

SMP044-P01

Room:Convention Hall

Time:May 25 14:00-16:30

Synthesis of polycrystalline sintered stishovite and its physical property measurements

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We synthesized polycrystalline sintered stishovite at pressure of 15 GPa and temperature of 1473 K. The starting material was a silica glass rod with diameter of 2.5 mm and height of 2.7 mm in. This was enclosed in a platinum capsule. The recovered sample was a whitish translucent rod with diameter of about 2 mm and height of about 2.3 mm. The recovered sample was examined by micro-focused X-ray diffraction and micro-Raman spectroscopic measurements. The results indicate that the recovered sample is a pure polycrystalline stishovite. Ultrasonic measurements were carried out at ambient conditions. We determined compressional wave velocity of 11.776 km/s and shear wave velocity of 7.174 km/s. We measured bulk density of this sintered stishovite by Archimedian method. The density is 4.282 g/cm³. Using these parameters, we calculated elastic moduli of this sample: Bulk modulus, 300 GPa; shear modulus, 220 GPa; Youngs modulus, 531 GPa; Poissons ratio, 0.204. These values are consistent with those obtained by previous studies. We have also conducted X-ray diffraction measurements under low and high temperature between 90 and 1000 K at BL02B2, SPring-8. We are going to report temperature dependence of thermal expansivity of stishovite in the session.

Keywords: stishovite, sintered polycrystalline sample, elastic wave velocity, thermal expansivity

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Anomalous behavior of low-density SiO₂ in helium under high pressure

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Properties of SiO₂ crystals and glass are one of the most classical and important issues in Earth science. Numerous studies on the high-pressure behavior of these materials have been conducted because of their importance not only in Earth science but also in materials science and condensed-matter physics. It is well known that SiO₂ crystals, such as cristobalite, tridymite, and quartz, and the glass obtained at ambient pressure have three-dimensional network structures consisting of SiO₄ tetrahedra. Considering the fact that the density of quartz is 2.65 g/cm³ (at ambient pressure) and that of stishovite is 4.29 g/cm³, it can be assumed that fourfold-coordinated structures contain a large amount of voids. If small gas molecules such as helium and hydrogen dissolve in these voids, it is expected that the compressional behavior of these materials may be changed and/or new structures may appear. In this presentation, we will report anomalous behavior of SiO₂ glass and cristobalite, fourfold-coordinated SiO₂ having especially low densities (2.20 and 2.33 g/cm³, respectively), in helium at high pressures. All experiments were conducted using a diamond-anvil cell.

The pressure dependence of the volume of SiO₂ glass was determined by measuring the change in the size of the bulk sample in microscope images [Meade & Jeanloz, 1987]. The result up to 10 GPa shows that the volume change of SiO₂ glass in a helium medium is much smaller than in a mixture of methanol-ethanol medium, suggesting that the voids in SiO₂ glass are prevented from contracting because a large amount of helium penetrates into these voids. The solubility of helium in SiO₂ glass is estimated to be more than 1 mol per mole of SiO₂ glass at 10 GPa. X-ray diffraction measurements also show that the pressure-induced shift of the position of the first sharp diffraction peak (FSDP) in helium is significantly smaller than that in previous studies. The FSDP is associated with the presence of the intermediate-range order and considered to arise from the periodicity of ordering of the rings consisting of SiO₄ tetrahedra [Elliott, 1991; Mei et al., 2008]. Therefore this small shift of the FSDP is consistent with the small volume change. The result of Raman scattering measurements is also significantly different from those in the previous studies with argon and other mediums.

On cristobalite, x-ray diffraction measurements were conducted. At around 10 GPa, a new phase, which has diffraction peaks at larger *d* values, was observed. It may be possible that the volume increases as helium dissolves in cristobalite. Above 20 GPa, another new phase, which has very broad diffraction peaks, was observed. At this pressure range, the transformation to a stishovite-like structure was observed in argon and other mediums [Yamakata and Yagi, 1997]. This second new phase might have a sixfold-coordinated structure. We are planning to obtain more precise data in order to determine the structures of these two new phases.

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High-pressure X-ray diffraction and Raman spectroscopic studies of magnetite, ulvospinel, and chromite

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Magnetite Fe_3O_4 , ulvospinel Fe_2TiO_4 , and chromite FeCr_2O_4 have been investigated by high-pressure x-ray diffraction and Raman spectroscopy with diamond anvil cell techniques. The crystals used as starting materials were synthesized in a 1-atm furnace at 1100 °C for 48 hours with a CO/CO_2 gas flow from dried powdered oxides of Fe, Ti and Cr. A powder x-ray diffraction study of magnetite was performed up to 154 GPa with and without laser heating. The x-ray diffraction profiles showed changes at 28 GPa. With further compression up to 154 GPa, a phase change occurred above 80 GPa. Powder x-ray diffraction measurements of ulvospinel were carried out up to 60 GPa at ambient temperature. Phase transitions in ulvospinel were found near 9 GPa, 12 GPa, and 50 GPa, respectively where the crystal structure transforms from cubic to orthorhombic through a tetragonal phase. The phase observed above 50 GPa was reversibly changed to the lower-pressure phase with decompression. The x-ray diffraction profile above 50 GPa can be explained by the high pressure phase of the CaTi_2O_4 type structure (space group $Cmcm$) with lattice parameters $a = 2.65$, $b = 9.25$, $c = 9.30$ Å, and $V = 228$ Å³. Structural refinements of chromite were obtained from single-crystal x-ray diffraction measurements collected at several pressures up to 15 GPa. A phase transition in chromite was found at 12 GPa. The crystal structure of chromite transforms from cubic to tetragonal in a manner similar to the pressure induced phase transitions in ulvospinel. Fitting of the P-V data to a Birch-Murnaghan equation of state (EoS) results in $K_0 = 196(5)$ GPa, $K' = 4.0$ (fixed), and $V_0 = 589.2(1)$ Å³. The K_0 value for chromite is in fair agreement with the experimental results of spinels and theoretical predictions of chromium spinels. From the structural refinements the tetrahedral site (T) is occupied by Fe^{2+} cation with a greater compressibility than the octahedral site (M) occupied by the Cr^{4+} cation owing to the Jahn-Teller effect at Fe^{2+} . The resulting EoS parameters for the T-site and M-site are $K_0 = 141(3)$ GPa, $K' = 4.0$ (fixed), $V_0 = 4.1(1)$ Å³ and $K_0 = 256(9)$ GPa, $K' = 4.0$ (fixed), $V_0 = 10.4(1)$ Å³, respectively.

The three spinels were studied by laser Raman spectroscopy using a laser power of the 532 nm laser line of 1-2 mW on the sample. There are five Raman-active modes ($A_{1g} + E_g + 3F_{2g}$) in the $Fd-3m$ space group of the cubic spinel according. Two Raman active modes assigned to A_{1g} and F_{2g} are clearly observed around 500 and 700 cm^{-1} under ambient conditions. The A_{1g} mode in the cubic structure transforms to the A_g mode in the tetragonal and orthorhombic structures. In contrast, the F_{2g} mode in the cubic structure splits into $B_{1g} + E_g$ modes in the tetragonal structure, and then into $B_{1g} + B_{2g} + B_{3g}$ modes in the orthorhombic structure. With increasing pressure, in the Raman spectra of chromite, the A_{1g} and F_{2g} modes in chromite do not change up to 20 GPa except for a continuous shift to higher frequencies. The Raman spectra of ulvospinel and magnetite start to broaden gradually with increasing pressure. It seems reasonable to attribute the observed broadening to the peak splitting caused by the structural phase transitions. The most striking characteristic of the Raman spectrum of ulvospinel is that compression leads to the extinction of the Raman active mode derived from F_{2g} symmetry. The F_{2g} mode in ulvospinel disappears completely at 20 GPa, but its A_{1g} mode can be observed continuously up to 57 GPa. The Raman spectra of both A_{1g} and F_{2g} modes in magnetite disappear at 30 GPa.

Keywords: magnetite, ulvospinel, chromite, phase transition, high-pressure & high-temperature, diamond anvil cell

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Measurement of elastic constants of single-crystal chromian spinel by high frequency resonant ultrasound spectroscopy

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Chromian spinel found in mantle xenoliths contains fluid inclusions whose residual pressure (fluid density) can provide us the origin depth of the xenoliths. A host chromian spinel grain should have deformed in response to a change in ambient temperature and pressure during its transport, and changed the fluid density. Elastic and plastic properties of chromian spinel are essential for evaluation of the deformation and precise estimation of the origin depth. Although elastic constants of spinel (MgAl_2O_4) and chromite (FeCr_2O_4) have been already reported, few studies have been done on chromian spinel. We thus have studied elastic constants of a single-crystal chromian spinel using a resonance method. Chromian spinel crystals were collected from mantle xenoliths from Sveyagin, Russia (Yamamoto et al., 2009, Island Arc). One crystal was selected in terms of the uniformity of crystallographic orientation examined by SEM-EBSD. Each face was polished flat (< 1 micrometer) in an orientation parallel or perpendicular to $\{100\}$ by the X-ray precession method. The selected crystal was shaped into a rectangular parallelepiped ($0.412 \times 0.407 \times 0.497$ mm³). The Mg/(Mg+Fe(II)) ratio is 0.75, and the Cr/(Cr+Al) ratio 0.08. The density is 4.3×10^3 kg/m³. The resonant frequency was measured from 3 to 11 MHz using a system specially designed for such a small sample (Yoneda et al., 2007, JJAP). Preliminary analysis has shown that C_{11} , C_{12} , and C_{44} are 263.26, 137.96, and 123.71 (GPa), respectively. Comparison with elastic constants of spinel and chromite will be discussed in terms of the compositional difference.

Keywords: elastic constants, resonance method, chromian spinel

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High-pressure stability field and thermodynamic properties of anhydrous phase B (Mg₁₄Si₅O₂₄)

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Anhydrous phase B (Anh-B: Mg₁₄Si₅O₂₄) is a high-pressure magnesium silicate possibly stable in magnesium-rich regions in the upper mantle (Ganguly and Frost, 2006). They examined the equilibrium boundary of the reaction, 5forsterite (Fo) + 4periclase(Per) = Anh-B, at 9.0-12.5 GPa and 1173-1873 K. Ottonello et al. (2010) computed thermo-chemical and -physical properties of Anh-B by first principles calculation. However, those of Anh-B have been poorly determined by experiments. In this study, we examined stability field of Anh-B by high pressure experiments, and performed drop-solution calorimetry, heat capacity measurement and high temperature X-ray diffraction measurement on Anh-B. Obtained data were applied to thermodynamic calculation of the phase equilibrium boundaries of the reactions Fo+Per =Anh-B and Anh-B=Wads+Per which were separately determined by high pressure experiments.

High-pressure high-temperature phase relations experiments on Anh-B were performed using a Kawai-type 6-8 multianvil high pressure apparatus. The starting material was a mixture of Per and Fo with 4:5 in molar ratio. The experimental conditions were in a pressure range of 12-23GPa and in a temperature range of 1673 -2073 K. Samples recovered after quenching were analyzed with powder X-ray diffraction method and SEM-EDS. Drop solution calorimetry of Anh-B was performed using a Calvet-type calorimeter with Ar bubbling technique and lead borate solvent. Heat capacity was measured by DSC in a range of 300-770 K. High temperature X-ray diffraction measurements were made in a range of 303-773K every 50 K step. Raman spectroscopy of Anh-B was done using a micro-Raman spectrometer (JASCO NRS-3100).

The results of the high-pressure and high-temperature experiments indicated that the equilibrium boundaries of 5 Fo + 4 Per = Anh-B and of Anh-B = 5 Wadsleyite(Wads)+ 4 Per are located at 13 GPa and at 19 GPa, respectively, at 1873 K. The drop-solution enthalpy of Anh-B was determined to be 844.3 +/- 29.9 kJ/mol. The drop-solution enthalpy of Fo was 168.2 +/- 0.9 kJ/mol, and that of Per was 33.7 +/- 1.0 kJ/mol (H. Kojitani, unpublished data), and that of Wads was 142.2 +/- 2.7 kJ/mol (Akaogi et.al., 2007). The enthalpy changes for the reactions 5 Fo+ 4 Per = Anh-B and Anh-B = 5 Wads + 4 Per were obtained as 130.9 +/- 30.5 kJ/mol and -1.46 +/- 33.08 kJ/mol, respectively. We obtained a heat capacity equation of $C_{P(Anh-B)} = 1.531 \times 10^3 - 1.315 \times 10^4 T^{-0.5} - 7.925 \times 10^8 T^{-3}$ and thermal expansivity of $2.07 \times 10^{-5} + 1.75 \times 10^{-8} T$. Raman spectrum of Anh-B were consistent with lattice vibrational mode frequencies calculated by Ottonello et al. (2010). By applying the C_P , thermal expansivity and Raman data to Kieffer model calculation, entropy of Anh-B was estimated to be 563.37 J/(mol K). Therefore, entropy changes for the reactions 5 Fo+ 4 Per = Anh-B and Anh-B = 5 Wads + 4 Per were obtained as -14.43 kJ/(mol K), and -24.07 kJ/(mol K), respectively. The phase boundaries by thermodynamic calculations were consistent with those obtained by the high pressure experiments. It is concluded that stability field of Anh-B is placed at the range of 13-19 GPa and 1873 K.

Keywords: anhydrous phase B, high pressure high temperature experiment, thermodynamic calculation

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Thermal properties of hydrous minerals under pressure

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We measured thermal diffusivity, thermal conductivity and heat capacity of hydrous minerals, and found their particular behavior in the thermal property. Among hydrous minerals, serpentine ($\text{Mg}_6\text{Si}_4\text{O}_{10}(\text{OH})_8$) and talc ($\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$) are thought to be important, because their water exchange among major components, olivine or peridotite. Measurements were conducted using a pulse heating method in the Kawai apparatus. Antigorite has much lower thermal diffusivity and thermal conductivity (less than half) of the major dry mantle minerals such as olivine. The pressure derivative of thermal diffusivity or thermal conductivity is small compared with olivine. On the other hand, talc has relatively higher thermal conductivity compared with antigorite. Antigorite and talc are quite similar hydrous sheet minerals. Although talc, one of the softest mineral, has lower acoustic velocity than antigorite, thermal conductivity of talc is two to three times greater than that of antigorite. This apparent contradiction may be attributed to alternating wavy structure in antigorite with a few nanometer wave length. In terms of a simple calculation, phonons excited at 300 K tend to have wavelengths shorter than 1 nm. Therefore, phonons may be trapped in a wavy segment of the antigorite crystal structure. This can cause lower thermal conductivity for antigorite. Heat capacity is obtained from the simultaneous measurement of thermal diffusivity and thermal conductivity. The heat capacity of antigorite at ambient temperature is ca. $1.0 \times 10^3 \text{ Jkg}^{-1}\text{K}^{-1}$ and nearly independent of pressure increase, that is, the pressure derivative of heat capacity of antigorite is nearly zero. This value contrasts with the negative value for olivine. On the other hand, preliminary results for talc showed the positive pressure derivative. From the pressure derivatives of heat capacity, we can evaluate characteristics of the thermal expansivity. We can expect nearly constant thermal expansivity for antigorite for some assumptions. In other words, the volume of antigorite increases almost linearly with temperature. From lattice dynamic theory, we can define the mode Grueneisen parameter for the normal mode of lattice vibration. Note that the positive parameter contributes to positive thermal expansion, and vice versa. Thus, the majority of the parameter for olivine is expected to be positive. On the other hand, some of the parameter for antigorite and talc are expected to be negative. It is quite probable that the hydrous mode, or normal mode related with hydrogen and hydroxyl groups, may have a negative mode Grueneisen parameter. Thus, hydrous minerals reveal particular thermal properties and its behavior is different from major mantle minerals such as olivine.

Keywords: thermal diffusivity, thermal conductivity, heat capacity, high-pressure, hydrous mineral

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Equation of state of antigorite at high pressure and temperature

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Antigorite plays key roles in subduction zone processes including transport of water and seismogenesis. The equation of state (EoS) of antigorite is critical for understanding of its stability field and for interpretation of seismological observations. Although a few compression tests have been conducted at room temperature, EoS is still poorly understood at high temperatures. We have investigated EoS of antigorite by in-situ synchrotron X-ray powder diffraction. Measurements were conducted at a beamline NE5C of Photon Factory-Advanced Ring (KEK, Tsukuba).

The sample was a natural antigorite collected from Inner Mongolia, China. Most of the Selected Area Electron Diffraction (SAED) patterns show the presence of the polysomes $m=15$, where m is the number of tetrahedral along a wave (Mellini et al., 1987). Some have $m=16$ and 17. The sample was finely ground and mixed with NaCl, and pressurized in a multi-anvil type high-pressure apparatus (MAX80). Measurements were made at pressures of 0~6 GPa and temperatures of 25~500 C. The pressure was estimated from the compression of NaCl. Diffraction peaks of antigorite were indexed with the aid of indices reported by Uehara and Shirozu (1985) and Capitani and Mellini (2004). Lattice parameters A , a , b , c and β were estimated by the least square method.

The compression in the c -axis dominates the isothermal compression in bulk. The compressibility in the c -axis is larger than those in the a - and b -axes by a factor of ~ 3 . This is consistent with Hilairet et al. (2006). The linear compressibility in the c -axis significantly increases with increasing temperature ($(6\pm 3)\times 10^{-11} \text{ Pa}^{-1}\text{K}^{-1}$), while those in a - and b -axes are almost independent of temperature. The isothermal bulk modulus is estimated to be 60 GPa at room temperature, which is close to the previously reported value.

The expansivity in the c -axis is the largest and largely decreases with increasing pressure, while that in the b -axis the smallest and almost independent of pressure. No significant difference can be seen between axes at the pressure of 5 GPa. The volumetric thermal expansivity is calculated to be $(3.8\pm 0.6)\times 10^{-5} \text{ K}^{-1}$ at $P=0$ GPa, which is consistent with the previous estimation (Holland and Powell, 1998). Our result clearly shows that the expansivity decreases with increasing pressure.

Keywords: equation of state, antigorite, serpentine, compressibility, thermal expansivity

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Microstructure in mullitization from sillimanite

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Transmission electron microscope (TEM) experiments were carried out of sillimanite which was heated in various conditions. The sample taken as starting material was sillimanite crystal in Rudvagssetta, Lutzow-Holm, Antarctica. This starting crystal has no microstructure and l -odd reflections in electron diffractio pattern.

Sample which was heated at 1470C for 1150 hours had two domains by TEM observation; in one domain the reflections with l -odd which is characteristic of sillimanite were obtained in the diffraction pattern, and in another those were not obtained. This In the domain with l -odd reflections, anti-phase boundary (APB) like structure was obaserved. And furthermore, both domains had SiO₂-rich glass inclusions. These relusts indicate that sillimanite decompose to mullite and SiO₂-rich melt/SiO₂-rich amorphous at high temperature. And sillimanite change to disordered sillimanite with mullite components at high temperature and then APB-like structure occures at cooling time.

Keywords: sillimanite, mullite, microstructure

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Experimental study on the phase transition of graphite to hexagonal diamond

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Hexagonal diamond (lonsdaleite) is a metastable polymorph of carbon and occurs as microscopic crystals associated with cubic diamond in some meteorites such as the Canyon Diablo meteorite. Recent theoretical studies reported a possibility that hexagonal diamond has indentation strength and a bulk modulus comparable to or even greater than those of cubic diamond. However, such physical properties of hexagonal diamond have not experimentally investigated due to a difficulty in synthesizing a single-phase bulk sample. Here, we investigated the P-T conditions required to obtain single-phase hexagonal diamond from graphite.

We performed a series of high-pressure and -temperature experiments using a laser-heated diamond anvil cell (DAC) at pressures of 25 and 50 GPa and temperatures ranging from 1400-3300 K. Highly oriented graphite (starting material) was compressed in a DAC without using a pressure transmitting medium and rapidly heated to a target temperature for ~1min using fiber laser. The sample became transparent upon laser heating above 2300 K at 25 GPa and above 1400 K at 50 GPa, suggesting that the phase transition of graphite to diamond phase(s) occurred under those P-T conditions. Interestingly, the transparent area became apparently smaller or almost disappeared (especially in the case of the experiments at 50 GPa) after decompression to room pressure.

The recovered samples were first examined by Raman spectroscopy for phase identification and then by transmission electron microscopy (TEM) for microtextural observations and electron diffraction analysis. The Raman spectra collected from the transparent area in the samples showed a broad peak at 1350-1450 cm^{-1} , but no cubic diamond peak (at 1332 cm^{-1}). TEM observation revealed that the transparent area in each recovered sample consists of hexagonal diamond with a layered structure similar to that of the graphite starting sample. The electron diffraction pattern collected from the sample is complex and can be interpreted as a superposition of three types of reciprocal patterns in which [100], [002] and [-212] of hexagonal diamond are all arranged in a coaxial relation with graphite [002]. This suggests that the martensitic phase transition from graphite to hexagonal diamond proceeds as a result of $1/2a$ or $1/3(1/2)a$ layer shifts of graphene layers along graphite [100].

Keywords: Hexagonal diamond, Highly oriented graphite, Laser-heated diamond anvil cell

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Temperature dependence of XANES spectra for BaTiO₃, SrTiO₃ and TiO₂ with structural phase transitions

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BaTiO₃ is perovskite structure and has good physical properties as industrial material. The main constituent (MgSiO₃CaSiO₃) of the lower mantle is the same structure. Therefore, BaTiO₃ has been intensively studied for a long time. But BaTiO₃ had not been so researched by using X-ray absorption near edge structure (XANES) spectra. XANES spectra of BaTiO₃, SrTiO₃ perovskite and TiO₂ rutile and anatase were measured at various temperatures. The composition, structure and temperature dependence of XANES spectra was investigated especially on the phase transition. The appropriate amount of fine powder sample and boron nitride powder was mixed and pressed into pellet of <0.2 mm thickness and 10.0 mm in diameter. All samples had edge-jumps with 0.7. The measurements of Ti k-edge XANES spectra were carried out in transmission mode at beam line BL-9A of the Photon Factory in KEK, Tsukuba. X-ray absorption measurements in the temperature range from 20K to 800K were made under a helium atmosphere. The XANES spectra for TiO₂ were largely different between anatase and rutile structure, although these two compounds have the same composition. Ti K-edge XANES spectra changed largely with different compositions, while the temperature dependence of XANES spectra is small in each compound even if undergoing structural phase transition. Perovskite-type ATiO₃ compounds reveal several phase transitions. SrTiO₃ and BaTiO₃ perovskite undergo several structural phase transitions in the temperature ranges in this study, SrTiO₃; rhombohedral-tetragonal-cubic, BaTiO₃; trigonal-orthorhombic-tetragonal-cubic, the distinct changing of pre-edge XANES spectra was observed near transition points. Ti atoms are located in TiO₆ octahedral site for the all samples, but Ti k-edge XANES spectra changed largely with different compositions

Keywords: XANES, BaTiO₃, Perovskite, Phase transition