

## Pattern formation of fracture phase separation and its implications for structuring process of continents and oceans

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Phase separation [1-4] is one of phase transitions in mixtures. In this phenomenon homogeneous mixtures of multi components separate into different phases depending on physical conditions such as temperature, pressure, and composition. All matters are mixtures, so that this phenomenon occurs in any matters and is important in any fields of sciences. Recently, a new phase separation was found in polymer solution. It named fracture phase separation [5] because it was characterized by brittle fracture occurred in polymer-rich phase that indicate viscoelasticity. The polymer-rich phase is torn in some regions keeping slight connections with each other. The indentation of the periphery of these regions resembles the form of cracks in brittle materials like glasses, concretes and rocks. Around these regions are filled by solvent-rich phase. These characteristics of the pattern of fracture phase separation are similar to that of the pattern of continents and oceans of the earth.

On the other hand, continents and oceans of the earth is a peculiar feature different from the surface of other planets. These surface structures of the earth have three characteristics: (1) two characteristic matters of both continental rocks composed mainly of SiO<sub>2</sub> and seawater composed mainly of H<sub>2</sub>O, (2) two regions of continents and oceans and the form of coastlines dividing the two and (3) two characteristic heights of both continents and ocean floors in solid surface of the earth. How do they occur? An interesting point is that all three characteristics are expressed by two factors. This seems to indicate that these three characteristics are three different sides of results of a phenomenon.

An idea that fracture phase separation concerned deeply with the formation of these surface structures of the earth will be presented. A possibility of occurrence of this phenomenon on the surface of the earth will also be discussed.

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Keywords: continents, oceans, structuring process, phase separation, fracture, viscoelasticity

## Pressure transmission in a granular system

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Usual fluids such as water obey a very simple pressure transmission law, so called the Pascal's principle. Then, how does pressure transmit in the earth? Since the structure of the earth is complex in terms of rheological properties, the answer for this question is not so easy. To understand the fundamental pressure transmission manner in the earth, we have experimentally studied the pressure transmission in a granular column. Although the actual constituents of the earth are much more complex than ideal granular matters, a small granular column has been used, as a first step. Moreover, we restrict ourselves within the pressure, i.e., we do not consider the shear stress components. Glass beads or sand are poured into a small cylindrical container. Then, an intruder is penetrated into it very slowly. The intruder is subjected to the drag force and induces pressure. We have simultaneously measured the drag force and the pressure at the wall. We found that they show nonlinear relations. It is known that the static wall pressure exhibits a saturation tendency in the deep region of the granular column. This tendency is called Janssen effect. The Janssen effect is based only on the static pressure balance. There has not been any pressure transmission law for pressed or plunged granular column, although it should be a fundamental principle to discuss the granular rheology. Only the penetration drag has been measured so far [1,2]. Our result reveals the simple but nontrivial scaling for the pressure transmission in a plunged granular column. More specifically, we obtained the empirical pressure transmission scaling, using the measured data. In the scaling, dimensionless thickness of the granular layer plays an important role. That is a source of nonlinearity of the drag force and pressure transmission. Moreover, we have checked the history dependence of the granular pressure transmission. Influences of various sample preparation methods and cyclic loading tests were experimentally evaluated. As a result, we found the universality and robustness of the nonlinear scaling.

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Keywords: granular matter

## Rate dependence of granular friction and its characteristic shear rate

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In geoscience, the rate and state-dependent friction law is established, showing negative shear-rate dependence (Scholz, 1998, Nature). In statistical physics, another empirical law holds for much faster deformation than the former, showing positive shear-rate dependence (Jop et al., 2006, Nature). However, it remains unknown how these two distinct laws are connected. In this study, we experimentally show that the crossover from negative to positive shear-rate dependence of friction coefficient occurs at a characteristic shear rate, relating to competition between two different physical processes, namely frictional healing and anelasticity. We determine the expression of the characteristic rate.

Keywords: friction, granular matter, rheology

## Physical processes of quartz amorphization due to friction

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Solid state amorphization of minerals occurs in indentation and shock experiments, and has been observed in high pressure metamorphic rocks. A production of amorphous material is also reported in experimentally created silicate gouges (Yund et al., 1990), and in San Andreas Fault core samples (Janssen et al., 2010). Rotary-shear friction experiments of quartz rocks imply dynamic weakening at seismic rates (Di Toro et al., 2004). These experiments have suggested that weakening is caused by formation and thixotropic behavior of a silica gel layer which comprises of very fine particles of hydrated amorphous silica on fault gouges (Goldsbey & Tullis, 2002; Hayashi & Tsutsumi, 2010). Therefore, physical processes of amorphization are essential to better understand the weakening mechanism of quartz bearing rocks. In this study, we conducted pin- and ball-on-disk friction experiments to investigate details of quartz amorphization (Muto et al, 2007). Disks were made of single crystals of synthetic and Brazilian quartz. The normal load  $F$  and sliding velocity  $V$  were ranged from 0.01 N to 1 N and from 0.01 m/s to 2.6 m/s, respectively. The friction was conducted using quartz and diamond pins (curvature radii of 0.2 ~ 3 mm) to large displacements ( $> 1000$  m) under controlled atmosphere. We analyzed experimental samples by Raman spectroscopy and Fourier transform infrared spectroscopy FT-IR. Raman spectroscopy (excitation wavelength 532.1 nm) provides lattice vibration modes, and was used to investigate the degree of amorphization of samples. Raman spectra of friction tracks on disks show clear bands at wavenumbers of 126, 204, 356, 394, and 464  $\text{cm}^{-1}$ , characteristics of intact  $\alpha$ -quartz. Remarkably, in experiments using diamond pins ( $F = 0.8$  N, normal stress calculated by contact area = 293 ~ 440 MPa,  $V = 0.12 \sim 0.23$  m/s), the bands at 128, 204 and 464  $\text{cm}^{-1}$  gradually broaden to reveal shoulders on the higher-wavenumber sides of these peaks. Especially, three distinguished peaks at 490, 500 and 515  $\text{cm}^{-1}$  and a weak broad peak at 606  $\text{cm}^{-1}$  appear sporadically on the track after the slip distance of  $> 7$  m. Moreover, in experiments using quartz pins ( $F = 1$  N, normal stress calculated by contact area = 1 MPa,  $V = 0.01 \sim 2.6$  m/s) after a large displacement ( $> 78$  m), the frequency shifts or appearance of new distinguished peaks similar to those in experiments using diamond pin are found. The bands at 490 and 606  $\text{cm}^{-1}$  can be assigned to the symmetric stretching of four-membered  $\text{SiO}_4$  ring (D1 band) and planar three-membered  $\text{SiO}_4$  ring (D2 band) in amorphous silica, respectively. The peaks at 500 and 515  $\text{cm}^{-1}$  correspond to the strongest moganite A1 mode and the strongest coesite A1 mode, which arise from four-membered  $\text{SiO}_4$  ring structure. These results indicate that quartz change intermediate range structure of  $\text{SiO}_4$  network during friction, and four or three-membered  $\text{SiO}_4$  rings gradually increase in six-membered quartz. The results of FT-IR analyses on friction tracks showed the presence of a broad peak at 3000 -3600  $\text{cm}^{-1}$  only at frictional tracks, which indicates the  $\nu$ OH symmetric stretching band of molecular  $\text{H}_2\text{O}$ . It shows that hydration of quartz occur only on friction tracks due to friction. The results of Raman spectroscopy and FT-IR as well as flow like structure of wear materials observed by SEM analyses imply that Si-O-Si bridging of strained rings preferentially react with water to form hydrated amorphous silica layer on friction surface. The formation of silica gel layer that occurs locally at real asperity contacts is likely to cause fault weakening observed by pin-on-disk friction experiment.

Keywords: quartz amorphization, friction experiment, fault weakening, raman spectroscopy

## Formation process and mechanism of slickenside

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Pseudotachylyte is well known rock to be produced by solidification of friction-induced melts during seismic fault slip. Therefore many researches of the pseudotachylyte are conducted to elucidate the microstructural process of seismic faulting. Slickenside is also known as a fault related rock (or structure), which shows smoothed surfaces developed on planes of movement. However the generation process and mechanism of slickenside has not been clarified so far, compared with pseudotachylyte. Now we study the microstructure of slickenside developed on chert block to get some information about generation process of slickenside.

The sample we studied is the chert block which occurs in the Jurassic accretionary complex in eastern Yamaguchi Prefecture, Japan. The complex, chaotic sediment, is composed of allochthonous blocks, mainly of chert, limestone, sandstone and mudstone in the argillaceous matrix, which has undergone very low grade metamorphism under conditions of Prehnite-Pumpellyite facies. The microstructural observations of quartz grains composed of the slickenside with an optical microscopy indicate that 1) they are not deformed by brittle manner even just vicinity of slickenside, 2) they show strong undulose extinction and bulging-recrystallization. TEM observation and EBSD measurement of these quartz grains reveal that 3) the dislocation tangling is typical, 4) they do not show LPO. The most important result is that 5) the amorphous layer having several ten nanometers thickness (~50nm) covers the top surface of slickenside, and 6) the composition of the amorphous layer is mainly Si, Fe and Al. These microstructural observations suggest that 1) the quartz grains were deformed plastically under high stress condition (but the plastic strain is small), 2) the amorphization of quartz grains occurred just near (~50nm) slip surface during faulting, and 3) the origin of Si, Fe and Al in the amorphous layer should be quartz grain itself and clay minerals (e.g. Biotite, Muscovite) contained in the chert. Because the quartz grains are not deformed by brittle manner, the fault slip probably occurred only within the amorphous layer.

We can not presently identify the mechanism of amorphization of quartz grains along slip surface. But we obtain the additional interesting microstructure which is the black veins and clusters in the vicinity of slickenside. These veins and clusters are mainly composed of apatite grains, whose size is ca. 5 to 10  $\mu\text{m}$ . Apatite is an important constituent mineral of chert, whose origin is microfossils such as fish scales, bones and so on. Therefore the generation of the black veins can be explained by the melting of the microfossils due to frictional heating of fault. We currently conclude that the slickenside was generated by frictional melting of quartz grains and clay mineral on the slip plane under high stress condition during faulting.

Keywords: slickenside, pseudotachylyte, fault, chert, microstructure

## Effect of pore pressure on frictional properties of talc 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. The role of pore fluid pressure at the brittle-plastic transition zone is especially important because this zone, located at the deepest part of the seismogenic zone, supports large shear stress during interseismic periods and therefore releases large energy at seismic events [1]. However, it is not well documented how pore fluid pressure influences frictional properties of faults at the depth, which is mainly due to the difficulty in conducting 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 used talc as an analogue material, which shows brittle-plastic transitional behaviors at relatively low pressures and temperatures.

Previous works on rock mechanics have suggested that the yield strength of rocks is governed by effective stresses  $S_e = S - C P_p$ , where  $S$  is total stress,  $P_p$  is pore fluid pressure, and  $C$  is a factor between 0 and 1. The observations in the brittle regime are well accounted for by  $C = 1$  [2]. In the fully plastic deformation regime, however, yield stress is not significantly affected by pore pressure and the strength would not be lost even if  $P_p$  is equal to  $C$  [3]. This is quite different from the brittle deformation at shallow crustal levels. In the brittle-plastic transitional regime, intermediate behaviors between fully brittle and fully plastic deformation are expected. As a first step to quantify the fluid effects, we conducted friction experiments of talc at various  $P_p$  and  $C$  conditions.

Cylindrical samples of talc, from Gvangsih, China, 20mm in diameter, were cut at an angle of  $30^\circ$  to the sample axis. The surfaces were ground with carborundum (#400). A small hole (3mm in diameter) through the center of each talc 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 gas-medium 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 the following four types of stress paths: (a) pore pressure was held constant at 0 MPa, and confining pressure was increased from 10 MPa to 110 MPa, and then decreased 10 MPa. (b) Confining pressure was held constant at 110 MPa, and pore pressure was decreased from 100 MPa to 0 MPa, then increased up to 100 MPa. (c) Confining pressure was held constant at 110 MPa, and pore pressure was increased from 0 MPa to 100 MPa. (d) Confining pressure was held constant at 110 MPa, and pore pressure was held at 100 MPa for a hour before axial loading, and then was decreased to 0 MPa and the specimen was sheared.

When we compared the results of shear stress measurements on the stress path (a) with others and derived the constant  $C$  in the effective stress law,  $C$  was almost equal to 1 through whole stress path in the case of (b). On the other hand, in the case of (c), the results could not be explained when  $C = 1$ .  $C$  of (d) was almost 1, but the peak shear stress were approx. 1.5 times larger than that of (a). These results indicate that  $C$  depends on the stress path and that the effective stress law cannot be directly applied to talc under these conditions.

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Keywords: fault mechanics, brittle-plastic transition, effective stress law, talc, friction experiment

## Brittle-ductile transitional zone in the Tohoku-oki interplate thrust fault

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Before the 2011 Tohoku earthquake (M9), the interplate coupling in NE Japan, where a cold slab of the Pacific plate is subducting, had been believed to be weak.

A strength envelope is proposed for the plate boundary megathrust that generated the 2011 Tohoku earthquake. It is assumed that the frictional properties of the plate interface is governed by subducting oceanic crust materials and sediments in the accretionary prism. For siliceous oceanic sediments, a dislocation creep flow law wet quartz (Paterson and Luan, 1990) was applied. The hypocenter of the M9 event is located at a depth the deepest part of the island arc crust or the uppermost part of the wedge mantle. The pressure and temperature conditions at this depth corresponds to the upper limit of the brittle-ductile transitional zone of wet quartz. The rupture of this high-strength zone at mainshock of the 3.11 event is possibly triggered by the collapse of the subducted seamount. The M7-class asperity of the Miyagi-oki earthquake in 1978 is considered as a seamount, which has subducted to the depth of the mantle wedge. The conditionally stable nature of their matrix is understood by the velocity-dependence of viscous/frictional properties in the brittle-ductile transitional zone.

Keywords: The 2011 off the Pacific coast of Tohoku Earthquake, asperity, rheology, subduction zone, brittle-ductile transition, quartz

## Rheology of quartz at high-temperature: an example from Rundvagshetta, Antarctica

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Quartz is one of the most common minerals in the continental crust and has a relatively low yield strength. The rheological behaviour of this mineral therefore has an important influence on the mechanical behaviour of the crust as a whole. High-temperature crystal plastic deformation is dominantly achieved by dislocation creep and diffusion creep. Dislocation creep is due to the movement and climb of line dislocations within crystals and shows power law stress-strain relationship with the stress exponent usually between 3 and 6. This represents non-Newtonian behaviour where the apparent viscosity depends on the differential stress: high stress domains have a low apparent viscosity. In contrast, diffusion creep is caused by the movement of point defects including vacancies and interstitial atoms and is characterized by a linear relationship between stress and strain representing a Newtonian rheology. The strain rate and temperature also affect the dominant deformation mechanism. To assess the bulk rheology to be used in tectonic studies it is important to assess the affects of these different parameters and to decide the dominant deformation mechanism. Laboratory experiments on quartz shows that that diffusion creep is dominant at relatively high T and small grain sizes (>900K, <1micro m). Plastic deformation at lower T and for larger grain sizes is expected to take place by dislocation creep. However, natural examples of diffusion creep taking place at temperatures of around <500°C in quartz-rich metacherts with a grain size of about 10-20micro m have recently been reported. Such examples suggest that diffusion creep may occur more commonly in natural examples than generally thought. To investigate this possibility, it is important to examine natural examples of deformation at higher temperatures. Two quartz-rich deformed samples collected from the ultra high temperature metamorphic region of Rundvagshetta (RH-112-20A, RH-112-20B) were studied to examine evidence for the dominant deformation mechanism. Both samples show a strong foliation and stretching lineation that are assumed to reflect the X-Y plane and X directions of the finite strain (X>Y>Z).

RH-112-20A has a grain size of 2.9+/-1.2mm, and a strong quartz c-axis preferred orientation with concentrations in three separate directions. The presence of a strong CPO is strong evidence for deformation by dislocation creep. The orientations of the c-axis concentrations are similar to those reported in other high temperature tectonites. RH-112-20B has a grain size of 0.93+/-0.03mm and the c-axes show no clear preferred orientation. The lack of a CPO is not compatible with dislocation creep, but can be readily explained if the deformation is by diffusion creep. Measurements of the Ti content of quartz can be used as an indication of the P-T conditions of deformation. A combination of these results with microstructural observations and the P-T path of this area allows the temperature of deformation to be estimated at around 600~700°C, 4~6kbar. The two samples come from the same outcrop and hence have undergone very similar physical conditions of deformation. However, they show contrasting deformation mechanisms. This can be used to investigate the applicability of published rheological models for quartz. A construction of deformation maps using flow laws derived from mainly from experimental work and those derived mainly from theoretical considerations shows that the results of this study are not compatible with extrapolation of experimental work. In theoretically focused flow laws can explain the observed change of dominant deformation mechanism at strain rates of around  $10^{-15} \text{ s}^{-1}$ .

This study implies deformation in the Rundvagshetta region was as geologically normal strain rates despite the very high temperatures. This study also implies that diffusion creep may be more widespread than generally thought by extrapolation of deformation experimental work.

Keywords: Quartz, Deformation mechanism, Crystal Preferred Orientation, Diffusion creep, Dislocation creep



## Phenomenological constitutive law for transient rheological behavior of rocks and minerals

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Phenomenological constitutive law for transient rheological behavior of rocks and minerals involving the work hardening effect is investigated in terms of viscoelastic theory and fractional rheology. Viscoelastic behavior is often defined in term of the linear response function, and output of stress is the convolution integral of viscoelastic response and strain-input. The response function is called relaxation modulus (a time-dependent modulus of stress to strain) and regarded as a constitutive equation. The convolution integral is identical to a definition function of fractional-order derivative, and stress is then equal to the noninteger-order derivative of strain, which expresses the behavior between Hookean elasticity and Newtonian viscosity. Using the constitutive law, we analyze the experimental data of high-temperature deformation of rocks and minerals such as marble, halite and orthopyroxene. The relaxation modulus of rocks and minerals shows a temporal power-law scaling, and the exponents of the power-law corresponds to the reciprocal of exponent of stress in the flow law. The constitutive equation then represents both transient and steady-state behaviors in the same mathematical structure. The exponents span the range from 0.04 to 0.13 (the stress exponent, from 7.5 to 25.0) for the transient behaviors and from 0.14 to 0.25 (the stress exponent, from 4.1 to 7.1) for the steady-state behaviors. The exponent of stress in the flow law is dependent on the deformation mechanisms such as diffusion and dislocation creeps. For orthopyroxene, the response function involves the strain nonlinear function, a power-law of strain, expressing the effect of work hardening, and can be transformed into the empirical evolution equation of work hardening, stress equals to the power-law of strain and strain-rate.

Keywords: viscoelasticity, fractional calculus, transient behavior, rheology, work hardening

## Olivine fabric transition during ductile shearing in the uppermost mantle: an example from Oman ophiolite

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A ductile shear zone across the crust-mantle boundary occurs in the Fizh massif, Oman ophiolite. The dunites in the ductile shear zone were classified into coarse granular texture, medium-grained texture, protomylonite, mylonite and ultramylonite. The average grain sizes of olivine decreased toward the shear zone, by which a high strain zone was estimated approximately 15 m. Amount of hydrous minerals (amphibole and chlorite) and spinel Cr# in the mylonites increased toward the gabbro boundary, suggesting that water infiltration into the ductile shear zone could occur from the gabbro boundary. The amphibole porphyroclasts show deformation structures, indicating that the water infiltration and subsequent water-induced metamorphic reactions occurred before or during shearing. P-T estimate and equilibrium temperatures show that the shear zone may be thought to preserve higher deformation temperature (around 900 degree C) for outside of the high strain zone and lower deformation temperatures (around 750 degree C) for inside of the high strain zone. Olivine CPO pattern evolutions indicate the following continuous deformation scenario. First, the deformation by dislocation creep at high temperature condition have formed A-type CPO or E-type CPO (more close to high strain zone). Next, the deformation by dislocation creep at low temperature and wet conditions have formed C-type CPO. Finally, superplastic deformation by grain boundary sliding at low temperature and wet conditions have occurred and formed random CPO.

Keywords: olivine, fabric analysis, ductile deformation, uppermost mantle

## The uppermost mantle evolution during back-arc spreading: Microstructural and petrological characteristics of Ichinomega

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This study deals with the microstructural development in the uppermost mantle associated with melt/fluid rock interactions in peridotites induced by the back-arc spreading. We have studied spinel peridotite xenoliths from Ichinomegata volcano, back-arc region of Japan Islands. The mineral chemistry shows a typical residual trend, depleted in LREE. Their strong Th-U positive anomaly indicates a possible metasomatic origin associated to the subduction of the Pacific plate. Water contents in olivine and pyroxenes were low, which values are in the same range of spinel peridotite xenoliths sampling the continental lithosphere. Olivine CPO are consistent with slip on (010)[100] and {0kl}[100]. Moreover, the peridotite xenoliths have distinct foliations defined by the compositional layers between olivine-rich and pyroxene rich layers as well as lineations defined by mineral shapes of olivine and pyroxene. The angles between the foliations and the olivine slip planes decrease with increasing J-index values (i.e. CPOs strength). Such composite planar relationships could result from shearing in the uppermost mantle, so that shear strains may be estimated by the angles between the foliation and the olivine slip plane in terms of simple shear strain. As a consequence, we argue that a suit of the peridotite xenoliths recorded a rare snapshot of the uppermost mantle flow related to back-arc spreading during the opening of Japan Sea. Moreover, the peridotites xenoliths with higher J-index values (higher shear strain) tend to have lower minimum temperature, indicating that a vertical strain gradient could take place from upper to lower in the uppermost mantle section.

Keywords: olivine, peridotite, fabric, back-arc spreading, upper mantle, mantle dynamics

## Influence of anisotropic grain growth on development of lattice preferred orientation of forsterite

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It is believed that LPO ( lattice preferred orientation ) of main mineral of mantle is caused by the flow of the Earth's interior, which introduces seismic anisotropy. Generally, it is considered that LPO (and seismic anisotropy ) is generated by rock deformation via dislocation creep. In this study, we deformed mineral aggregates of forsterite + diopside by uniaxial compression and tension tests at atmospheric pressure, temperature of 1200 to 1350 degree, and strain rate of  $1 \times 10^{-6}$ -  $1 \times 10^{-4} \text{ s}^{-1}$ . The stress exponent (n) of 1 was obtained from the analysis of stress-strain relationship showing the aggregates deformation via diffusion creep mechanism. Crystallographic orientation analysis of forsterite grains in the deformed aggregates was conducted using electron backscatter diffractometry. The results show b-axis alignment parallel to the compression axis in the compressed samples and a-axis alignment in the tensiled samples. Long axis of grains in statically annealed samples is often parallel to a-axis of forsterite. We consider that the LPO development under diffusion creep is attributed to the alignment of anisotropic shaped of forsterite to the tensile direction after its anisotropic grain growth.

## Deformation experiments of (Mg,Fe)SiO<sub>3</sub>-Perovskite at the lower mantle conditions

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The Earth's lower mantle is thought to be consisting of ~77 vol% (Mg,Fe)SiO<sub>3</sub>-perovskite (Pv), ~16 vol% ferropericlae and ~8 vol% CaSiO<sub>3</sub>-perovskite in pyrolite model. In the uppermost and lowermost lower mantle, presence of seismic anisotropy has been reported. The anisotropy may be produced by lattice preferred orientation of the majority phase Pv. Thus, knowledge of slip system of Pv is important for understanding of rheology in the lower mantle.

Shear deformation experiments at the lower mantle conditions were conducted using Kawai-type apparatus triaxial deformation (KATD) at Tokyo Institute of Technology. Dense aggregates of synthetic (Mg,Fe)SiO<sub>3</sub>- orthopyroxene (Mg# = 0.97) was prepared as starting material using Kawai-type multi anvil apparatus at 2 GPa and 1273 K. The shear deformation assembly consists of a Cr<sub>2</sub>O<sub>3</sub>-doped MgO pressure medium with 7 mm edge length and a cylindrical LaCrO<sub>3</sub> furnace. Temperature was estimated from power-temperature relationship in a similar cell assembly. Pt foil is placed at the back of Al<sub>2</sub>O<sub>3</sub> piston to assist sideslip of the piston, and Ni foil placed at the center of sample is used as strain marker. Undeformed runs, which were not deliberately deformed by the differential rams, were quenched after phase transitions and relaxation of stress at 25 GPa and 1873 K. In deformation runs, samples were deformed by moving differential rams in the guide blocks each 75 μm (total 150 μm) during 1 h after the annealing process (for 30 min or 1 h).

Lattice preferred orientation (LPO) of sample was determined using 2D-Xray diffraction patterns of sample. The 2D-Xray diffraction patterns were measured using Imaging plate in the SPring-8 (BL04B1). Analysis of LPO was conducted by the software "Recipro".

Strains of undeformed and deformed recovered samples measured from rotation of strain marker were ~0.4 ± 1 and ~1.3 ± 1, respectively. Then total strains of samples during deformation process in deformation runs are calculated to be ~0.9 ± 1. Average strain rates of sample are  $3 \times 10^{-4} \text{ s}^{-1}$ . LPO of perovskite in shear deformation experiments developed greatly in comparison with that at undeformed experiment. The results suggest that main slip system of Pv at 25 GPa and 1873 K is [100](001).

Keywords: Lower mantle, Deformation experiments, (Mg,Fe)SiO<sub>3</sub>-Perovskite

## Si and Mg diffusion in aluminous perovskite at 25 GPa

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Silicate perovskite is thought to be a major constituent mineral in the lower mantle. In order to understand rheological properties of the lower mantle, it is essential to determine the diffusion rates of the slowest diffusing species which control high-temperature creep processes involving diffusion creep and climb-controlled dislocation creep. It has been reported that Si diffusion rates in MgSiO<sub>3</sub> perovskite and Mg-Fe interdiffusion rates in (Mg,Fe)SiO<sub>3</sub> perovskite are almost comparable (Yamazaki et al., 2000; Holzapfel et al. 2005). In addition, O diffusion rates in MgSiO<sub>3</sub> perovskite are faster than Si diffusion and Mg-Fe interdiffusion rates (Dobson et al. 2005). Thus, Si or Mg is a candidate for the rate-controlling species in perovskite. In this study, we determined Si and Mg diffusion rates simultaneously in (Mg,Fe)(Si,Al)O<sub>3</sub> perovskite by utilizing <sup>25</sup>Mg and <sup>29</sup>Si enriched (Mg,Fe)(Si,Al)O<sub>3</sub> thin film as diffusion source. Based on the result, we discuss the rate-controlling species in (Mg,Fe)(Si,Al)O<sub>3</sub> perovskite and rheological properties in the lower mantle.

High-temperature and high-pressure experiments were performed using a Kawai-type high-pressure apparatus. Starting material of polycrystalline (Mg,Fe)(Si,Al)O<sub>3</sub> perovskite was synthesized from San Carols orthopyroxene powder at 25 GPa and 1973K. Surface of the polycrystalline perovskite was polished and then coated with <sup>25</sup>Mg and <sup>29</sup>Si enriched (Mg,Fe)(Si,Al)O<sub>3</sub> thin film using pulsed laser deposition (Dohmen et al. 2002). Diffusion experiments were conducted at 25 GPa and 1773-2073K. After the diffusion experiments, concentration profiles of <sup>25</sup>Mg and <sup>29</sup>Si were obtained by the depth-profiling mode using secondary ion mass spectrometry.

It was found that Si and Mg diffusion rates in (Mg,Fe)(Si,Al)O<sub>3</sub> perovskite are almost comparable under our experimental conditions. Thus, Si and Mg are likely to be rate-controlling species in (Mg,Fe)(Si,Al)O<sub>3</sub> perovskite. Si and Mg diffusion rates in (Mg,Fe)(Si,Al)O<sub>3</sub> perovskite could be slightly slower than previously reported Si diffusion rates in MgSiO<sub>3</sub> perovskite.

Keywords: perovskite, lower mantle, diffusion, rheology

## Predictions of the shear response of Fe-bearing MgSiO<sub>3</sub> post-perovskite at lowermost mantle pressures

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Observation of seismic data put in forth evidence of a spatial anisotropy in the seismic wave velocities in the D'' layer, the lowermost part of the mantle. (Mg,Fe)SiO<sub>3</sub> post-perovskite (PPv) is thought to be the most abundant phase in this part of the mantle. This mineral exhibits a strong elastic anisotropy and may contribute significantly to the seismic anisotropy in the D'' layer. However, the seismic anisotropy cannot be expressed at the rock scale if the orientations of the grains are distributed randomly. Consequently, the formation of lattice preferred orientations with an anisotropic mechanism of plasticity, such as dislocation creep, can cause the seismic anisotropy in the D'' layer. Some experiments have been done on the plasticity of pure and Fe-bearing MgSiO<sub>3</sub> post-perovskite and lead to textures of deformation dominated by the (100) and (110) slip planes (Merkel et al., 2007) or by the (001) slip plane (Miyagi et al., 2010). On the other hand, theoretical calculations on the dislocations mobility on pure MgSiO<sub>3</sub> (Carrez et al., 2007; Metsue et al., 2009) suggested a texture dominated by the (010) slip plane. A first step to understanding the mechanisms of plasticity and, therefore, the shear wave splitting occurring in the deep Earth is to test the response of the PPv phase to a plastic shear in a geophysical relevant composition.

In this study, we present new results from first-principles calculations on the shear response of pure and ferrous Fe-bearing MgSiO<sub>3</sub> PPv. The originality of this work is the use of internally consistent LSDA+U formalism to accurately describe the local interactions between the d-states of Fe. About 8% of Fe<sup>2+</sup> is incorporated in the high spin and low spin states, as a Mg substitution defect, to test if a spin transition could be induced by shearing mechanisms, even several studies report that Fe<sup>2+</sup> is in the high spin in the D'' layer pressure range (Stackhouse et al., 2006; Metsue and Tsuchiya, 2011). The response of the PPv to a plastic shear is investigated at 120 GPa through the calculations of the Generalized Stacking Faults (GSF) energy in pure and Fe-bearing systems for 10 potential  $\langle uvw \rangle \{hkl\}$  slip systems, since these latter are not well constrained for the PPv phase. The GSF energies are obtained by shearing homogeneously half of an infinite crystal over the other half for every slip system and give the value of the ideal shear stress (ISS), which can be defined as the theoretical elastic limit of the crystal. The [100](001) slip system in pure and Fe-bearing phases exhibits the lowest ISS and may play an important role in the plastic deformation of the PPv phase. The activation of this slip system is compatible with the observed shear wave splitting  $V_{SH} > V_{SV}$ . We show that incorporation of Fe decreases the GSF energy and the ISS of all slip systems. In particular, the decreasing of the energy of stable stacking faults indicates that Fe tends to be adsorbed in the stacking faults, which increases the width of the defect and could have some implications on deformation mechanisms. Finally, we discuss the plastic anisotropy of pure and Fe-bearing phases from the values of the ISS and the orientation of applied tensile stress. Our results suggest that the incorporation of ferrous Fe in the PPv phase has a limited effect on its plastic anisotropy, and, therefore, on the deformation texture.

Keywords: MgSiO<sub>3</sub> post-perovskite, stacking faults, deformation mechanisms

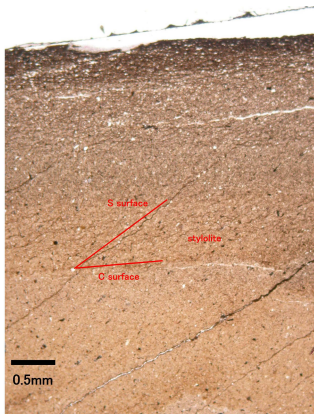
## Deformation mechanism of spherical/elliptical sandstone bodies

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Spherical blocks (10 cm to 10 m in diameter) and lenticular sandstone bodies (several centimeters to several tens of centimeters) with S-C structure are observed in Shimanto supergroup, Miyazaki Prefecture, southwest Japan. Those are emplaced under rheological conditions with frictional heat caused earthquake on fault.

Keywords: S-C structure, friction solution, rheology transition



Spherical tectonic block 12cm in diameter  
(pelagic clay)



## Factors controlling entablature formation in columnar joints: Suggestions from the analogue experiments

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Columnar joints of lava and ignimbrite have two types of structure within one flow unit: colonnade and entablature. Colonnade have relatively wide, straight and directionally ordered columns, on the other hand, entablature have relatively narrow, curved and disordered columns in different directions. Columnar joints are formed by volume contraction due to temperature decrease during cooling. The fractures in colonnade are thought to develop perpendicular to the isotherm. In entablature, however, how the complex structure is related to the isotherm and what causes such a complex structure is still unclear. It has been well known that in desiccation experiments using starch-water mixtures, structures similar to colonnade form, but structures similar to entablature have not been reproduced so far. Therefore, this study aims at reproducing the entablature structure with additional conditions different from experiments before. We conducted consisting of three experiments: Experiment 1: To reproduce the typical threefold structure: upper colonnade, entablature and lower colonnade, we designed an experimental setup in which water can evaporate from upper and lower surface of mixture by attaching a membrane. The drying rate is controlled by changing the distance between the lamp to the starch surface. For a monotonic drying rate with a constant distance being 10cm, the colonnade structure developed from upper and lower surface. Experiment 2: Under the same condition of Experiment 1, after the colonnade structure developed partially, we suddenly increased the drying rate by shortening the distance between the lamp and the mixture surface from 10cm to 1.5cm. As a result, the colonnade structure developed discontinuously correlating with the sudden change of the drying rate. A curved structure to the rim of the container developed after changing the drying rate. To observe the whole structure by Experiment 2 in detail, we took images by Micro-focus X-ray CT (manufactured by Tesco at 119 micro meter resolution, at Fukuoka Industrial Technology Center). From analysis of images, we found following three facts: 1) the number of fractures suddenly increase after changing the drying rate, 2) the widths of columns, which developed before changing the drying rate, become smaller and 3) the new columns form at the triple or quadruple junctions of cracks constructing columns which developed before changing the drying rate. Experiment 3: To examine the cause that forms a curved structure, we conducted the experiment under the constant drying rate with the distance being 1.5cm. From the images which we took before the mixture dried perfectly, the curved cracks develop perpendicular to an iso-surface of water concentration. From these results, we concluded that a sudden increase in contraction rate forms new columns at the triple or quadruple junctions to release accumulated tensile stress, which remains after the growth of pre-existing cracks. The inhomogeneous concentration distribution with a curved iso-surface results in the formation of curved cracks. We could substantiate the possibility that the entablature structure in columnar joints is caused for the inhomogeneous temperature distribution of rocks. As formation processes of new columns, two cases are expected 1) the case that one column was divided into multiple columns by forming new cracks inside the columns and 2) the case that new columns nucleate at triple and quadruple junction of cracks, although it was difficult to distinguish these two cases with conventional X-ray CTs. In order to closely observe the columns nucleation at triple and quadruple junctions, we use High Resolution 3D X-Ray Microscope VersaXRM-500 (manufactured by Xradia (U.S) at 3.7 micro meter resolution). As a result, it was confirmed that columns nucleations take place at triple and quadruple junction of cracks.

Keywords: columnar joint, analogue experiment, crack, formation process, Micro-focus X-ray CT, 3D X-ray Microscope

## Rheological Characteristics Leading to Magma Flow Instability

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During volcanic eruptions alternate transitions between two contrasting states are known to generate oscillatory phenomena. Switching between tremor stage and non-tremor stage and periodic transition between eruption stage and quiescent stage are typical examples. These should reflect dynamics of magma inside volcanoes, which gives us indispensable information about physics of volcanic eruption. One of the main causes for this transition is instability due to double-valued relation between flow rate and driving pressure. It indicates that two flow rates exist at one pressure and jump between two states causes transition. The origin of this double-valued nature of magma flow has not been clarified yet though volatile-dependent viscosity is suggested. To figure out the dynamics of this transition and physical origin of the double-valued relation, rheology of magma should be a key. Similar phenomenon is known as spurt in the fields of polymer science. It is controlled by a jump of flow rate due to the wall friction controlled by the double-valued relation of stress and strain rate. In this phenomenon an abrupt increase in flow rate under certain range of driving pressure is observed.

We focus on rheology of suspension and explore the possibility of the rheology, which has the double-valued nature. In this study PNIPAM aqueous suspension was used as an analogue material of multiphase magma. Since the volume fraction of PNIPAM systematically changes with the concentration of gel powder and temperature, it is possible to measure change of rheology continuously associated with change of the fraction of solid phase. By experiments with controlled shear rate, we revealed the double-valued relation in shear stress and shear rate at certain range of volume fraction of the solid phase. We would like to remark magma has a similar characteristic rheology, which can explain volcanic oscillatory phenomena. This work was collaborated with E.D Giuseppe and A. Davaille.

Keywords: Magma, Rheology, PNIPAN, Instability, Complex fluid

## Why the black fault rocks appear black color? -Enrichment and alteration processes of minerals in the fault zones-

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Color of rock including fault rocks is one of the important material properties, which is affected by its condition, mineralogy, elements and so on. Black-colored fault rocks frequently found from the intensely-comminuted fault core in different faults (e.g., the Median Tectonic Line, the Tanakura Tectonic Line, the Atotsugawa fault system and the Chelungpu fault). However, origin for black color is still unknown since the coloring agent for each black fault rocks is not examined up to now. I thus performed detailed material analyses and color measurement with spectrophotometer to determine the coloring agent for the black fault rocks.

Keywords: Graphite, Carbon, Black fault rock, Elemental mobilization

## Rheological crossover within the framework of rate- and state-dependent friction

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We derive the rate- and state-dependent friction law starting from creep constitutive laws for a true contact patch. Consequently, the microscopic expressions for phenomenological parameters are obtained that govern the velocity dependence of steady-state dynamic friction. We show that positive velocity dependence is unlikely if the sliding and frictional healing (due to uniaxial compression) are accommodated by the same creep mechanism. We also show that friction may exhibit positive velocity dependence if the frictional healing is dominated by pressure solution.

Keywords: rate- and state-dependent friction, creep, pressure solution

## LPO development of single crystals of wet synthetic quartz sheared at low temperature

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Shear zone develops through strain softening of rocks during progressive deformation. One of the strain softening mechanisms is geometrical softening caused by crystal changes its orientations for easier slip system. Geometrical softening causes development of lattice preferred orientation (LPO) closely related to dynamic recrystallization.

The mechanism of dynamic recrystallization of quartz which is a common crustal mineral is separated into regimes 1-3 by increasing the temperature and decreasing the strain rate. The c axis LPO of quartz changes from a crossed-girdle LPO to Ymax LPO with increasing the temperature and strain (Hirth and Tullis, 1992JSG).

Heilbronner and Tullis (2002Geol. Soc. Spec. Publ.) conducted general shear experiment in each of the three dislocation creep regimes of Hirth and Tullis (1992JSG) and examined the LPOs in each regime. Heilbronner and Tullis (2006JGR) sheared quartz samples experimentally at regime 3 conditions of Hirth and Tullis (1992JSG) where grain boundary migration is the dominant recrystallization mechanism, and examined the changes in c axis LPO with increasing shear strain and degree of recrystallization. Muto et al. (2011JGR) have undertaken an experimental study using single crystals of wet synthetic quartz to investigate the development of LPO in dynamically recrystallized grains and its effect on the flow strength of quartz aggregates. They observed that domains of recrystallized grains with a Y max LPO developed at moderate to high shear strain in all cases by grain boundary migration.

Although Heilbronner and Tullis (2006JGR) and Muto et al. (2011JGR) clarified that the effect of dynamic recrystallization on LPO development in regime 3 conditions, the effect of dynamic recrystallization in lower temperature where subgrain rotation is the dominant recrystallization mechanism is not clear yet because Heilbronner and Tullis (2002) have used the natural quartzites. The use of single crystals of known initial crystallographic orientations makes it possible to determine relationships between the initial crystallographic orientation and those of recrystallized grains.

In this study, we conduct general shear experiments in a Griggs apparatus using single crystals of synthetic quartz in order to investigate the development of LPO in dynamically recrystallized grains at a low temperature where subgrain rotation is a dominant recrystallization mechanism.

Experiments are conducted at  $P_c=1.5$  GPa,  $P=600-700$ °C at a constant strain rate of 10-5 /s. Starting materials oriented to activate three main slip systems are sandwiched by alumina pistons that cut at 45° from the maximum compression direction. Before experiment, water content of samples are measured by FTIR analysis. After deformation experiments, crystallographic orientations were measured using EBSD analysis. We investigate the development of LPO of recrystallized quartz aggregates in three different initial orientations with increasing shear strain and degree of recrystallization.

The basal<a> sample ( $\gamma \sim 0.7$ , no recrystallization) show the c axis LPO of a symmetric single broad peripheral maximum, rotated 35° with the sense of shear. Other basal<a> sample deformed to  $\gamma \sim 2.5$  shows strain hardening after yielding at  $\gamma \sim 1$ . Its c axis LPO has a symmetric single broad peripheral maximum, rotated 90° with the sense of shear from initial Z axis to X axis. This indicates that the shear deformation and thinning rotates crystal that causes the activation of hard prism[c] slip system. The prism<a> sample deformed to  $\gamma \sim 2.5$  becomes steady state with a flow stress of 300MPa after yielding at  $\gamma \sim 0.5$ . In the presentation, we will show the experimental results on the LPO development in three different initial orientations as a function of strains and degree of dynamic recrystallizations.

## Deformation experiment on synthetic polycrystalline anorthite: effect of water

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Feldspar is a major constituent of the crust as well as quartz. The rheology of the middle-lower crust is mainly governed by that of feldspar. Therefore, the deformation mechanisms of feldspar have been investigated from analyses of natural samples and by deformation experiments. Plastic deformation of materials including feldspar is broadly classified into two main mechanisms; grain-size-sensitive creep (dislocation creep) and grain-size-insensitive creep (diffusion creep and grain boundary sliding). The former mechanism exhibits lattice preferred orientation (LPO) and the latter does not.

It is well known that increasing water contents in polycrystalline feldspar promote plastic deformation, as reported in Dimanov et al. (1999, JGR) and Rybacki et al. (2006, JGR). However, capability of a pressure range by a gas a gas (Paterson-type) deformation apparatus used in their studies is limited up to 400 MPa. Therefore, a solid-medium deformation apparatus should be used to investigate the middle-lower crustal condition, where water circulation is important as suggested in recent tomographic studies. Also, H<sub>2</sub>O fluid easily diffuses into a polycrystal through grain boundaries as fast mass paths. Then, the subsequent solution-precipitation process may occur, as inferred from grain morphologies and development of LPO controlled by the solution-precipitation process (Vernooij et al., 2006 Tectonophysics for quartz; Heidelbach et al., 2000, JSG for feldspar). Therefore, the effects of water on e.g., transition from brittle deformation to plastic deformation, and relationship between stress/strain and deformation mechanisms should be quantitatively determined.

In this study, we focus on the middle-lower crustal conditions, external influx of water, and polycrystalline feldspar, and performed following deformation experiments. Shear and axial deformation experiments were conducted for initially dry feldspar samples with water added using a Griggs-type deformation apparatus. The temperature and confining pressure are up to 950 C and 1 GPa. As a starting material, fine-grained (<5 μm) glass powders with the composition of anorthite 100 were sintered in vacuum at 1400 C during 4 hours. Then, polycrystalline anorthite with a grain size less than 5 μm were prepared. Electron backscattered diffraction (EBSD) measurements revealed that crystallographic orientations of the synthesized polycrystalline anorthite were random, although elongated grains with the aspect ratio of up to 2 were observed in a band contrast image. Observation of sample morphology shows that pores up to 1 μm are present at grain junctions. Infrared (IR) spectroscopy revealed that the sample is dry without water absorption bands. Using these samples, 0.1-0.5 wt % water was introduced into the sample by adding distilled water or by dehydration of pyrophyllite powders at high pressure and temperature. After the deformation experiments, concentration of interference color at 300 μm from the sample edges were observed under a polarized optical microscope with a gypsum plate, indicating development of LPO. Pores were not recognized in these regions. These observations indicate that plastic deformation occurred due to introduction of water into the sample. In the IR spectra for this region, water absorption bands due to zoisite, which must be produced as a reaction product under differential stress, were recognized. The creep mechanisms of feldspar, which are dominated during deformation due to water distribution will be determined from electron backscattered diffraction (EBSD) analyses and observations of grain morphologies. We will discuss changes in stress/strain due to introduction of water into the sample, and relationship between water distribution and deformation mechanisms.

Keywords: anorthite, water introduction, Griggs deformation apparatus, EBSD analysis, infrared spectroscopy

## FABRICATION OF HIGHLY DENSE AND FINE-GRAINED POLYCRYSTALLINE ANORTHITE BY VACUUM SINTERING

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**Introduction:** In laboratory measurements of physical and chemical properties of the earth's lower crust, highly dense polycrystalline aggregates of major constituent minerals such as anorthite ( $\text{CaAl}_2\text{Si}_2\text{O}_8$ ) are required. For precise measurements of these properties, the aggregates should have (1) high density, (2) fine grain size (to escape from cracking during quenching), (3) homogeneous microstructure, (4) controlled sample shape and size, and (5) controlled chemistry. We have developed a technique to synthesize anorthite aggregates which meet the above conditions.

**Experimental:** We used nano-sized powders of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{CaCO}_3$  and  $\text{Mg}(\text{OH})_2$  as starting material to synthesize single phase aggregates of anorthite and, two phase composite of anorthite + diopside and anorthite + quartz. Those powders were mixed to provide a resulting mole ratio of CaO,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ , and MgO to obtain desired mineral assemblies. Densified aggregates were obtained through the following stages: calcination, forming and sintering. The final materials were characterized with X-ray powder diffraction (XRD) and secondary electron microscopy (SEM).

### Results and Discussion:

*Anorthite:* Calcination was conducted under temperature of 670-970oC with fixed duration of 30-180 min. Coalescence of the powder was observed > 820oC, which was revealed by SEM. XRD result indicates that decarboxylation of anorthite solid state reaction completes at > 770oC for 90 min. For sintering, a temperature of 1210-1260oC with controlled time can provide essentially full dense aggregates of anorthite with an average grain size of 1.7 micro m and porosity of 0.2 vol%.

*Two-phase materials (anorthite + diopside):* Diopside grains of 10 vol% was introduced to anorthite aggregates. Calcination at 770oC for 30 min and sintering at 1200oC for 50h were found to be the best conditions so far to synthesize highly dense aggregates. Average grain sizes of 1.6 micro m and 0.8 micro m were detected for anorthite and diopside grains, respectively. Density of 99.6% was achieved.

**References:** S. Koizumi et al., Phys. Chem. Miner. 37, 505-518 (2010)

Keywords: lower crust, anorthite, grain size, sintering

## Anisotropic strength and deformation behavior of antigorite serpentinite

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Introduction: Recent seismic wave analysis suggest that serpentinite exists along the subducting plate. Serpentinite is important to understand about subducting oceanic plate and high-pressure metamorphic rocks. Most Antigorite serpentinite which exist on the ground show lattice preferred orientation and they have foliation. Maximum strength of the rock with foliation varies with the angle between maximum compressional axis and foliation. This angle is said to be Azimuth. Deformation experiment using the metamorphic rock, peak of the maximum strength is when Azimuth is 0 degrees and 90 degrees, and least at 30~45 degrees. (Nasseri et al., 2003) The value of strength decrease from peak to least differ from metamorphic rocks. The biggest strength decrease seen in the experiment is 75% at phyllite and the smallest is 11% at biotite schist. In this study, we have conducted axial compression deformation experiment using the solid state pressure deformation experiment apparatus to investigate the strength decrease and azimuthal anisotropy of serpentinite with foliation and lattice preferred orientation. And observed about the recovered samples to clarify the characteristics of plastic deformation of Antigorite.

Experimental: We have conducted constant strain rate experiment of Antigorite serpentinite, in order to understand the effect of microstructural anisotropy on deformation behavior. The sample is naturally deformed foliated Antigorite serpentinite which is characterized by preferential arrangement of (001) of Antigorites parallel to the foliation. We prepared three types of oriented starting samples, whose foliations were set at 0 degrees, 30 degrees and 90 degrees with respect to the axial stress. Experimental conditions were 500 C temperature at 1 GPa confining pressure with 500 um/h displacement rate of piston.

Results and Discussion: Maximum strength of the sample 30 degrees is lower than that of 0 degrees and 90 degrees. (Strength decrease is seen.) The experimental data indicate that the maximum strength of 0 degrees is 40 % bigger than that of 90 degrees, and 90 degrees is 35 % bigger than that of 30 degrees. Suggested from the micro-structure observation, sliding occurs along the foliation at sample 30 degrees and begin to fold since it cannot slide any more bound by upper and lower pistons. There is small plastic deformation area at sample 0 degrees that stress drop is suggested to be occur by brittle fracture. Those of 90 degrees were due to plastic deformation of antigorite itself and extensional breakage of antigorite grains, respectively. These experimental result show that serpentinite has very strong azimuthal dependence. Noticeable stress drop could occur at subducting plate in case serpentinite has lattice preferred orientation by shear stress.

Keywords: Antigorite Serpentinite, Deformation Experiment, SEM, Deformation Mechanism



## Coalescence and Zener pinning of mineral grains in mylonite

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Recently, we have succeeded in demonstrating true superplasticity in geological materials (Hiraga et al. 2010). Superplastic deformation is commonly considered to proceed via grain boundary sliding (GBS) which results in grain switching in the samples. As a result, initial equigranular shape of grains can be remained even after severe deformation of the sample, which is one of the characteristic microstructure of superplastically deformed materials. Further, due to large contribution of intergranular deformation on total strain, development of crystallographic preferred orientation in the materials is not expected either. Thus, it is very difficult to recognize the operation of GBS in microstructure of the deformed rocks. Hiraga et al. (2010) showed coalescence of periclase grains almost perpendicular to the tensile direction after superplastic deformation in forsterite + periclase aggregates, although the paper focused on the deformation enhanced grain growth during superplastic deformation. Here we present more details of the coalescence microstructure and compare it with that of ultramylonite, often considered to have deformed by GBS creep. We show the microstructures of coalescence of similar mineral phases and Zener controlled grain sizes. Such observations indicate pervasive operation of GBS in the earth's mantle and crust.

Keywords: Mylonite, Grain size, Coalescence, Zener relation, Superplasticity

## Exploration of microstructure induced by ultra low strain rate in mantle derived olivine

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Kitamura et al. (1986) and Ando et al. (2001) found Fe concentration on dislocation core created in olivine grains of deformed alpine type peridotite. They believe that the Fe concentration occurs during dislocation creep at very low strain rate condition in the upper mantle. This phenomenon is known as Cottrell atmosphere in the material science. On the basis of this detection phenomenon, they demonstrate that the study of ultra low strain rate effect on olivine plasticity is a very important to understand the dynamics of the upper mantle.

We try to confirm whether Fe concentration on dislocation core is a common phenomenon in deformed olivine grains of mantle derived peridotite. We are now observing microstructures of three types of peridotite, namely xenoliths from basalt and kimberlite and alpine, by using optical microscopy, EPMA, EBSD, TEM, ATEM, and STEM technique.

We obtained presently the following results. The Fe concentration is detected in the alpine type peridotites collected from Uenzaru, Horoman and Oman, but not in the xenoliths of basalt collected from Takashima, Megata, Kurose and Salt Lake. Microstructural observations can explain the later result by three different possibilities: Fe concentration on dislocation core is not occurred in olivine grains 1) in whole upper mantle, 2) only in the high stress (namely high strain rate) region of the upper mantle, and 3) it diffused away due to static recovery in basaltic magma.

Reference : Ando et al. (2001) *Nature*, 414, 893. Jung et al. (2006) *Tectonophysics*, 421, 1. Kitamura et al. (1986) *Proc. Japan Acad.*, 62, 149.

Keywords: Olivine, Cottrell Atmosphere, Dislocation Creep, Lattice Preferred Orientation

## The compression experiments on forsterite-melt system

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It is known that the earth mantle has anisotropy in seismic wave velocity (Tanimoto and Anderson, 1984). Fast direction of the wave propagation corresponds approximately to the direction of the plate motion, indicating that the mantle anisotropy is originated from the mantle flow. As the mantle flow, olivine crystallographic axes might align to the specific direction resulting in the formation of anisotropy in the wave velocity. Partial melt can be present where the olivine deforms significantly in the mantle so that it is required to know how olivine grains acquire lattice preferred orientation (LPO) under the presence of melt experimentally.

We conducted compression creep experiments on forsterite + anorthitic melt samples with a dimension of  $\phi 10 \times 10$  mm. We used an Instron type deformation testing machine equipped with a vertical furnace. Temperature condition of 1270 degrees (Celsius) and strain rate of  $10^{-6} \sim 10^{-8}$  /s were used. During the experiment, most of the samples exhibited the strain hardening. Microstructure observations after the tests revealed the occurrence of the significant grain growth during the tests. We attribute the hardening to the increase of the grain size. The samples demonstrated strain weakening after the hardening stage. Such weakening is possibly due to the crack formation in samples. After the deformation, well polished sample sections were prepared for SEM/EBSD analysis. We found b-axis alignment of forsterite parallel to the compression direction. Since creep strength of the sample is grain size sensitive, the samples are estimated to have deformed via diffusion creep mechanism. LPO development under the deformation mechanism should be explored.

Keywords: forsterite-melt system, EBSD, creep, LPO

## Superplasticity in hydrous melt-bearing dunite: Implications for shear localization in Earth's upper mantle

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The rheological properties of olivine, the major constituent mineral in Earth's upper mantle, control the dynamics of the upper mantle. Many experimental studies have been performed on the plastic flow behaviors of olivine at high temperatures (i.e., temperatures at the upper mantle) and low pressures (< 0.5 GPa) (e.g., Durham and Goetze, 1977). Previous studies showed that the plastic flow of olivine at high temperatures ( $T > 1500$  K) is controlled by two creep mechanisms, power-law dislocation creep and diffusion creep (e.g., Karato et al., 1986). Some authors argued that other creep mechanisms such as dislocation-accommodated grain boundary sliding and diffusion-accommodated grain boundary sliding also play an important role in the upper mantle (e.g., Hirth and Kohlstedt, 1995). Both of them (dislocation- and diffusion-accommodated grain boundary sliding) are often termed as superplasticity. It has been reported that superplasticity may dominate the plastic flow of minerals in some parts of the Earth (glaciers: Goldsby and Kohlstedt, 2001; shear zones in the lower crust: e.g., Behrmann and Mainprice, 1987; shear zones in the upper mantle: Hiraga et al., 2010; lower mantle: Karato et al., 1995).

It has been reported that intergranular melt/fluid phases decrease the creep strength of olivine. In the olivine-basalt system, power-law dislocation creep and diffusion creep are enhanced by the presence of a melt phase (Mei et al., 2002). Moreover, it has been reported that grain boundary sliding (GBS) dominates the deformation of olivine in the olivine-basalt system with a high volume fraction of melt (> 4 vol.%) (Hirth and Kohlstedt, 1995). Similar observations have been reported in aqueous fluid-bearing peridotites (McDonnell et al., 2000). It is known that the dihedral angle between olivine and fluid decreases with pressure (Mibe et al., 1999; Yoshino et al., 2007), which shows a reduction in the solid-solid grain boundary area with increase in pressure. Thus, a significant weakening of olivine aggregates by addition of fluids is expected at high pressures. However, the effects of intergranular fluids on the creep strength of olivine aggregates have not been evaluated at high pressures (pressure range in previous studies: 0.3-0.6 GPa).

In order to explore the rheological properties of fluid-bearing dunite (i.e., olivine aggregate) under the conditions of Earth's upper mantle, we conducted deformation experiments on hydrous melt-bearing dunite (olivine + 4 vol.% orthopyroxene + 4 vol.% clinopyroxene with less than 2.5 vol.% of the melt phase) were conducted at pressures of 1.3-5.7 GPa and temperatures of 1270-1490 K. The strain rate was proportional to steady-state creep strength to the 2.1 power, and the creep strength markedly increased with increase in grain size. Developments of the crystallographic preferred orientation of olivine and flattening of olivine grains were hardly observed even after 33-55 % shortening of the samples. These observations show that grain boundary sliding (GBS) dominated the deformation of olivine (i.e., superplasticity). The creep strength of hydrous melt-bearing dunite was 2-5 times lower than that of melt-free dunite. Superplasticity is the dominant creep mechanism of olivine in fluid-bearing fine-grained peridotites under low-temperature and high-stress conditions (i.e., peridotite shear zones in the upper mantle). Superplasticity induced by geological fluids would play an important role in the shear localization (and thus initiation of subduction) in the upper mantle.

Keywords: olivine, hydrous melt, grain boundary sliding, superplasticity, shear localization, subduction

## Simultaneous analysis of strain and texture of polycrystalline materials using two-dimensional X-ray diffraction pattern

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Angle dispersive powder X-ray diffraction experiments using a two-dimensional area detector are one of the most powerful methods for Earth material sciences, particularly under high pressure conditions. A two-dimensional intensity distribution on Debye rings immediately involves information of the number of crystallites, lattice preferred orientation (LPO) and lattice strains under stress; i.e. the number of crystallites is directly related with the spottiness on the Debye rings. When a LPO is developed, diffracted intensities along the Debye rings show circumferential oscillations unique to a manner of the three-dimensional orientation distribution. The effect of lattice strain appears in elliptic distortions of the each ring or a deviation of the original crystallographic geometry between rings. These are substantial factors of the bulk physical properties of a polycrystalline material, including seismic velocity, thermal/electric conductivity and so on, but in many cases the quantitative treatments have not yet been developed into a standard technique.

In the present study, the author developed a software code, which simulates a two-dimensional diffraction pattern based on given experimental parameters (e.g. wave length, beam convergence, camera length, pixel size of a detector, and so on) and (poly)crystalline properties (crystal structure, crystallite number, size, orientation distribution, lattice strain and so on). Through the simulation, the effect of the parameters on the diffraction pattern can be quantitatively and visually estimated. Furthermore, to find the orientation distribution and the lattice strain from the observed diffraction pattern, a fitting procedure is incorporated into the code as follows: The software initially generates a large number ( $10^6$ - $10^7$ ) of crystallites with random orientation under a strain-free condition, evaluates the residual of the simulated/observed patterns, and then iteratively modifies the orientation distribution and stress condition. In each iterative step, randomly selected crystallites (0.1-1.0 %) are roughly orientated toward a randomly generated direction, and the modified distribution are (or not) adopted if the residual becomes small (or large). Components of stress tensor are also modified at several intervals. By repeating the iteration step many times ( $>10^5$ ), the simulation seems to converge to a certain condition. A pole figure (density map of a crystallographic axis or plane direction) can be calculated from the obtained orientation distribution. The fitting procedure was applied for several actual diffraction patterns from in-situ uniaxial compression experiments, and its validity was confirmed.

Keywords: X-ray powder diffraction, lattice preferred orientation, lattice strain, two-dimensional detector, polycrystalline material

## Brine distribution in halite rocks - Inference from measured electrical conductivity

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Intercrystalline fluid can significantly affect rheological and transport properties of rocks. Its influences are strongly dependent on its distribution. The dihedral angle between solid and liquid phases has been widely accepted as a key parameter that controls solid-liquid textures. The liquid phase is not expected to be interconnected if the dihedral angle is larger than 60 degrees. However, observations contradictory to dihedral angle values have been reported. The grain boundary fluid coexists with a positive dihedral angle. Similar thin fluid films might exist in grain boundaries of crustal materials, and play important roles in crustal processes. In order to understand the nature of grain boundary fluid, we measured electrical conductivity of synthetic wet halite rocks at various temperature and pressure conditions.

Halite-water system is used as an analog for crustal rocks. The dihedral angle has been studied systematically at various pressure and temperature conditions. The dihedral angle is larger than 60 degrees at lower pressure and temperature. It decreases to be less than 60 degrees with increasing pressure and temperature. A sample is prepared by cold-pressing (140MPa for 20 minutes) and annealing (T=160C and P=180MPa for 20 hours) of wet NaCl powder. Grains are polygonal and equidimensional with diameters of 50-100 micrometers.

Experiments are performed using a conventional cold-seal vessel with an external heater. The pressure medium is silicon oil (viscosity=0.1 Pa s). Dimensions of a sample are 9 mm in diameter and 7 mm in length. Viton is used as a jacketing material. Platinum electrodes are placed at both ends of the sample. The confining pressure of 30 MPa is first applied to a sample, and then the temperature is increased to 120C and kept for 5days. The temperature is then changed to 180C (162C@sample) and 140C (126C@sample). Electrical impedance of the sample is measured at different pressures. Impedance measurements are made with an LCR meter (NF ZM2353) (40Hz to 200kHz), and a lock-in amplifier (SRS SR830) and a current amplifier (SRS SR570) (40mHz to 400Hz). Debye-type impedance spectra are observed, to which a parallel array of a capacitor and a resistor can be applied as an equivalent circuit. Measured resistance is converted to conductivity.

Measured conductivity is higher than the conductivity of NaCl by 2-3 orders of magnitude, implying that the electrical conduction is dominated by that through brine. Quasi-stationary conductivity observed at T=180C is almost independent of the pressure. This is consistent with a slight change in the dihedral angle with the pressure. The connectivity of brine should mainly be governed by the triple junction tubes, which are difficult to deform. If the interconnection is governed by grain boundary fluid films, the conductivity should be very sensitive to the pressure increase.

The progressive decrease in conductivity at 140C and P<50MPa reflects the increase in the dihedral angle, while the increase in conductivity at 100MPa the decrease in the dihedral angle.

Even at conditions of the dihedral angle larger than 60 degrees (e.g., T=140C, P=30MPa), brine is interconnected. However, we cannot say that connected paths are stable or not. No stationary value is observed. Longer runs should be done to study the connectivity of brine at lower temperature and pressure conditions.

Keywords: electrical conductivity, halite rocks, dihedral angle, grain boundary fluid, fluid distribution

## Experimental study of seismic attenuation by using a rock analogue

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The purpose of this study is to investigate the high-temperature anelasticity of rocks in the seismic frequency range. In order to estimate temperature anomaly and melt distribution in the upper mantle quantitatively by inversion analyses of three dimensional seismic velocity structures, it is absolutely necessary to understand the anelasticity of mantle rocks in detail. Anelasticity is the transitional property between elasticity and viscosity. Change of seismic velocity is caused both by the anharmonic effect and the anelastic effect. The former can be evaluated quantitatively but the latter can not. To better understand the anelastic effect, it is important to measure anelasticity as functions of frequency, temperature, grain size and melt fraction. By using custom fabricated forced oscillation apparatus[Takei et al., 2011], we have measured the viscoelastic properties of polycrystalline organic borneol as an analogue to mantle rock.

In previous studies[McCarthy et al., 2011; McCarthy and Takei, 2011], dispersion of Young's Modulus  $E$  and spectrum of attenuation  $Q^{-1}$  were measured as functions of temperature, grain size and melt fraction over a broad frequency range ( $10^{-4} < f$  (Hz)  $< 2.15$ ). Using viscosity  $\eta$  measured at each temperature, grain size and melt fraction, the Maxwell frequency  $f_M = E_U/\eta$  was calculated, where  $E_U$  represents the unrelaxed Young's modulus measured at ultrasonic frequency. When  $Q^{-1}$  spectrum was plotted as a function of normalized frequency  $f/f_M$ , all  $Q^{-1}$  spectra collapsed onto a single master curve. The  $Q^{-1}$  spectra obtained from olivine aggregates[Gribb and Cooper, 1998; Jackson et al, 2002] also collapsed onto the same master curve, when plotted as a function of  $f/f_M$ . This efficiency of the Maxwell frequency scaling strongly suggests that the dominant mechanism of anelasticity in the experimental frequency range is "diffusionally accommodated grain boundary sliding". However, the seismic frequencies normalized by the Maxwell frequency in the mantle are considerably higher than the experimentally measured frequencies. Therefore, experiments at higher frequencies, lower temperatures and larger grain size would be needed.

In this study, in order to measure anelasticity at higher normalized frequency, we have modified the apparatus by using high-speed displacement meters that handle higher sampling rate, and low temperature incubator. At higher frequency, lower temperature and/or larger grain size,  $Q^{-1}$  of the sample is low. In order to measure low  $Q^{-1}$  accurately, we found it important to improve the rigidity of the apparatus. Also, small time delay of load and displacement meters comes to the issue. So we have to measure the delay accurately. The effect of the delay on attenuation is nonnegligible at high frequency. Based on the result of this calibration, we will check the validity of the high frequency part of the  $Q^{-1}$  spectrum in our previous study.

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Keywords: anelasticity, seismic attenuation

## Estimates of internal friction by Sompi spectral analysis for resonant sphere spectroscopy

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The Resonant Sphere Technique (RST) is a powerful tool to measure elastic and anelastic properties of a solid sample. In RST, free oscillations of the sample are excited by impulsive input, and the output waveform data are acquired as a function of time (FT method). Generally, the resonant spectrum has been obtained by spectral analysis using Fast Fourier Transform (FFT) in which the degree of dissipation of the vibration energy (internal friction) is measured by the broadening of the resonance peaks. As reported in the previous conference of the Japan Geoscience Union Meeting (Yamamoto et al., 2011), we applied the Sompi method to RST data and determined elastic moduli and internal friction. Yamamoto et al. (2011) concluded that internal friction  $Q_{12}^{-1}$  of a single crystal MgO by the Sompi spectral analysis demonstrates positive value although our FFT analysis as well as previous works (Sumino et al., 1976; Oda et al., 1994) showed negative values. This suggests that the Sompi analysis for RST may be more effective than FFT technique for estimates of internal friction values. However, we found the strong dependence of internal friction values on the number of modes used for the estimates of internal friction. We acquired the waveform data by changing the force holding the specimen in order to extrapolate to zero-force. The data were analysed by both FFT and Sompi methods, and frequencies and half-widths (FFT)/decaying rates (Sompi) at zero-force were estimated. The modes with enough amplitude and good reproducibility were selected, and the internal friction parameters were estimated according to Sumino et al. (1976). The results show that the errors in internal friction determined using both FFT and Sompi method become smaller than those reported by Yamamoto et al. (2011). The present study suggests that Sompi analysis may be more effective to estimate internal friction.

Keywords: Resonant Sphere Technique, Sompi method, internal friction, elastic constant