

Relationship between Skempton's coefficient and diagenesis of the Quaternary Kazusa Group siltstones

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Skempton's coefficient B is one of fundamental properties of sediments and rocks. B is defined as the change in pore pressure per unit change in total stress applied under undrained conditions. To reveal the evolution of B of sediments and sedimentary rocks during diagenesis is critical for some processes relating to geophysics and geology, such as mechanisms of developing abnormal pore pressure in sedimentary basins (Tanikawa et al., 2008). However, how B depends on diagenesis is still not clear. To understand the dependency, we evaluated B based on porosity measurements under effective stress for siltstones collected from various formations in the Quaternary Kazusa Group. We also tried to measure B directly, and compared the results with B values obtained from measurements of porosity.

We used siltstones of Umegase, Otadai, Kiwada, Ohara and Katsuura Formations of the Kazusa Group as the samples for the experiments. The specimens from the samples were 30 mm in diameter and 40 mm in length. The laboratory experiments were performed using an intra-vessel deformation fluid-flow apparatus at Toho University, at room temperature and under confining pressures from 2 MPa to 35 MPa. Distilled water was used for pore fluid. From the results of the porosity measurements under effective pressure, we estimated the compressibilities of the rock on the assumption that volume change of the rock at effective stress change equals to the pore volume change, and calculated B from the results. In the direct measurements of B , we measured pore pressure changes when confining pressure were applied under undrained conditions.

The results of B estimations from porosity measurements indicated that B tends to decrease with increasing burial depth. But, B of Ohara siltstones was somewhat higher than other samples despite Ohara Formation is relatively lower in the Kazusa Group. This is probably because siltstones at Ohara Formation were not consolidated enough as compared with those at other formations due to some reasons such as developing abnormal pore pressure. Results indicated that the dependency of B on effective pressure is not simple. B was not simply decreased with effective pressure increases, but B was increased at some range of effective pressure, which mostly reflected that the compressibility was increased at the transition from overconsolidation to normal consolidation state. B depends on both compressibility and porosity, and in the case of the Kazusa Group siltstones, the behavior of compressibility has greater effect on B . Thus, B is decreased as a grade of diagenesis increases because compressibility is decreased. The values of B measured directly tended to be higher than B estimated from measurements of porosity. This is probably because a period between step changes of effective stress was not enough for the rocks to reach steady state.

Keywords: Skempton's coefficient, diagenesis, Kazusa Group, porosity, compressibility, laboratory rock experiment

Effects of pore pressure changes on frictional behaviors of talc

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Since the discovery of low frequency earthquake, several classes of physical mechanisms have been proposed to explain this events. Several models and geophysical observations have suggested that the overpressurized fluid along the subducting plate interface have an important role in triggering such events. In addition, the existence of hydrous minerals along the plate boundary at fore-arc wedge may also be important for the seismogenic properties of the slab-mantle interface. Especially, serpentine minerals are generally expected to be the main hydrous mineral present in the fore-arc minerals, and the existence of serpentine in the wedge mantle has suggested by seismological studies. However, talc may also be important in fault mechanism at the plate boundary in the fore-arc wedge mantle, because slab-derived fluids are likely to lead replacement of serpentine by talc at the slab-mantle interface in the fore-arc wedge mantle, and talc is one of the weakest minerals that constitute natural fault zones. However the quantitative influences of pore pressure on the frictional properties of talc are not well constrained. We conducted friction experiments using pre-cut samples of talc with controlling pore pressure P_p and confining pressure P_c adopting several kinds of stress paths during an experiment.

Cylindrical samples of talc (Gvangjsih, China), 20 mm in diameter, were cut at an angle of 30° to the sample axis. The sliding surfaces were ground with carborundum (#400). A small hole (3 mm in diameter) through the center of each piece ensured adequate communication of the water between the pre-cut surfaces with the rest of the pore pressure system. The specimen was loaded by a triaxial apparatus and sheared under an axial displacement rate of $1 \mu\text{m/s}$. We used water as a pore fluid. All measurements were performed at room temperature. Experiments were conducted under several paths of P_c (up to 110 MPa) and P_p (up to 100 MPa). During steady axial loading, either P_c or P_p was changed stepwise.

The stepwise changes of effective normal stress $\sigma (= \sigma_t - P_p$, where σ_t is total normal stress) resulted in a linear elastic response of shear stress followed by a transient evolution of friction. In the case that σ was decreased, friction coefficient μ was temporally increased and then decreased back to steady state, and the normalized transient change of μ to the logarithm of normalized amplitude of σ change ranged from 0.2 to 0.28, which is comparable to that for quartz and Westerly granite reported by previous studies (Linker and Dieterich, *J. Geophys. Res.*, 1992; Hong and Marone, *Geochem. Geophys. Geosyst.*, 2005). While in the case that σ was increased (μ was temporally decreased then increased), the values were smaller (less than 0.12, and negative in some cases), which means that a transient change of μ was less dependent on a change of σ than that for quartz and granite, which may reflect ductile deformation of contacts on fault surfaces during the evolutionary transition.

This frictional property might cause slow slip phenomena. After an initiation of a fault slip, possibly triggered by an increase of pore pressure, partially undrained conditions on the slip surface may cause dilatancy hardening, and therefore σ may be increased during the slip. The results of this study suggest that, in the case that a fault plane is covered by talc, the temporal decrease of μ following an increase of σ might be smaller than the case that fault planes are covered by other ordinary minerals. Consequently, a frictional resistance might act more effectively than faults covered by other ordinary minerals and an acceleration of fault slip rate might be mitigated, and therefore a slip rate might be smaller than regular earthquakes.

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Keywords: talc, friction experiment, pore pressure, low frequency earthquake

Sintering experiments on fine-grained polycrystalline orthoclase

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K-feldspar is one of the major mineral components of granitic rocks and a wide variety of metamorphic rocks. Its deformational behavior is important for establishing the overall rheology of continental crust. The creep strength is influenced by various factors such as mineral species, grain size and pores. Therefore control of these factors in polycrystalline minerals is essential for rheological experiments. In order to make dense polycrystalline orthoclase, we have carried out sintering experiments.

We prepared submicron mineral powders from a single crystal of orthoclase ($K_{0.83}Na_{0.17}Al_{1.04}Si_{2.96}O_8$). As a result of X-ray fluorescence (XRF) analysis, ZrO_2 (<5.65wt.%) which is considered to be contamination from a mill was detected. We formed cylindrical compacts from fine-grained mineral powders by uniaxial dry pressing at room temperature and pressure of 20MPa. Sintering was carried out using a tube furnace at temperature of 970 °C for 4 hours, achieving a vacuum condition of $\sim 4.1 \times 10^4$ Pa. We also compared the sintered body with a sample sintered at atmospheric pressure using muffle furnace at the same temperature and the time. Sintered bodies were observed using scanning electron microscope (SEM), and analyzed by XRF and X-ray diffraction (XRD).

As the result of vacuum and atmospheric pressure sintering, we obtained sintered bodies with volume reduction of 52.2% and 44.5%, and porosity of 0.15 and 0.17, respectively. SEM images showed that densification process was advanced by both vacuum and atmospheric pressure sintering. We confirmed that crystal structures (Al/Si order-disorder) were not changed from compacts by XRD patterns.

In this study, we found that dense submicron polycrystalline orthoclase can be made from fine-grained powders by either of vacuum and atmospheric pressure sintering, and confirmed that orthoclase does not cause order-disorder phase transition by a sintering for 4 hours.

Keywords: submicron, orthoclase, sintering

Doping effect on high-temperature creep of polycrystalline anorthite

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Rheological properties of lower crust are considered to play important roles on the cause of inland earthquakes. Previous studies on creep properties of polycrystalline anorthite indicate that the polycrystalline anorthite will deform under diffusion creep at temperature condition of 400 to 1000C and grain size of <100 um where such conditions are identified in mylonites which are of lower crust origin. Therefore, it is important to know a precise strength of polycrystalline anorthite during diffusion creep.

Previous studies have shown the influence of grain size, temperature, stress, and water content on the strength of polycrystalline anorthite. It is well known that a small amount of impurities segregated at grain boundaries has a significant effect on the strength of polycrystalline oxides. We have shown that our pure anorthite aggregate, which was synthesized using the technique that could minimize the contamination of impurities, had two orders of magnitude larger strength than anorthite aggregates used in previous studies. In this study, we examine the effect of doping a small amount of MgO on high-temperature creep of anorthite aggregates.

MgO-doped anorthite aggregates were fabricated from nano-sized powders of CaCO₃, Al₂O₃, SiO₂, and Mg(OH)₂, all of which have <50 nm in diameter, and vacuum sintering of the powders. We controlled the amount of Mg(OH)₂ powders to obtain anorthite doped with 1wt% of MgO. Constant load tests were performed at temperatures ranging from 1150 to 1380C, stresses from 10 to 120 MPa, and confining pressure of 0.1 MPa. We measured Arithmetic mean grain size of specimens by microstructural observations using scanning electron microscopy (SEM) before and after creep tests.

Grain sizes of the specimens were 1~2um before and after the creep test. Log stress versus log strain rate showed a linear relationship where its slope gave a stress exponent, n of 1, indicating that samples were deformed under diffusion creep. MgO-doped anorthite aggregates exhibited more than one order of magnitude weaker than pure anorthite. We obtained activation energy, Q of 702 kJ/mol which was higher than that of our pure anorthite. The difference in strength between pure and MgO-doped anorthite was attributed to the presence of a small amount of MgO which was probably segregated at grain boundaries.

Keywords: polycrystalline anorthite, diffusion creep, effect of doping

Synthesis of textural polycrystalline forsterite using colloidal processing in a strong magnetic field.

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It is well known that the crystallographic preferred orientation (CPO) of minerals is commonly produced in the Earth's interior. Thus, it is important to understand the physical properties of the mineral aggregates that exhibit CPO. However, silicate minerals are often feeble magnetic and have small anisotropic susceptibilities so that it is difficult to apply a magnetic field effectively to rotate the mineral particles. Tendency of finer particles to spontaneously agglomerate due to strong attractive interactions (van der Waals forces) add further difficulty. We used a technique of slip casting in a high magnetic field (12T) to align certain crystallographic axis of mineral particles. For the particles to rotate easily in the solvent under a strong magnetic field, we improve the method of deflocculating. To control the surface potential of the particles, we applied various types of polymer modification. Vacuum sintering of the powders that were composed of the aligned particles was expected to produce a polycrystalline material aggregate that exhibits CPO. The resultant materials were characterized by X-ray powder diffraction (XRD), secondary electron microscope (SEM) and Electron Backscatter Diffraction (EBSD).

The specimen exposed to a strong magnetic field exhibits preferential A-axis alignment to the magnetic direction. Those synthetic specimens allow us to examine the effect of CPO on the physical properties of the earth's materials in future room experiments.

Keywords: forsterite, polycrystalline, magnetic field, orientation, CPO

Grain growth experiment on pyrolite material under lower mantle conditions

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Grain size is a key parameter for understanding viscosity of Earth's mantle. Grain growth rate is one of important factors controlling the grain size. Especially, it is indispensable to examine grain growth kinetics in multiple phases because the grain growth rate of major phase drastically changes with the proportion of secondary phases (e.g., Hiraga et al., 2010). In the lower mantle, Mg-perovskite is major phase, and ferro-periclase, Ca-perovskite, and majoritic garnet are present as secondary phases (e.g., Irifune, 1994; Nishiyama and Yagi, 2003). The previous grain growth experiment (Yamazaki et al., 1996) in the two-phase system of MgSiO₃ perovskite and MgO periclase using Mg₂SiO₄ forsterite as a starting material suggests that the grain growth rate is too slow to explain the lower mantle viscosity constrained by geophysical observations. This inconsistency may arise from effects of the eutectoid transformation prior to the grain growth process (e.g., Solomatov et al., 2002). It is also necessary to examine effects of the chemical composition that affects the proportion of secondary phases and diffusivity. Here, we report preliminary results of the grain growth experiment on pyrolite material under lower mantle conditions.

High-pressure and temperature experiments were conducted using a Kawai-type multi-anvil apparatus (QDES) installed at Kyushu University. Starting material is a powder with pyrolite composition that was used in the previous phase equilibrium study (e.g., Irifune, 1994). We conducted annealing experiments at 25-28 GPa and 1600-1800 °C for 6-600 min. Chemical compositions, microstructures and grain sizes of recovered samples were examined using a FE-SEM with an energy-dispersive analytical system.

Four phases of Mg-perovskite, Ca-perovskite, ferro-periclase and majoritic garnet were present in recovered samples annealed at 25 GPa and 1600-1800 °C. The normalized grain size distribution in the recovered samples showed Gaussian-like shape and the largest grain size is smaller than three times of the mean grain size, suggesting that normal grain growth occurred. The grain growth rate is faster than that of the previous study (Yamazaki et al., 1996). Preliminary analysis of the kinetic data of Mg-perovskite obtained showed the smaller grain growth exponent of 4.3 than that reported in the previous study. On the other hand, three phases of Mg-perovskite, Ca-perovskite and ferro-periclase were present at higher pressure of 28 GPa and 1800 °C, in which the volume fraction of Mg-perovskite increased compared to the four-phases experiment. While the microstructure and the grain size distribution in the three-phase assemblage was similar to those of the four-phase assemblage, the grain size was larger probably due to the smaller proportion of the secondary phases. Our preliminary results provide some insights into the grain-size evolution in the lower mantle and suggest that further quantitative grain growth data with possible lower mantle conditions are needed.

Keywords: lower mantle, multi-anvil, pyrolite, grain growth

Grain boundary diffusion of noble metal elements in mantle composites

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So far, it is not clear whether Earth's core and mantle have been chemically isolated through geological time. It has been believed that highly incompatible elements such as siderophile elements in the mantle minerals have not been moved from the core to the mantles so that the elemental abundance of highly siderophile elements (HSEs) in the core and mantle were determined when both were separated. Although HSEs are refractory, amounts of HSEs are very little in the mantle (Wood, 2006) so that these elements are expected to be highly concentrated in the core relative to the silicate mantle. However, a recent study has shown that incompatible elements can be concentrated and quickly diffuse at grain boundaries (Hiraga et al., 2004). If HSEs can diffuse from the core to the mantle, the concentration of HSEs in the mantle can change through Earth's history. Therefore, HSEs can be a good tracer to detect the chemical interaction of the core and the mantle.

We conducted grain growth experiments on Au particles in forsterite (Fo) aggregates at 1 atmosphere pressure and temperature of 1360 °C. We prepared several sintered bodies which were made by dispersing 10vol% Au particles in Fo aggregates and then annealed for several hours. We observed these bodies using a scanning electronic microscope. In the result, Au particles changed their shape from spherical to polygonal. This is due to a balance of interfacial tensions between Au and Fo phases. Further, average grain size of Au particles was found to increase with time. Based on these observations, we conclude that Ostwald ripening of grains, by which Au atoms move from small particles to larger ones to minimize entire interfacial energies of the system, occurred in our experiment. Grain boundaries as diffusion paths should be responsible for Au diffusion. In this case, grain growth of Au particles will follow the relationship of $d^4 - d_0^4 = kt$, where d is the average grain size of Au particles after annealing, d_0 is the initial average grain size, k is the grain growth coefficient, and t is annealing time. Using the average grain size of each body, we calculated k . In addition, we estimated the interfacial energy of the system from the shape of Au particles and calculated the product of concentration of Au particles at grain boundaries, c , and diffusivity of Au atoms at grain boundaries, D .

Keywords: grain boundary diffusion, grain growth, core-mantle interaction

The effect of partial melting on the mantle viscosity and electrical

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In this study, in order to know the change in mantle viscosity and electrical conductivity during partial melting, which corresponds to decompression melting of ascending mantle beneath mid-ocean ridge, mantle analogue sample was synthesized and used to measure its viscosity and electrical conductivity under atmospheric pressure and high temperature conditions. The sample has lherzolite composition of olivine (50%), orthopyroxene(40%) and clinopyroxene(10%) with addition of 0.5 vol% spinel. Constant force was applied to the sample under increasing the temperature where its range includes sample solidus. Sample viscosity and the electrical conductivity by the impedance measurement were calculated for every temperature. We particularly examined how viscosity and conductivity change when the sample transforms from melt-free to melt-bearing system. Temperature ranged from 1100 to 1390 °C, which resulted in the change of melt fraction (ϕ) from 0 to 0.09, where the melt composition becomes enriched in clinopyroxene component as the temperature increases.

We observed a continuous and gradual reduction of sample viscosity with increasing temperature. The effect of the increasing melt fraction on the sample viscosity should have been added to the viscosity change simply due to thermally activation process. There is a linear relationship between measured $1/T$ and $\log(1/\eta)$, which goes well with the previous proposed empirical expression of flow law that, which is an function of melt fraction.

Analyzing the observed viscosity change with temperature with this law, the apparent activation energy of 970 kJ/mol is obtained at a temperature range of 1220 °C to 1340 °C and the effect of increasing melt fraction on sample viscosity roughly corresponds to the activation energy of \sim +35 kJ/mol. The activation energy on the melt free system is estimated to be 935 kJ/mol. This value is close to the activation energy of the dislocation creep of orthopyroxene and clinopyroxene indicating that the sample viscosity was essentially controlled by deformation of pyroxenes.

Electrical conductivity did not change dramatically when the experimental temperature reached and exceeded the sample solidus. Grain size dependency on the conductivity was observed at all temperature conditions indicating that the conductivity is simply determined by grain boundary conductivity even at higher melt fraction condition, probably due to fine grain size of the samples. Compared with previously reported grain boundary conductivity in the melt-free forsterite system, grain boundaries in our sample have 3 to 4 times higher conductivity indicating that the pyroxene grain boundaries have a large effect on the bulk sample conductivity in our experiment.

In this study, it was demonstrated that previously proposed empirical flow law as a function of melt fraction can approximate the viscosity change of the mantle during its transition from melt-free to melt-bearing. Taking into account of the incremental rate of melt fraction with temperature and the connectivity of intergranular melt in the mantle highly depend on the volume fraction of the pyroxenes and spinel phases, mineral mode in the mantle should have substantial effects on the mantle rheology and electrical conductivity during mantle melting.

Keywords: rheology, lherzolite, melt, viscosity, electrical conductivity

Stress calibration of Griggs-type deformation apparatus with solid salt assemblies

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Mechanical properties of rocks and minerals can be quantitatively studied by deformation experiments under high-temperature and high-pressure conditions relevant to the Earth's interior. There are several types of deformation apparatus using different confining media such as gases, liquids or weak solids (e.g., Tullis and Tullis, 1986). Liquid medium apparatus have the disadvantage that they cannot be used for temperatures above 500 °C because of prevention from alteration of oil at high temperature. Solid medium apparatus can provide us high pressure safely and stably for a long time. However, stress accuracy is not high due to frictional forces between pressure medium and load piston or samples (e.g., Tullis and Tullis, 1986). Gas apparatus has most accurate stress measurement because of the usage of internal load cell; thus measured stresses do not include frictional forces. However, experiments are restricted to confining pressures less than 200 MPa in Japan due to safety issues on the usage of high-pressure gas. Therefore, solid medium apparatus is necessary for generating high temperature and high pressure required to investigate rheological behaviors of rocks and minerals in lower crust or uppermost mantle.

Recently, comparisons of mechanical results obtained for metals at the same confining pressure, temperatures and strain rates deformed in a Griggs apparatus with solid salt assemblies (SSA) and a gas apparatus provide calibration for Griggs apparatus with SSA (Holyoke and Kronenberg, 2010). This calibration law allowed differential stresses to be measured accurately to within ± 30 MPa. However, we have not been able to reproduce elastic, yielding and post-yield behaviors because the calibration law was obtained by the comparison of strengths at 5% strain of mechanical results. Calibration law for measured stresses using Griggs apparatus in all deformation behaviors are required for revealing detailed rheological behavior of rocks and minerals.

In this study, we performed axial compression experiments on high-purity nickels to measure differential stresses using a Griggs apparatus with SSA at Tohoku University. Samples were given by Drs. Holyoke and Kronenberg. Experimental conditions were confining pressures of 300 and 1200 MPa, temperatures of 600, 700, and 800 °C and strain rates of 2×10^{-4} , 2×10^{-5} and 2×10^{-6} /s. Measured differential stresses agreed with results of the former study within ± 30 MPa under the identical confining pressure of 300 MPa. However, differential stresses were larger with confining pressure. We analyzed obtained mechanical data of nickels based on the high temperature viscoelastic constitutive law developed by Shimamoto (1987). We made the master curve which normalized temperature and strain conditions. In the same way, we also made the master curve from mechanical data of nickels using a gas apparatus (mechanical data are from Holyoke and Kronenberg, 2010). Master curves were made of mechanical data of the identical material between Griggs apparatus and gas apparatus under normalized temperature and strain conditions. Therefore, difference in master curves is thought to be derived from apparatus and assembly. We derived calibration law for Griggs apparatus from difference in master curves. Applying this calibration law to differential stresses of nickels obtained using Griggs apparatus with SSA, it became possible to reproduce gas apparatus's differential stresses not only steady state but also elastic, yield and post-yield behaviors within the systematic error of ± 30 MPa. However, the error was ± 70 MPa when the calibration law was applied to the mechanical data of carbonate rock. Moreover, unlike gas apparatus, measured differential stresses using Griggs apparatus tend to become larger with confining pressures. Therefore, it is necessary to investigate the effect of confining pressures on measured stresses in the calibration law.

Keywords: rheology, deformation experiment, calibration of Griggs-type apparatus

Preliminary experiments on in-situ stress-strain measurements of Ca-Pv and Mg-Pv up to 23 GPa

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In order to discuss mantle dynamics in the Earth's interior, knowledge of viscosity of the Earth's lower mantle, which is the highest of the whole mantle, is important. Viscosity models of the Earth's lower mantle were reported by geophysical observations. However, observation values of viscosity have large variety (2~3 order magnitude). Although determination of viscosity of lower mantle minerals by high pressure experiments is needed to understand mantle dynamics, stress-strain relationship for MgSiO₃-perovskite (Mg-Pv) and CaSiO₃-perovskite (Ca-Pv), which are principal minerals of the Earth's lower mantle, are not reported due to difficulty of high pressure deformation experiments. In this study, we tried in-situ stress-strain measurements of Ca-Pv and Mg-Pv up to 23.0 GPa.

In-situ uniaxial deformation experiments were conducted using a deformation DIA apparatus (SPEED-Mk.II) as Kawai-type apparatus at SPring-8 BL04B1. Experimental conditions of Ca-Pv and Mg-Pv are 13.8 GPa, 1473 K and 23.0 GPa, 1273 K, respectively. cBN anvils, which was transparent material against X-ray, was used along X-ray path. Two-dimensional X-ray diffraction patterns were taken for 120-180 s using CCD detector. To calculate the stress magnitude from the X-ray diffraction data, we used a model of stress-lattice strain relationship (Singh et al. 1998),

$$d_{hkl}(\psi) = d_{0hkl} [1 + (1 - 3\cos^2\psi) \sigma / 6 G_{hkl}] \quad (1)$$

where d_{hkl} is the d-spacing measured as a function of azimuth angle ψ , d_{0hkl} is the d-spacing under the hydrostatic pressure, G_{hkl} is the appropriate shear modulus for a given hkl, and σ is the uniaxial stress. Pressure and stress were estimated using Ca-Pv (110) (200) and Au (111) diffraction in Pressure marker (Au : Fo = 1 : 2 volume ratio) at deformation experiments of Ca-Pv and Mg-Pv, respectively. An X-ray radiograph of the strain markers was taken using an imaging system composed of a YAG crystal and a CCD camera with an exposure time of 60 s.

Uniaxial stress of Ca-Pv at 13.8 GPa, 1473 K and $\sim 1.2 \times 10^{-5}$ /s and Mg-Pv at 23.0 GPa, 1273 K and $\sim 1.5 \times 10^{-5}$ /s were estimated as ~ 2 GPa and ~ 0.25 GPa, respectively. Stress of Mg-Pv was significantly smaller than that of Ca-Pv though temperature condition of Mg-Pv was lower than that of Ca-Pv. This fact is doubtful. This reason is thought that stress estimated by Au was much smaller than that of Mg-Pv because of framework made by Ringeoodite, which was polymorphic phase of Fo in pressure marker.

Keywords: In-situ measurements, deformation experiments, Stress, Strain, Perovskite, The Earth's lower mantle