same, which suggests that these microstructures were created under steady state creep.

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Keywords: Olivine, Peridotite, Slip system, Dislocation, Weak-Beam Dark-Field method

## Role of dynamic recrystallization in lattice preferred orientation of quartz rocks

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From the microstructural analysis of plastically deformed rocks, the c-axis lattice preferred orientation (LPO) of quartz has been utilized to infer deformation conditions: sense of shear, deformation temperature, and/or water content. The c-axis LPO patterns change from type I crossed-girdle at lower temperature to point maxima at Y axis of strain ellipsoid (Y max LPO) at intermediate temperature, to point maximum at X axis (X max LPO) at higher temperature. The change in LPO patterns is known to reflect the change in the dominant slip systems of quartz from basal  $\langle a \rangle$  and rhomb  $\langle a \rangle$  slip, to mainly prism  $\langle a \rangle$  slip, to prism [c] slip with deformation temperature (Stipp et al., 2002). Most naturally deformed rocks with a strong LPO are dynamically recrystallized. It is not clear whether the LPO patterns are controlled only by the dominant slip systems or by dynamic recrystallization processes such as grain boundary migration and subgrain rotation. Recent experiments have clarified the formation of Y max LPO patterns with increasing strains and degree of dynamic recrystallization where grain boundary migration is the dominant mechanisms of dynamic recrystallization (Heilbronner and Tullis, 2006; Muto et al., 2011). However, the effect of other mechanisms occurring under lower temperature conditions has been not clear yet. In order to clarify how dynamic recrystallization affects the LPO development under lower temperature than previous experiments, we conducted general shear experiments in a Griggs apparatus using single crystals of synthetic quartz. We utilized three different initial orientations to activate three dominant slip systems of quartz: basal  $\langle a \rangle$ , prism  $\langle a \rangle$  and prism [c] slip. The c-axes of samples with initial orientations for basal  $\langle a \rangle$  slip system and prism [c] slip system progressively rotated with the sense of shear with strains. The amount of the rotation at a given strain is larger in the samples with prism [c] initial orientation than those of basal  $\langle a \rangle$  initial orientation, implying the rapid consumption of the harder slip system. The c-axis of samples in the basal  $\langle a \rangle$  initial orientation rotated 90 degrees to prism [c] orientation at gamma of 2 and further to the orientation suitable for basal  $\langle a \rangle$  slip. The samples of the prism [c] initial orientation rotated 90 degrees to orientation suitable for basal a slip and completely recrystallized at gamma ~ 6. Recrystallized grains show symmetric broad single maximum at the Z axis of the strain ellipsoid, consistent with the c-axis LPO of recrystallized grains (Heilbronner and Tullis, 2002) where bulging is the dominant recrystallization mechanism. On the other hand, the samples with prism  $\langle a \rangle$  initial orientations did not show any recrystallization up to strains as high as gamma of 7 and kept its c-axis orientation located at the Y axis of the strain ellipsoid. This indicates that grains in basal  $\langle a \rangle$  and prism [c] initial orientations were recrystallized to activate easy basal  $\langle a \rangle$  slip with progressive deformation. On the other hand, the grains with prism  $\langle a \rangle$  initial orientation do not change their c-axis orientations with progressive shear. Therefore, the activation of easy basal  $\langle a \rangle$  slip and additional prism  $\langle a \rangle$  and/or rhomb  $\langle a \rangle$  slip to satisfy von Mises criterion results in the development of type-I crossed or inclined single girdle depending on the deformation geometry. With increasing deformation temperature to higher greenschist to amphibole facies conditions where rapid grain boundary migration can occur, grains oriented for weak prism  $\langle a \rangle$  slip can grow at the expense of grains in other orientations, results in development of Y max LPO with dynamic recrystallization (Heilbronner and Tullis, 2006; Muto et al., 2011). Therefore, the LPO transition observed in natural deformed rocks from type I crossed or single girdle to Y max LPO may basically reflect the change in dominant mechanisms of dynamic recrystallization with temperature.

Keywords: Lattice preferred orientation, Quartz, Dynamic recrystallization, Rheology of crust

## Al, Si interdiffusion in majoritic garnet and the dislocation microstructures.

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Plastic deformation by dislocations and atomic diffusion by vacancies of minerals at high pressures are important for the rheology of the Earth's mantle. Because those processes are controlled by moving of two agents (line and point defects) in deformations at high temperature, the post-mortem examination by analytical transmission electron microscope is indispensable for evaluating those agents (carriers). Majoritic garnet (MajGt) and magnesium silicate perovskite (MgPv) are major constituents in the mantle transition zone and the lower mantle, respectively. Diffusivity differences of the slowest species between these mantle minerals are very important to understand the changes of the nature of chemical heterogeneity, viscosity through those creep law, and other various transport properties across the upper and lower mantle boundary.

Here I report an Al + Al = Si + Mg interdiffusion between MajGt and pyrope garnet. The diffusion couples using a multi-anvil press are pre-synthetic Mg<sub>3</sub>Al<sub>2</sub>Si<sub>3</sub>O<sub>12</sub> pyrope and majoritic garnet. The annealing condition is at 18.5 GPa and 1750-1950 degree Celsius for 120-300 minutes, corresponding to a MajGt-single phase region in the binary system MgSiO<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub>. Diffusion profiles of the recovered sample were examined with electron probe microanalyser (EPMA), scanning electron microscopes (SEM) and scanning transmission electron microscopes (STEM) equipped with an energy dispersive X-ray spectrometer (EDXS). Dislocation microstructures were also examined in weak-beam dark-field images using the thickness-contour fringe method (Ishida et al., 1980; Miyajima and Walte, 2009).

MajGt displays <100> and 1/2 <111> free dislocations and subgrain textures consisting of a dislocation array, suggesting that climb of dislocations was occurred during diffusion annealing. The obtained Al + Al = Mg + Si interdiffusion coefficient ( $D_{Al}$ ) of MajGt at 18.5 GPa and 1750 degree Celsius is  $6.2(4) \times 10^{-19}$  (m<sup>2</sup>/s), which is comparable with those of Mg and Si self-diffusion coefficients in MgPv under lower mantle conditions (Xu et al., 2011). However, the  $D_{Al}$  is significantly higher than those of in previous studies in majoritic garnets at temperatures less than 1750 degree Celsius (e.g., Nishi et al. 2013). The preliminary obtained activation energy in this study is much higher in the temperature from 1750 to 1950 degree Celsius, where is likely to be the intrinsic regime in the interdiffusion. Comparisons with Al, Si interdiffusion in Fe-bearing majoritic garnets are given to highlight the effect of impurities and temperature on those diffusion rates. Considerations for further diffusion experiments in MajGt and aluminous MgPv are discussed toward the rheology from the transition zone from the lower mantle. I thank the generous support from BGI colleagues for commissioning of this study.

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Keywords: majoritic garnet, Al, Si interdiffusion, analytical transmission electron microscope, weak-beam dark-field image, dislocation microstructures

## Grain-growth experiments of olivine using 2D monochromatic X-ray diffraction pattern.

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Rheology is one of the most important mineral properties which plays a major role in controlling dynamic processes in the Earth's mantle. Olivine is the most abundant mineral in the upper mantle (60vol% in pyrolite mantle). Rheology in the upper mantle is dominated by this mineral. If diffusion creep is the most dominant deformation mechanism, the rheology of an aggregate is a function of the grain size of the constituent minerals. Thus, knowledge of grain size of olivine is important for understanding the rheology of the upper mantle.

The grain size of rock is controlled by several mechanisms (e.g. grain-growth, dynamic recrystallization and recrystallization by phase transformation). Grain-growth is one of the most important processes in controlling the grain size of the rock by which grain size is enlarged. Grain-growth experiments of olivine were conducted by some workers (e.g. Karato 1989, Ohuchi and Nakamura 2006). However grain-growth exponent, which is a parameter of time dependence, is controversial between previous studies.

Thus, in this study, we have investigated grain-growth kinetics in the olivine single phase system at high pressure and temperature (1373 - 1573 K, 2.3 - 10.5 GPa) using a in situ monochromatic X-ray diffraction and Kawai-type multi-anvil apparatus (SPEED-1500) installed at the synchrotron beam line, BL04B1, in the SPring-8 at the Japan Synchrotron Radiation Research Institute. The San Carlos olivine powder was used as starting material. The grain size was estimated by the relationship between the number of diffraction spot and the number of grains per radiated volume reported by Hirsch (1955).

The grain-growth kinetics of olivine is described by  $G^n - G_0^n = k_0 \exp(-(E + PV)/RT)t$  where  $G$  is the average grain size at annealing time  $t$ ;  $G_0$ , the initial average grain size;  $k_0$  is the pre-exponential constant,  $E$  is activation energy,  $P$  is pressure,  $V$  is activation volume,  $R$  is the gas constant and  $T$  is absolute temperature, with  $n = 2.5 \pm 0.2$ ,  $\log_{10} k_0 = -9.2 \pm 2.6 \text{ m}^{-2.5}/\text{s}$ ,  $E = 184 \pm 10 \text{ kJ/mol}$ ,  $V = 0.4 \pm 0.2 \text{ cm}^3/\text{mol}$ . The activation energy of grain-growth is similar to that of grain boundary migration (Toriumi, 1982). Thus, in terms activation energy and grain-growth exponent, in this study, grain-growth could be caused by grain boundary migration in the single phase system.

Keywords: olivine, grain-growth, in-situ experiments, upper mantle

## Texture and strain analyses using 2-dimensional X-ray diffraction patterns under DAC experiments

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Angle dispersive X-ray diffraction experiments using area detectors (CCD and CMOS cameras and image-plate recorders) provide wide opportunity for the determination of lattice preferred crystallite orientation (LPO) and lattice strain under stress condition in polycrystalline materials. LPO is reflected in circumferential oscillations along Debye rings, while the effect of lattice strain appears in elliptic distortions of the each ring and a deviation of the original crystallographic geometry among rings. These are substantial factors of bulk physical properties in polycrystalline materials, including seismic velocity, thermal/electric conductivity and so on. Diamond anvil cell (DAC) is the only technique that can create at extreme pressures corresponding to the Earth's core, and it simultaneously involves non-hydrostatic, uniaxial stress in the sample. Although such non-hydrostatic effects under DAC experiments has been reported many previous studies, in many cases the quantitative treatments have not yet been developed into a standard technique.

In order to examine quantitative stress conditions under DAC experiments, high pressure experiments were carried out in a symmetrical DAC in the present study. Two stating materials, Al<sub>2</sub>O<sub>3</sub> (~1um in diameter) and MgO (<0.1 um), were used as stating materials, and no pressure media were loaded. Each runs were performed at the pressures from 0 GPa to 70 GPa by 10 GPa step under room temperatures, and synchrotron X-ray diffraction patterns were collected using a flat imag-plate at BL10XU at SPring-8. A software code was also developed by the author, which simulates a two-dimensional diffraction pattern based on given experimental parameters and (poly)crystalline properties. A fitting procedure was also incorporated into the code, where the orientation distribution and stress condition were iteratively modified according to a residual value of the simulated/observed patterns.

In runs of Al<sub>2</sub>O<sub>3</sub> experiments, the diffraction peaks became distinctly broad and asymmetric shapes with increasing pressures, whereas the scattering angles (2theta) were apparently almost constant. This means that lattice compression involved by pressures was cancelled out by deviatoric stress. Nonetheless, the stress conditions could be derived mainly from the shapes of the peaks using the fitting procedure; e.g. at the highest pressure condition in the present study, maximum and minimum principal stresses could be estimated as 73GPa (parallel to compression axis) and 25 GPa, respectively, corresponding to the deviatoric stress of 50 GPa. The maximum principal stress was consistent with the estimated pressure by the diamond Raman pressure scale. On the other hand, MgO experiments maintained pseudo-hydrostatic conditions with small deviatoric stress only up to ~1GPa under all performed pressures. A whole pattern fitting method such as the code developed in the present study may help us understand the stress conditions under DAC experiments

Keywords: powder X-ray diffraction, diamond anvil cell, area detector, polycrystalline material, lattice preferred orientation, lattice strain

## Experimental study of bulk viscosity of partially molten rock analogue

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Viscosities of partially molten rock change significantly due to melt fraction. However its quantitative effects have not been well constrained theoretically nor experimentally. Deformation of partially molten rock is controlled by two independent viscosities: shear viscosity for shear deformation and bulk viscosity for compaction/decompaction. Bulk viscosity and its ratio to shear viscosity,  $h_b/h_s$ , play an important role in melt segregation dynamics (Katz, 2008). Most numerical studies have used the theoretically predicted value of  $h_b/h_s = \sim f^{-1}$ , where  $f$  is the melt fraction. However, Takei and Holtzman (2009a) theoretically obtained a constant value of  $h_b/h_s$  by taking into account a diffusion creep mechanism. The discrepancy between two models is significant at small melt fractions. There has not been experimentally determined value of  $h_b/h_s$  because very few experimental studies have been done about bulk viscosity although shear viscosity has been measured extensively. Therefore, the purpose of this study is to measure a pair of the bulk and shear viscosities for the same sample. As the first step of the experimental examination, we measured bulk viscosity experimentally as a function of melt fraction using a partially molten rock analogue.

Samples were polycrystalline aggregates of borneol-diphenylamine binary with eutectic temperature of 316K, which has a quite similar equilibrium microstructure to olivine + basalt system (Takei, 2000). Initial melt fraction can be controlled precisely by the concentration of diphenylamine because of its simple eutectic reaction. Before deformation experiments, samples were annealed at 320K for  $\sim 100$  hours in a sealed capsule to make those grain size large enough ( $\sim 0.030$  mm), resulted in negligible grain growth during the successive deformation tests at the same temperature.

To measure the bulk viscosity, we carried out compaction experiments in which melt was squeezed from the partially molten sample under the diffusion creep regime. A cylindrical sample contacted with a porous metal at the top end was compacted uniaxially in a rigid sleeve (horizontal strain = 0, vertical strain < 0). Melt can flow out into the porous metal until its fraction becomes nearly zero. Evolution of melt fraction in the sample was calculated from the sample length measured with digital gauge. Apparent viscosity is calculated as a function of melt fraction from an instantaneous strain rate and a constant stress. Precise measurements of melt fractions at very small amounts of melt ( $f < 1\%$ ) is crucial to test the predictions of models. Data obtained so far show the viscosity is proportional to  $\exp(-af)$  with  $a = \sim 30$  at  $f > 3\%$ , which is quite consistent with the olivine + melt systems (Renner et al., 2003). At  $f < 3\%$ , deviation of the viscosity from the exponential curve occurs, suggesting the possible effects of permeability and change of rate limiting process of the volumetric creep (Takei & Holtzman, 2009b).

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Keywords: viscosity, bulk viscosity, partial melt, compaction

## Simultaneous measurements of creep strength and electron conductivity of polycrystalline olivine

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It is important to know the value of activation energy with a small error range for creep of geomaterials when we apply an experimentally obtained flow law to the geological conditions. This study reports the result of uniaxial compression experiments of polycrystalline forsterite at atmospheric pressure and high temperature in order to analyze the change of flow stress and deformation mechanism with temperature. In addition, we also report the result of the measurements of electrical conductivity using an impedance technique. Creep rate is controlled by diffusion process of the slowest ionic species in the crystal whereas electrical conductivity is controlled by the process of the fastest one. We expect to understand the detail of diffusion mechanism, which controls the creep rate, with the help of electrical conductivity. Such studies will allow us to predict the rate controlling process of flow in the Earth interior.

The polycrystalline sample is composed of forsterite ( $Mg_2SiO_4$ ) and enstatite ( $MgSiO_3$ ), where their volume fractions are 90% and 10%, respectively. Instron-type testing machine equipped with tubular furnace was used for compression creep experiments. Prior to the compression experiment, grain size in the sample was saturated by heating at  $1360^\circ C$  for 24 hours to inhibit grain growth during the experiment. We measured electrical conductivity over range of  $1360 - 1200^\circ C$  at constant normal stresses of 10 and 20 MPa and under decreasing temperature with the rates of  $0.11^\circ C/min$ ,  $0.03^\circ C/min$  and  $0.02^\circ C/min$  at high, mid and low temperatures, respectively. Such slow cooling rates can provide enough strain of the sample at each temperature so that the relationship between the stress and strain rate at all temperatures is obtained. We also measured impedance of the sample at every  $10^\circ C$  from  $1360^\circ C$  to  $1200^\circ C$ . This impedance is obtained from the relationship between voltage and current, which are measured when voltage of 2V was applied to electrodes of SiC. After the experiment, the sample microstructure was analyzed by scanning electron microscopy to know sample grain size. Viscosity - temperature relationship was analyzed by Arrhenius plots of stress divided by strain rate obtaining the activation energy of  $706 \pm 1 kJ/mol$ . This result shows that the diffusion mechanism during this experiment did not change indicating that our obtained flow law can be applied to the Earth's interior where lower temperature condition than that in the experiments. The result of impedance measurement showed that electrical conductivity systematically decreased with decreasing temperature.

Keywords: olivine, creep, polycrystal, activation energy, electrical conductivity, impedance

## Element migration via fluids with progress of fracturing along the Median Tectonic Line, Mie Prefecture, southwest Jap

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Along the Median Tectonic Line (MTL) formed in the Cretaceous period, which separates the Ryoke belt from the Sanbagawa belt, localized deformation which resulted in the formation of mylonite to cataclasite occurred in the Ryoke belt with decreasing temperature. The borehole at the Iitaka Observatory, Mie Prefecture, southwest Japan, drilled through the upper cataclasite zone of Sanbagawa belt, penetrates the MTL at the depth of 473.9 m. In this study, for samples from the depths of c. 450-470 m that originated from tonalite, we investigated the major element migration based on the whole rock composition determined by X-ray fluorescence analysis. Further, we showed changes of the mineral assemblage resulting from element migration with a point counting method aided by image analyses. All analyzed rocks once became mylonite by plastic deformation, which were later fractured to various degree with decreasing temperature. Based on optical thin section observations, we classified the cataclasite samples into four groups: relatively undeformed (UN), weakly (W), moderately (M) and strongly (S) deformed rocks based on the degree of cataclasis. For the correct mode analyses of mineral composition in the deformed rock samples, we classified plagioclase into three groups based on the degree of muscovitization: non-, moderately and the strongly altered plagioclase. Further, we estimated an area of mica fraction in a grain of feldspar by analyzing images of elements (i.e. K) obtained by EDS mapping. The mineral compositions of gauges of strongly deformed rocks were estimated by analyzing images of element mapping of EDS. From these analyses, we found that there are feldspar, quartz and hornblende as fragments, and are chlorite, calcite and titanite as precipitated minerals. To analyze element migration with the increasing degree of fracturing, we determined the changes in the whole rock major elements in deformed samples using isocon plots. In this study, we treated Al as an immovable element. We calculated the volume change of deformed rock samples as  $V=[(1/S)-1] * 100$ , assuming no density change during deformation, where S is the slope of the line connecting the origin of isocon plot and an immovable element. Further, we calculated the change of elements with the increasing degree of fracturing (coefficient of variation of element) from the following formula,  $(El_f/Al_f)/(El_h/Al_h)$ , where El is any element, Al is an immovable element. Subscripts f and h indicate a fault rock and an undeformed host rock. We analyzed these for the following three pairs, which showed the volume changes of +24 % for W vs UN, -26 % for M vs W, and -19 % for S vs W, respectively. In the cataclasite samples, since the change of SiO<sub>2</sub> is the largest, the volume increase from UN to W rocks was perhaps caused by deposition of quartz. With the increasing degree of fracturing from UN to W rocks, Si and Na increased, because fluids, which included Si and Na as solutes released by feldspar-to-mica reaction, invaded into the pore spaces created by fracturing and deposited there. The volume decrease from W to M or S rocks was caused by dissolution of quartz and subsequent fluid moving out from the fault rocks by strong compression, accompanied by the decrease of Si and Na contained in the fluids. On the other hand, Fe, Mg, Ca and Ti increased during the further fracturing. In the gauge of M or S rocks, although it seems that those mineral phases consisting of Fe, Mg, Ca and Ti deposited from the fluids despite strong compression, the reason why the volume of these elements in fact increased from W to M or S rocks remains to be investigated.

Keywords: element migration, cataclasite, isocon diagram

## Mechanism of cataclasite occurring in Hiraodai Karst region

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Geofluids are considered to have important effect on earthquake generation and volcanic activities related to the subducting slab. Different aspects of geofluids have become the highlights of recent research. In the present study, we focus on the exposed cataclastic layer and have tried to unravel the mechanism of the formation of such disintegration of rocks due to fluid activity. The studied cataclastic layer occurs in Hiraodai karst region, where limestones were thermally altered to marble by contact metamorphism of the adjacent granodiorite pluton. In the vicinity of the cataclasite layer, calcite crystals have selectively high amount of fluid inclusions. Moreover, many of the disintegrated gravels inside the cataclasite layer are rounded. All these facts are strongly pointing towards participation of fluids during the formation of the studied cataclasite layer. Here, we present the observations of deformation microstructure of calcite and geochemical characterization of fluid inclusions in it, which lead to the understanding of origin of fluid producing the cataclasite layer as well as the process of disintegration of the studied marble. The data reveals that (1) fluid-induced differential stress produces cataclasite layer in the marble body, (2) the fine-grained matrix of the cataclasite layer is formed by calcite which were crystallized in the presence of fluid, and (3) this cataclasite-producing fluid is possibly cogenetic to the magma of adjacent the granodiorite.

Keywords: Cataclasite, Geofluid, Hydrofracturing, Sr isotope, Fluid inclusion

## Amorphization of clay minerals and crystallinity

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FWHM of the basal peak of clay minerals is used as an index of crystallinity. On the other hand, clay minerals are easily transformed into amorphous material by mechanical grinding. FWHM value might be increased during amorphization.

We conducted pulverization experiments of some clay minerals (kaolinite, sericite, saponite) using planetary ball mill. The FWHM values slightly increased by pulverization, however, the difference were not so large. SEM observation showed that the shape of the grains was not so much changed during pulverization even after the basal peak diminished. We consider the process of amorphization by these observations.

Keywords: Clay minelas, Amorphization, Crystallinity

## Effect of pore pressure and stress paths on frictional properties of talc and serpentinite under high normal stress

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Pore fluid pressure is a critical parameter governing the overall mechanical strength of plate boundary faults. Recent geophysical observations have suggested the importance of fluids in seismogenic processes. Previous works on rock mechanics have suggested that the yield strength of rocks is governed by effective stresses  $S_e = S - aP_p$ , where  $S$  is total stress,  $P_p$  is pore fluid pressure, and  $a$  is a factor between 0 and 1. The observations in the brittle regime are well accounted for by  $a = 1$  [1]. However, it is not well documented how pore fluid pressure influences frictional properties of faults at the brittle-ductile transition zone. Ductile deformation might play important roles in contacts of topographies of fault surfaces, or asperities, at the brittle-ductile transition zone, and therefore there is a possibility that  $a$  in the effective stress law may not be 1 and/or pore pressure distributions on the slip surfaces may be inhomogeneous and time-dependent due to reduction of permeability between the slip surfaces. It is also expected that shear stress may depend highly on stress history in the brittle-ductile transition zone. It is generally difficult to conduct laboratory friction experiments at high pressures and temperatures that are comparable to the middle to lower crust and mantle. To overcome the limitation of experimental conditions, we conducted friction experiments by using talc and serpentinite (antigorite) as an analogue material, which shows brittle-ductile transitional behaviors at relatively low pressures and temperatures. In addition, investigating frictional properties of these rocks under high stress is important because these rocks receive attention as a material giving important influences on fault mechanics. Cylindrical samples of talc (Gvangjsih, China) and serpentinite (Nagasaki), 20mm in diameter, were cut at an angle of 30° to the sample axis. The surfaces were ground with carborundum (#400 and #80 for talc and serpentine specimens, respectively). A small hole (3mm in diameter) through the center of each piece ensured adequate communication of the water between the pre-cut surfaces with the rest of the pore pressure system. The specimen was loaded by a triaxial apparatus and sheared under an axial displacement rate of 1  $\mu\text{m/s}$ . We used water as a pore fluid. All measurements were performed under conditions of room temperature. Experiments were conducted under several paths of confining pressure and pore pressure. The results indicate a possibility that the shear stresses of these rocks under high normal stress may not be able to be simply explained by an effective stress law with  $a = 1$ , and stress paths affect the shear stress.

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References: [1] M.S. Paterson and T.-F. Wong, Experimental rock deformation - the brittle field, 2nd ed., 2005.

Keywords: frictional property, talc, serpentinite, pore pressure, laboratory experiment

## Synthesis of highly dense and fine-grained lower crustal minerals by vacuum sintering technique

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It is important to fabricate polycrystalline rock-forming minerals which have controlled crystallographic orientation, grain size, sample shape, mineral composition, chemistry (e.g., trace elements), and phases (including melt) for investigating the physical and chemical properties of the Earth' interior by room experiments. The vacuum sintering method at ambient pressure has been applied. We developed synthesis method of a wide variety of polycrystalline minerals, including single phase aggregates of anorthite ( $\text{CaAl}_2\text{Si}_2\text{O}_8$ ) and, two phase composite of anorthite + diopside ( $\text{CaMgSi}_2\text{O}_6$ ), anorthite + quartz ( $\text{SiO}_2$ ) with homogeneous microstructure, which are good analogues for lower crustal composites.

Keywords: polycrystal, lower crust, mineral

## Growth kinetics of forsterite reaction rim at high pressure

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Element diffusion is one of the most important processes which control rheology of the minerals. For example, a strain rate in the diffusion creep is generally rate-limited by slowest diffusing element among the major elements in the mineral. From previous studies, Si is known as the slowest diffusing element among the major components in olivine, and O is the fastest below pressure of 1 GPa at wide range of grain size (e.g. Hirth and Kohlstedt, 2003). However, effect of pressure on the relative diffusion-rate among component species of olivine is not well known. In order to assess major element diffusion in olivine, we have carried out forsterite reaction rim growth experiments under high pressure.

The forsterite rim was formed by the chemical reaction,  $\text{MgO}$  (periclase) +  $\text{MgSiO}_3$  (enstatite)  $\Rightarrow$   $\text{Mg}_2\text{SiO}_4$  (forsterite). The starting materials were single crystal periclase and powder or sintered polycrystal enstatite. The forsterite rim width and grain size lead us to quantify a diffusion coefficient of the rate-limiting species of the forsterite rim growth. A Pt paste, which acts as a marker defining the slowest diffusing element in the forsterite major elements, was placed between periclase and enstatite. These samples were put in a welded Pt capsule to maintain dry condition. The reaction rim growth experiments were carried out by using Kawai-type multi-anvil apparatus at temperature of 1673 and 1873 K and pressure of 5.7-12.7 GPa with duration of 0-780 min. After experiments, the Pt marker position and the forsterite rim were observed using scanning electron microscope.

The Pt marker position was always found on the periclase and forsterite phase boundary. This indicates that Si is the slowest diffusing element among the major elements of forsterite at all studied conditions. Assuming the O is the fastest diffusing element in forsterite under studied P-T conditions, Mg diffusion in forsterite is judged to be the rate-limiting process in the rim growth. Gardes et al. (2011) also showed that Si is the slowest diffusing element in major elements of forsterite at 1.5 GPa based on rim growth experiments. The results of this study and Gardes et al. (2011) suggest that Si diffusion limits the strain rate of olivine in the entire upper mantle.

It is not clear whether the rate-limiting step of the forsterite rim growth is Mg lattice diffusion or Mg grain boundary diffusion solely from our results. We calculated lattice and grain boundary diffusion coefficients of Mg assuming these diffusion processes, respectively, are rate-limiting in the rim-growth. The activation volumes are determined to be  $7 \text{ cm}^3\text{mol}^{-1}$  for lattice diffusion and  $10 \text{ cm}^3\text{mol}^{-1}$  for grain boundary diffusion, respectively.

Keywords: forsterite, high pressure, rim growth, diffusion

## In-situ observation of crystallographic preferred orientation of olivine aggregates deformed in simple shear

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The characteristics of the seismic anisotropy vary depending on the types of crystallographic preferred orientation (CPO) of olivine. Therefore, the pattern of the seismic anisotropy has been interpreted by taking into account the water-induced olivine fabric transitions in recent studies (Jung and Karato, 2001). The fabric strength of olivine aggregates is also important when we evaluate the magnitude of the seismic anisotropy in the upper mantle. In the actual upper mantle, the steady-state fabric strength of olivine is expected to be achieved due to long time-scales of flows.

The dependency of the fabric strength of olivine aggregates on strain has been evaluated in only limited numbers of experimental studies (e.g., Bystricky et al., 2000). Bystricky et al. (2000) showed that total shear strains higher than 4 are needed to achieve the steady-state fabric strength of olivine (D-type fabric) at 0.3 GPa and 1473 K. However, it has been difficult to evaluate the detailed processes of the developments of fabrics because fabrics of recovered samples have been used. Recently, we have developed experimental techniques for in-situ simple-shear deformation experiments using a D-DIA apparatus. In this paper, we briefly show that our recent experimental results on in-situ observations of stress, strain, and fabric developments in olivine samples.

Simple-shear deformation experiments on olivine aggregates at pressures  $P = 1-2$  GPa, temperatures  $T = 1290-1490$  K, and shear strain rates of  $3E-4$  s<sup>-1</sup> were performed using a deformation-DIA apparatus installed at Spring-8. The MA6-6 system (Nishiyama et al., 2008) with a truncated edge length of the second-stage anvils of 5 mm was adopted for the experiments. A sectioned sample of anhydrous olivine aggregates (diameter = 1.5 mm; thickness = 300-500  $\mu$ m) was placed into a nickel capsule and then sandwiched between two alumina pistons. Shear strain (up to 5) was measured by the rotation of a platinum strain-marker, which was initially placed perpendicular to the shear direction. Differential stress, generated pressure, and CPO patterns of olivine samples were determined from two-dimensional X-ray diffraction patterns using software (IPAnalyzer, PDIndexer, and ReciPro: Seto et al., 2010; Seto, 2012). The CPO patterns of olivine in the recovered samples were also evaluated by the indexation of the electron backscattered diffraction (EBSD) patterns using a JEOL JSM-7000F at Ehime University. The CPO patterns determined from two-dimensional X-ray diffraction patterns were consistent with those obtained from the EBSD analyses.

A-type olivine fabric was developed at high temperatures (1490 K). CPO patterns showing A-type fabric were observed at strains higher than 1. The fabric strength increased with strain ( $< 3$ ). Steady-state fabric strength was achieved at shear strains about 3. Strain softening was observed in most of samples due to the developments of CPO of olivine. Developments of B-type and C-type-like fabric were observed at low temperatures ( $< 1440$  K) in relatively wet samples (about 300-400 ppm H/Si in olivine: caused by absorption of water in olivine during deformation).

It has been reported that the developments of the A-type fabric, which is the most commonly observed olivine fabric in natural peridotites (Ismail and Mainprice, 1998). The threshold shear strain of 3, which is needed for the achievement of steady-state fabric strength, corresponds to 100 Myr of mantle flow (under the assumption of a shear strain rate of  $1E-15$  s<sup>-1</sup>). Our results implies that overwriting of an olivine CPO pattern due to a change of the deformation condition requires a long time-scale (i.e., 100 Myr or longer). The seismic anisotropy observed in the upper mantle would reflect the olivine CPOs formed within 100 Myr ago.

Keywords: olivine, crystallographic preferred orientation, in-situ, simple-shear deformation

## High-pressure phase transition of olivine in shear stress condition

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High-pressure phase transition has been considered to be a possible trigger of deep focused earthquakes which were observed in subducting slab. In the deep slab condition, considerable shear stress was expected due to the sinking force by own weight. However, phase transition in a non-hydrostatic condition was not fully understood at the condition of deep transition zone. In this study, we tried to develop a new method for deformation experiment in a high-confining pressure corresponding to the lower mantle condition and investigated its effect to high pressure phase transition in olivine. High-pressure experiments were conducted using laser-heated diamond anvil cell (LHDAC). Starting materials are single crystals of natural olivine (San Carlos, USA) and pyrope garnet (Czech). They were thinned to have a wedge-shaped plate and single-sided coated with metallic iron to stabilize laser heating. Then a set of two plates are confined in a sample hole of rhenium gasket with surrounding pressure medium of sodium chloride to make a direct contact on tilt boundary to compression axis. The shear stress at the contact surface was estimated by major pressure difference between sample center and edge of the sample and was to be around 0.4-0.6 GPa. In-situ X-ray diffraction experiments to evaluate stress evolution in the sample under pressures and temperatures using a LHDAC were performed at KEK-AR-NE1A station, Tsukuba, Japan. After the sample was compressed to the nominal pressure at room temperature, it was heated to the temperature around high-pressure phase boundary. Pressures were determined using the equation of state of sodium chloride (Brown, 1998). The X-ray diffraction pattern at each condition was collected on an imaging plate. High temperatures generated by a Nd:YAG laser driven in multimode were measured based on the emission spectra from the heated area of about 50-70 microns in diameter. We observed high pressure phase of wadsleyite and/or ringwoodite in the laser-scanned area up to 23GPa and 1600K. The result of Hall-Williamson analysis (Hall, 1949) from the X-ray diffraction pattern for high pressure phase indicated a significant non-homogeneous strain or shear stress in the high pressure phase than that in single plate experiments, which suggest this method can generate appropriate stress in the sample. The quenched samples were recovered to ambient condition to make thin sections for observation by a scanning electron microscope. High-pressure phase in the heated sample was localized on the contact region between two plates. No significant textural evolution was observed in the outer rim of the same sample. This result is contrasted to the report by Kubo et al. (1998) in which nucleation starts from outer rim of the single crystal sample in a hydrostatic condition. Planar shear bands without phase transition were also observed in the low temperature less than 1000K and 34GPa. Our results indicate that shear stress promotes the transition to high pressure phase and also induces a possible shear instability in the deep slab.

Keywords: phase transition, shear stress deformation, high pressure and high temperature

## In-situ observations of reaction, plastic flow, and shear instability by using synchrotron X-rays and the AE 6-6 system

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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. Dehydration embrittlement of serpentinite (Raleigh and Paterson, 1965) is an important mechanism for the seismicity at lower pressures than  $\sim 2.2$  GPa. To understand the fault mechanisms above this pressure, there have been some acoustic emission (AE) measurements with multi-anvil apparatus to monitor shear instabilities (e.g., Dobson et al., 2002; Jung et al., 2006 and 2009; Gasc et al., 2011). However in these studies, the relationships among dehydration, plastic flow and shear instability were unclear because quantitative flow and reaction kinetics data could not be obtained simultaneously. To conduct quantitative measurements of these processes, we developed a new in-situ observation system combined with synchrotron monochromatic X-ray and AE 6-6 system (multiple acoustic emission measurement for multi-anvil 6-6 type system) using Deformation-DIA (D-DIA) apparatus. In this study, we report results of some preliminary experiments using this system.

In this system, deformation experiments with constant strain rate mode are conducted at high pressure and high temperature using a 1500-ton uniaxial press (SPEED-Mk.ii) with a D-DIA type guide block installed at BL04B1, SPring-8 (Katsura et al., 2004; Kawazoe et al., 2011). 50 keV monochromatic X-ray are used to measure two-dimensional X-ray diffraction (2D-XRD) patterns and X-ray radiography images of sample. Reaction kinetics can be monitored by time-resolved 2D-XRD measurements. Stress and strain of sample are determined by d-value variations with azimuth angle from 2D-XRD patterns and by distance of strain markers from X-ray radiography image, respectively. We developed MA 6-6 type system (Nishiyama et al., 2008) to monitor shear instabilities by AEs from maximum six piezoelectric devices positioned between first and second stage anvils. The multiple AE measurements enable us to determine characters of the seismic event such as origin time and location of seismic source, and focal mechanism.

In the present study, two kinds of experiments were performed at high pressure and room temperature using the new AE 6-6 system, where an X-ray transparent cBN anvil was used as one of the second-stage anvils in down-stream side to collect 2D-XRD patterns. One is cold compression of quartz beads (grain size  $\sim 0.1$  mm). Another is in-situ X-ray observation of constant strain rate deformation of polycrystalline antigorite cylinder cored from high-temperature serpentinite (Eigami, Nagasaki, Japan). A total of four PZT transducers were used to monitor AEs arising from the sample. AE waveforms were recorded using a four-channel 8-bit digital oscilloscope, which has a resolution of 1000-10000 point at a sampling rate of 50MHz. The AE recording was triggered when the amplitude of the signal was higher than a threshold level.

In the quartz beads experiment, the sample was pressed to 20 ton ( $\sim 2$  GPa) with monitoring AE by three AE detectors (East, West, and Bottom anvils). Many AEs were recorded during cold compression. The AE frequencies became maximum at the load of about 7 ton, and no AEs were recorded at more than 12 ton. These data suggest that the quartz beads were compacted to almost zero porosity by reaching 12 ton. Differences in arrival time from two detectors (E and W) indicate that most sources of those events were located within the sample.

Three deformation experiments of polycrystalline antigorite were conducted with a strain rate of about  $3 \times 10^{-5} \text{ s}^{-1}$  at pressures of  $\sim 0.1$ -3 GPa. We observed that the constant flow stress of  $\sim 2$  GPa reached at the sample strain of more than 5%. AE events were not recorded during the deformation stage. These results suggest that mechanical behavior of antigorite is plastic flow under this condition, which is consistent with previous studies (e.g., Escartin et al., 1997).

Keywords: acoustic emission, stress and strain, deformation-DIA, in situ X-ray observation, high pressure, antigorite

## Synthesis of wet halite rock for the study on brine morphology via physical property measurement

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Intercrystalline fluid can significantly affect rheological and transport properties of rocks. Its influences are strongly dependent on its distribution. Fluid distribution is mainly controlled by the dihedral angle between solid and liquid phase. The liquid phase is not expected to be interconnected when the dihedral angle is larger than 60 degrees. However, cryo-SEM observations (Schenk et al., 2006) and electrical impedance measurement (Watanabe, 2010) of synthetic halite rocks have indicated the coexistence of grain boundary brine with a positive dihedral angle. Similar thin fluid films might exist at grain boundaries in crustal materials. In order to understand the nature of grain boundary brine, we study the distribution of brine in halite rocks via measurements of electrical conductivity and elastic wave velocity.

A wet halite rock sample is prepared by cold-pressing (140MPa, 40 min.) of wet NaCl powder and annealing (180C and 180MPa). A sample must meet the following requirements: (1) Halite grains must be sufficiently grown to see clearly the morphological change of brine. (2) Pores must be eliminated to infer the brine distribution from elastic wave velocities. This is also required by the evaluation of water content via FTIR measurement. In order to see how long time is required for annealing to make a requisite sample, we examined halite samples annealed for 40, 80, 120, 160 hours.

The mean grain size increases by 20% as the annealing time increases from 40 to 80 hours. No significant difference can be seen in the mean grain size among samples annealed for 80, 120 and 160 hours. On the other hand, it took 150 hours for the electrical conductivity to be a stationary value. This suggests that some structural change still continues by 150 hours. Longer time of annealing diminishes porosity, and makes a sample more transparent. FTIR measurement have shown that water content is fairly uniform in the sample annealed for 160 hours. At least 150 hours is required for annealing to make a requisite sample.