Japan Geoscience Union Meeting 2013
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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 alterated 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 W vs S, respectively. In the cataclasite samples, since the change of SiO2 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

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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

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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

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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 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 (antigolite) 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^o 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 precut 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 um/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.
This research is supported by Grant-in-Aid for Scientific Research on Innovative Areas, KAKENHI (21109005), JSPS.

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

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

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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 ($CaAl_2Si_2O_8$) and, two phase composite of anorthite + diopside ($CaMgSi_2O_6$), anorthite + quartz (SiO_2) with homogeneous microstructure, which are good analogues for lower crustal composites.

Keywords: polycrystal, lower crust, mineral

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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 spices 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, MgO (periclase) + $MgSiO_3$ (enstatite) => Mg_2SiO_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 judjed 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 cm^3mol^{-1} for lattice diffusion and 10 cm^3mol^{-1} for grain boundary diffusion, respectively.

Keywords: forsterite, high pressure, rim growth, diffusion

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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). Bystrikcy 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-1 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 um) 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-1). 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

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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-confine 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-heted diamond anvil cell (LHDAC). Starting materials are single crystals of natural olivine (San Carlos, USA) and pyrope garnet (Czecho). They were thinned to have a wedge-shaped plate and single-sided coated with metaric 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 generate 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 wasleyite and/or ringwoodite in the laser-scanned area up to 23GPa and 1600K. The result of Hall-Williamson analysys (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 scaning electron microscope. High-pressure phase in the heated sample was localized on the contact region between two plates. No significant texual 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. Planner 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

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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 ˜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 ˜0.1 mm). Another is in-situ X-ray observation of constant strain rate deformation of polycrystalline antigorite cylinder cored from high-temperature serpetinite (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 (˜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 $3 * 10^{-5}$ s^{-1} at pressures of ˜0.1-3 GPa. We observed that the constant flow stress of ˜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

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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.