Japan Geoscience Union Meeting 2012
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SMP47-P01 Room:Convention Hall Time:May 24 13:45-15:15

The formation process of the oligoclases and ternary feldspars in the felsic gneiss from Mt. Riiser-Larsen in Napier Com

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Mt. Riiser-Larsen, East Antarctica is the one component of the Napier Complex. The Napier Complex consists of granulite-facies metamorphic rocks formed by multiple thermal events, including Late-Archean ultrahigh temperature metamorphism(Harley and Black, 1997). Based on ternary feldspar solvus models, the bulk compositions of ternary feldspars in felsic gneiss from Mt. Riiser-Larsen yield the minimum of metamorphic temperatures ranging from 1000 to 1110C(Hokada, 2001). TH97012006(hereafter shortend to 12006) is the garnet-porphyroblast-bearing portion of a garnet-bearing felsic gneiss from Mt. Riiser Larsen. This sample is almost composed of oligoclase(An29Ab70Or1; hereafter shortend to Olg), and anti to mesoperthitic ternary feldspar(hereafter shortend to TF) composed of Olg lamellae and orthoclase(An2Ab8Or90; hereafter shortend to Or). The mode of occurrence and textures of Olg grains and TF grains are heterogeneous.
We observed the micro-textures of TF grains using by transmission electron microscope(TEM) and revealed the formation process of the common type of micro-textures of TF grains in 12006, (-901) exsolution lamellae of TF grains. But it became apparent that some micro-textures were not explained by (-901) exsolution lamellae and coexistence of Olg grains and TF grains was not explained.

In this study, some elemental analyses and cathodo luminescence (CL) observations on Olg grains and TF grains that have different exsolution lamellae in 12006 were carried out using by scanning electron microprobe(SEM) and wavelength-dispersive X-ray spectroscopy(WDX), and more detailed formation process of these feldspars in 12006 was suggested.

In this sample, Olg grains and TF grains are distributed heterogeneously. Hokada(2001) and Kodama(2010, Annual Meeting of Japan Association of Mineralogical Sciences) reported that no significant compositional difference was found between the chemical compositions of Olg grain and those of Olg lamellae in TF grains. However, K-rich Olg grains were found in this study. Significant differences in distribution of K and CL are observed between some Olg grains. Therefore, these Olg grains formed at different stages. This could result from the following process; First, K-free Olg grains and K-rich melt arose from Olg-rich protolith by partial melting caused by metamorphism. After that, during cooling process, K-rich Olg grains crystalized from melt.
On the other hand, TF grains that have An23Ab56Or21 as bulk composition, and are composed of coarse Olg lamellae about 100um in width and TF lamellae-like textures that are composed of (-901) fine Olg lamellae and Or lamellae up to 10um in width(hereafter shortend to TF lamellae) were found. The TF lamellae are consistent with common TF grains in the orientation of Olg-Or boundaries and the bulk composition(TF lamellae: An17Ab43Or40, common TF grains: An21Ab48Or31). Therefore, these TF lamellae were formed by spinodal decomposition too. Orientation of boundaries of coarse Olg lamellae and Or lamellae is not consistent with (-901),and the scale of these TF lamellae almost correspond to coarse oligoclase lamellae. Therefore, these textures suggest that coarse Olg and TF lamellae decomposed at higher temperature, and after, fine Olg and Or lamellae decomposed from coarse TF lamellae by spinodal composition at lower temperature. However, the former decomposition is not explained by any decompositional process in current phase diagram. For more informations about these textures, the reexamination of phase relationships of An-Ab-Or system at high temperature is needed.

Keywords: ternary feldspar, exsolution texture

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Measurements of the vacant sites in crystal structure of magnetite by Mossbauer spectroscopy

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Magnetite ($[Fe^{3+}][Fe^{3+}\ Fe^{2+}]O_4$) is a strongly magnetic oxide iron mineral. When magnetite undergoes low temperature oxidation, vacant sites are formed at octahedral sites, and magnetite changes to maghemite($(Fe^{3+}][Fe^{3+}\ {Fe^{2+}}_{2/3}]\ X_{1/3}\ O_4$ (X shows vacant sites.). Magnetite changes to maghemite with increasing vacant sites, so a degree of low temperature oxidation can be estimated from the vacant sites. According to Nishitani and Kono (1982), grain size controls the transformation of titanomagnetites.When the grain size of titanomagnetite is less than 1 micrometer, titanomagnetite transforms to titanomaghemite under low temperature heating.However, titanomagnetite larger than 1 micrometer breaks into Fe-rich titanomagnetite and Ti-rich ilmenite under low temperature heating .In this case, titanomagnetite suffers high temperature oxidation.Nishitani and Kono(1982) estimated the degree of low temperature oxidation of titanomaghemite by X-ray diffractometer and Curie temperature. Another method to estimate vacant sites is a Mossbauer spectroscopy.Mossbauer spectroscopy is a sensitive method to estimate degree of low temperature oxidation than X-ray diffraction.

In this study, we measured Mossbauer spectra of natural titanomagnetite of Oarai, Ibaragi Prefecture,and estimated vacant sites in titanomagnetite.Titanomagnetite samples of various grain diameter were heated at fixed time and temperature.After measuring those heated samples by X-ray diffractometer to distinguish low temperature oxidation samples from high temperature oxidation samples, Mossbauer spectra of samples which underwent low temperature oxidation were measured .The vacant sites in titanomagnetite is calculated from the results of Mossbauer spectra.

Natural magnetite has solid solution between Fe_3O_4 , Fe_2TiO_4 and $FeAl_2O_4$ .Ti^{4+} and Al^{3+} must be considered to estimate vacant site of titanomagnetite by Mossbauer spectra, because Ti^{4+} and Al^{3+} must be vacant for Mossbauer spectra.However, method of correction remains unsolved.Therefore, we considered a new correction method.

Keywords: Mossbauer spectroscopy, Magnetite, TitanoMagnetite

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High-pressure X-ray absorption measurement of FeS: K-edge absorption spectra of Fe

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Iron sulfide (FeS) is important material to consider the cores of terrestrial planets such as Earth and Mars. FeS has been known to undergo several structural and electro-magnetic phase transformations with increasing with pressure and temperature. FeS takes a troilite structure (FeS I) and antiferromagnetic insulator (T_N = 598 K) at ambient condition, and it undergoes three pressure-induced transitions from FeS I to FeS II (orthorhombic MnP related structure) above 3.4 GPa and then to FeS III (monoclinic phase), and further transition to FeS VI reported recently above 36 GPa. The structural transition from FeS II to III is accompanied by a loss of magnetic ordering induced by high-spin to low-spin transition of Fe. In this study, we observed electronic state of Fe in high pressure by X-ray absorption near edge structure (XANES) experiment combined with diamond anvil cell (DAC), for understanding the relation between structural transition up to FeS VI and electronic transition.

X-ray absorption experiments under high pressure were performed at the BL-3A in KEK-PF, Tsukuba, Japan. We compressed powdered FeS sample with NaCl and measured X-ray energy dependence of absorption in the range of X-ray absorption near edge structure (7.0-7.2 keV) until 47 GPa. It was irradiated in three different beam diameters 0.1 mm, 0.3 mm and over 0.5 mm. We analyzed the difference of spectral patterns caused by change of structure and beam size. Absorption edges did not show a significant difference in each structure. But the pre-edge of absorption edge became sharp with structural transformation to high pressure phase.

Keywords: FeS, XANES

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Measurements of Elastic Constants of Single-crystal Garnet by High Frequency Resonant Ultrasound Spectroscopy

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Garnet is a major constituent mineral in the lower continental crust, upper mantle, and subducted slab. Gabbroic rock in slab transforms to denser elcogitic rock along with subduction. This larger density, which gives a negative buoyancy to slab, is attributed to the existence of garnet. The abundance of garnet is thus a key to understand the dynamics of subduction. Elastic property of garnet is critical to constrain its abundance in subducted slab from seismic observations. We thus have studied elastic constants of a single-crystal garnet using a resonance method.

We used a natural single-crystal garnet of composition Alm80Py16Sps4 (source unknown). It was selected in terms of the uniformity of crystallographic orientation examined by SEM-EBSD. The orientation of the single crystal was determined by the X-ray precession method. The crystal was shaped into a rectangular parallelepiped (0.890x0.690x0.440 mm^3). Each face was polished flat ($<$ 1 micrometer) in an orientation parallel or perpendicular to $\{100\}$. The density, which is calculated from the chemical composition analyzed with EPMA and the lattice parameter (a= 1.154(1) nm) measured by XRD, is 4.091(8) x 10^3 kg/m^3.

The resonance spectrum was obtained from 3 to 11 MHz by using a measurement system specially designed for such a small sample (Yoneda et al., 2007, JJAP). Elastic constants were determined by repeatedly comparing the obtained resonance spectrum with a theoretical one, which was calculated for trial values of elastic constants. C_{11}, C_{12}, and C_{44} are determined to be 296.0, 111.2, and 94.4 (GPa), respectively. These are quite close to values previously reported for garnet single crystals with similar compositions. We will also report the temperature dependence of elastic constants in this presentation.

Keywords: elastic constant, resonance method, garnet, EBSD, SEM

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Crystal structure refinement of ferrihydrite using atomic pair distribution function (PDF) method

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Ferrihydrite is a short-range ordered nano-crystalline iron (III) oxyhydroxide that has been recognized to play an important role in iron cycling in environmental, biological, and geochemical processes. Despite intensive research for the two last decades, the exact atomic structure of ferrihydrite is not fully established and is still matter of debate. Atomic pair distribution function (PDF) analysis is a powerful technique to obtain structural information of nanoparticulate materials. Michel et al. (2007) have recently presented a structure for ferrihydrite that was determined using the PDF method and synchrotron X-ray scattering technique. In the study, the PDF analysis is used to investigate and compare the structure of 6-line ferrihydrite and dehydrated ferrihydrite with the results of Michel et al. (2007). Ferrihydrite was synthesized by the method of Schwertmann and Cornell (1991). TEM observations show that the particle size of the ferrihydrite is highly uniform with a size distribution between 3 nm and 5 nm. X-ray measurements were performed on a Rigaku RAXIS RAPID imaging plate area detector with sealed-tube X-ray source (Mo-Kalpha radiation, lambda = 0.71073 A, graphite monochromated) operating at 50 kV and 40 mA. The X-ray total scattering data were collected up to 2theta = 72.2 degree that corresponds to the value of Qmax = 10.4 A-1. The crystal structure of ferrihydrite was refined in space group P63mc, with a = 5.974(2), c = 9.130(2) A, V = 282.18 A3, spherical diameter = 2.09 (1) nm, and that of ferrihydrite heated was with a = 5.924(1), c = 9.130(2) A, V = 277.48 A3, spherical diameter = 2.002 (4) nm, respectively. The crystal structures are composed of edge-sharing FeO6 octahedra and corner-sharing FeO4 tetrahedra, which form a two-dimensional layer-like structure perpendicular to the c axis. The tetrahedra exhibit significantly distorted configuration such that three oxygen atoms are found at a distance of 1.96 A and the other at 2.08 A. The dehydration process causes a unit cell contraction accompanied by a shrinkage of the a lattice parameter. The release of water molecules enhances the bond length and angular distortions of the octahedra and tetrahedra in ferrihydrite.

Keywords: Ferryhidrite, Nano-mineral, Transmission electron microscope, Atomic pair distribution function (PDF) method, Crystal structure

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Transformation mechanism of the sodalite-to-cancrinite phase transformation in oxalate-bearing solution

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Cancrnite, $Ca_6Ca_2(AlSiO_4)_6(CO_3)_2(H_2O)_2$], is one of feldspathoid minerals occrring in alkaline rocks. The crystal structure of cancrinite consists of three-dimensional aluminosilicate framework in which various cations (e.g., Na^+, K^+, Ca^{2+}) and anions (e.g., $CO_3{}^{2-}$, Cl^-, OH^-, $SO_4{}^{2-}$, $NO_3{}^-$, $C_2O_4{}^{2-}$) are occluded. Recently, Chukanov et al. (2010) reported a new cancrintie group mineral, kyanoxalite [$Na_7(AlSiO_4)_6(C_2O_4)_{0.5-1.0}(H_2O)_5$], from the Khibiny?Lovozero Alkaline Complex,Kola Peninsula, Russia. The authors mentioned that kyanoxalite was formed by hydrothemal alternation of sodalite [$Na_4(AlSiO_4)_3Cl$], however, experimetanl evidence of the transformation of sodalite into cancrinite was not provided. In this study, we synthesis oxalate-bearing cancrinite and discuss the fomation mechanism.

Keywords: Cancrinite, Sodalite, Oxalate

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Pressure dependance of swelling state of Na-montmorillonite

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It is known that montmorillonite clay minerals have the property of swelling with water molecules hydrated to interlayer cations. Clay minerals present on and under the Earth’s surface, forming a card-house structure consisting of montmorillonite stacks, and they hold interlayer water and external pore water. The relative abundance of interlayer water and external pore water contributes the macro-scale material properties such as permeability and viscosity. Sample preparation is following. Bentonite sample used in this study is mined from Tsukinuno Mine, Yamagata Prefecture, and it is purified to obtain montmorillonite (Kunipia-F, Kunimine Industry Co., Ltd.). Na-montmorillonite, of which interlayer cation was replaced by sodium ion, is used in this study. Dry Na-montmorillonite and pure water with appropriate ratio were sealed in a sample tube. Water contents were measured after experiments. Water content is between 30 ˜ 50 wt%, and they have properties between highly viscous fluid and solid. Relation between dry density, water content and swelling state is observed. At small water content (˜30wt%), swelling state is a 2-layer hydration state, and a 3-layer hydration state at the large water content(˜50%). At intermediate water contents, 2-layer and 3-layer hydration state coexist. Experimental procedure is following. Basal spacings of Na-montmorillonite is examined using in-situ X-ray diffraction methods. To obtain the temperature and pressure conditions near the earth’s surface (a depth of up to 1 km) in the laboratory, the environment-control system was used for in-situ X-ray diffraction methods. The experiments were carried out to varying sample temperature from 30 to 80 degree at 10 MPa, and sample pressure from 10 MPa to 50 MPa at 30 degree. The exchange of water between interlayer and external pore was determined from swelling state change due to pressure and temperature. As a result, the swelling states, which is determined strongly by the water content or dry density, changes slightly due to pressure and temperature. By increasing temperature, there is a tendency that swelling state changes from 3-layer to 2-layer. Temperature dependance of swelling state is mainly on entropy. So, by increasing temperature, water moves from interlayer to external pore, which has higher entropy, and swelling state changes from 3-layer to 2-layer. Pressure dependance is mainly on molar volume, but there is almost no difference between the volume of interlayer water and external pore water. So, it is difficult to observe the pressure dependance. We are going to observe pressure dependance at higher than 50 MPa of swelling state.

Keywords: Montmorillonite, Smectite, Fault gouge, Radioactive waste

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Molecular dynamics simulations of sodium tri-silicate melt under high pressure

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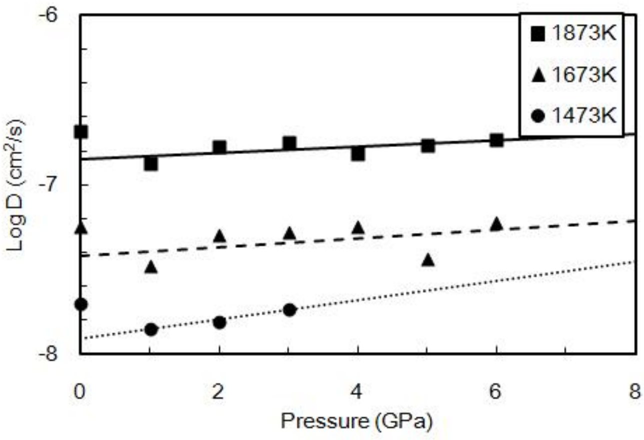
To understand volcanic process, genesis of magma, its ascent, and terrestrial magma ocean, the properties of silicate melts under high pressure must be investigated. The properties of silicate melts show peculiar behaviors under high pressures. It is reported that viscosity of acidic silicate melts decreases with increasing pressure (Scarfe et. al 1979). Moreover, it is reported that self diffusion coefficients of O and Si increases with increasing pressure (Rubie et al. 1993). These show that silicate melts soften under high pressure. In this work, the relationship between the structure and properties of Na_2O3SiO_2melt was investigated by molecular dynamics (MD) simulations. In MD simulations, it is possible to obtain changing configuration of every atom as a function of time. It is also possible it identify the individual atoms or groups of atoms which play a deciding role in diffusion processes.

MD simulations were performed with NPT ensemble (N = 8002) using MXDORTO. Pressure range is 0.1 MPa to 6 GPa (Every 1 GPa). Temperature range is 1473K to 2073K (Every 200K). Time step of equations of motion of atoms is 0.5 fs. The inter-atomic potential model implicitly includes Coulomb potential, short-range repulsion, van der Waals force and covalent terms. Starting from a random coordination and velocities, all the atoms are moved in Newtonian equation maintaining isobaric and isothermal through 2 ns as structural relaxation. After relaxation, statistical averages of physical properties were obtained through 200ps.

In 0 to 2 GPa, the density increases rapidly and Q^4 species increases with increasing pressure. This might means, the polymerization affect to the melt density in that pressure range. Shrinking of Si-O network is composed from increasing large membered rings and decreasing Si-O-Si angles. Small membered rings have small degree of freedom of deformation and large membered rings have large degree of freedom of deformation. Consequently, large membered rings might be easy to deform. In the MD simulations, the number of small membered rings decreases with increasing pressure and the number of large membered rings increases with increasing pressure. Adding to that, Si-O-Si angle decreases with increasing pressure. Consequently, this might be said that degree of freedom of deformation of Si-O network increases under high pressure and Si-O network is collapsed. Densification of Na_2O3SiO_2 melt is occur as a result of that.

In MD simulation results, the self diffusion coefficient of oxygen decreases up to 1 GPa, then increases with increasing pressure. Using Einstein-Stokes relation, viscosity increases up to 1 GPa, then decreases with increasing pressure since self diffusion coefficient of oxygen controls the viscosity of silicate melts. The anomalous pressure dependence might be caused by distortion of Si-O network. One obvious structural distortion is decreasing of Si-O-Si angle. Si-O-Si angle become smaller than stable angle when Si-O-Si angle decreases under high pressure. And then, bridging oxygen becomes instable. Adding to that, distance between SiO_4 tetrahedra shorten. This structural change might activate diffusion of oxygen and SiO_4 tetrahedra. The other obvious structural distortion is decreasing of O-Si-O angle. The average O-Si-O angle decreases from the angle of regular tetrahedra with increasing pressure in MD simulations. Oxygen becomes instable when tetrahedra distorts because of repulsion between oxygen and distortion of sp^3orbital.

Keywords: Molecular dynamics simulation, silicate melt, high pressure



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Properties of water in rocks and minerals

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Water is ubiquitously distributed in the interior of the earth, forming various species. In rocks, water is trapped at intergranular regions as the form of H2O fluid which are constructed by clustered H2O molecules and as the form of -OH in mineral crystal structures. Also, in open cavities which are constructed by crystal structures, H2O molecules are not clustered but isolated H2O molecules are incorporated. It is well known that these water species contribute to earth dynamics such as reactions and deformation of minerals (recent studies of these subjects are summarized in Dysthe & Wogelius, 2006, Chem Geol). Based on these backgrounds, determinations of contents, distributions, states, migration rates, etc. are one of the most important subjects in earth sciences.

Infrared (IR) spectroscopy has widely been used to determine the above properties of water in rocks and minerals (See Aines & Rossman, 1984, JGR; Keppler & Smith, 2006, RiMG for IR spectra of various rocks and minerals). In this study, for advanced IR spectroscopic measurements, I will introduce high temperature in situ measurements and 2-dimensional mapping measurements based on my previous studies. First, I use chalcedonic quartz, which is constructed by microcrystalline quartz. Chalcedonic quartz contains abundant H2O fluid at intergranular regions and mainly Si-OH in quartz crystal structures. Therefore, the properties of water are easily measured by IR spectroscopy. The sample is heated from room temperature up to 500 degree C, and changes of the states by temperature changes and dehydration will be discussed. Next, I use beryl, which contains isolated H2O molecules in its open spaces of the crystal structure. Polarized IR spectra are measured at room temperature and high temperatures. Then, changes of the states of water will be compared with those of chalcedonic quartz. Finally, IR mapping measurements are performed for plastically-deformed granitoid (mylonite), and 2-dimmensional distributions of water in polymineralic rocks are determined. Then, I will discuss relations of mineral phases, species and contents of water, in comparison with the results from the analyses of the states, diffusivity by dehydration as described above. I will also discuss possible microscale processes of water in relation to the deformation mechanism.

Keywords: H_2O, -OH, grain boundary, crystal structure, infrared spectroscopy

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Effect of Al content and oxygen fugacity on water partitioning between olivine and orthopyroxene

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Water affects physical property of minerals (e.g. elemental diffusion rates, melting points). Because small amount of water plays key roles in mantle rheology, precise knowledge on partitioning of water among mantle minerals is very important in understanding the earth dynamics. Rauch and Keppler (2002) investigated effect of Al content on water solubility in orthopyroxene. Al solubility of orthopyroxene decreases with increasing pressure above 3 GPa. Thus water partitioning coefficient may change significantly above 3 GPa. Moreover, Nishihara et al. (2008) indicated that substituting mechanism of OH in a mineral changes considerably with concentration of OH. Although water partitioning has been studied by many workers under water saturated conditions, experiments under low OH concentration are very limited.

In order to investigate the partitioning coefficient of water between olivine and orthopyroxene under low OH concentration (4~200 ppm), we performed high-temperature and high-pressure experiments using Kawai-type multi-anvil apparatus (SPI-1000) and piston-cylinder apparatus at the Magma Factory, Tokyo Institute of Technology, using starting materials of natural olivine (Ol; KLB-1) and synthetic orthopyroxene with various Al content (Opx; $(Mg,Fe)_{2-x}Al_{2x}Si_{2-x}O_6$ (x=0, 0.025, 0.05)). Powdered minerals were enclosed in metal foil capsule (Ni, Mo) to form monomineralic layers with more than 300 micron meters in thickness each and put it in a $Au_{75}Pd_{25}$ capsule at pressures of 1, 3 GPa and temperature of 1300 ^{o}C. Oxygen fugacity was controlled by Ni-NiO and Mo-MoO_2 buffers. Water contents were obtained with a vacuum type Fourier transform infrared spectrometer (FT-IR6100, IRT5000). Water content of minerals was calculated based on Paterson's (1982) calibration. Run products were polished down to doubly polished slab. After polishing and prior to FT-IR analysis, samples were stored in a vacuum oven at ~120 ^{o}C over night. Detection limit in the IR spectra at 3200-4000 cm^{-1} is typically less than 1 ppm due to very low back ground of vacuum type FT-IR.

Water partitioning coefficient between Ol and Al free Opx are $D_{(Al\ free\ Opx/Ol)}$ = 1~2.3. On the other hand, that between Al bearing Opx and Ol are $D_{(Al\ bearing\ Opx/Ol)} > 4.1$. Thus $D_{(Opx/Ol)}$ becomes larger with Al content of Opx. At constant temperatures, Al solubility of orthopyroxene stays nearly constant at 1~3 GPa but becomes smaller with increasing pressure above ~3 GPa. Results of this study shows that amount of water in Opx is much larger than that in coexisting Ol below ~3 GPa. On the other hand, water content of Ol would become much larger than that of Opx above ~3 GPa. The IR spectra of Al bearing Opx show peaks broader than those of Al free Opx. Peak shapes of Al bearing Opx are similar to those of natural samples. The IR spectra of Ol in recovered samples under Ni-NiO buffer show additional OH band (3700 cm^{-1}) that are not seen in the spectra of run products under Mo-MoO_2 buffer. We are performing further high pressure experiments using Ol single crystal to avoid grain-boundary effects on adsorbing water.

Keywords: FT-IR, partitioning coefficient, upper mantle, olivine, orthopyroxene

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Iron diffusion in mantle olivine under steep temperature gradient

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Laser-heated diamond anvil cell (LHDAC) has been generally used as a major method in which we can generate high temperature and pressure conditions of the Earth's interior. However, a strong temperature gradient is formed in the sample, because only a local region can be raised to high temperature by the laser heating technique. The Soret effect is known as a phenomenon of chemical diffusion induced by a temperature gradient, which causes a change of homogeneous material to heterogeneous chemistry. The Soret diffusion in liquids has popularly been studied and is fast, while that in solids and its pressure dependence have not been well examined because the Soret effect is relatively slow and more complex in solids than in liquids. Moreover, the investigation of Soret effect in solids under high temperature and pressure may be helpful to understand the possible diffusion in the Earth.

In this study, we studied the material experienced a steep temperature gradients using LHDAC and $(Mg_{0.89},Fe_{0.11})_2SiO_4$ San Carlos olivine as the starting material. The recovered samples were examined using a Field Emission-Scanning Electron Microscope (FE-SEM) and analyzed Mg-Fe interdiffusion. Chemical heterogeneity formation due to temperature gradient was observed in different experimental conditions such as temperature gradient, heating duration and phase transition. We will report the effect of these parameters on Soret diffusion in solid.

Keywords: LHDAC, Soret effect, diffusion in solid

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Development of multi-axis DAC oscillation system for x-ray powder diffraction

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Recent advanced experimental techniques of a laser-heated diamond anvil cell (DAC) provide wide P-T conditions in the earth's interior. Particularly, angular dispersive x-ray diffraction method combining with a high brilliance synchrotron radiation (SR) x-ray enables an access to the physical properties of the earth's constituent minerals. In this method, however, there is a significant quantitative problem to be solved. The spotty diffractions caused by coarse grains, which are likely generated during laser heating experiments, give an influence on the diffracted x-ray. In fact, the inhomogeneous x-ray profile leads to unreliable structure factors in Rietveld analyses. Thus, we hardly obtain ideal Debye rings being essential for the precise structural analyses due to the difficulty in acquiring sufficient particles satisfying Bragg condition. To improve this kind of statistical problem in the x-ray experiments using DAC, we have constructed the multi-axis oscillation system for x-ray powder diffraction measurements under high-pressure.

The oscillation system equipped a goniometer which was designed after the Gandolfi oscillation camera [1,2]. The goniometer has three oscillation axes, which are horizontal (theta), vertical swivel (omega), and rotating axes (phi) perpendicular to theta. The DAC with a large angular aperture (90 deg.), which is also newly developed for the oscillation system, is inserted into the holder on phi axis. The diffracted x-rays are detected with an imaging plate. This system has been installed in SR x-ray beam lines (BL04B2 or BL10XU) at SPring-8. In this presentation, we introduce details of the experimental set-up and a few experimental results using this system.

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[2]H.H. Otto, W. Hofmann, K. Schroder, J. Appl. Cryst., 35, 13-16 (2002)

Keywords: high pressure, DAC, x-ray diffraction, Gandolfi camera, synchrotron radiation