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Perovskite–post-perovskite transformations in transition metal fluorides and oxides

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It is widely accepted that $MgSiO_3$-rich perovskite transforms to $CaIrO_3$-type post-perovskite at P,T conditions of the D" layer in the lowermost mantle. However, because of its stability exceeding 120 GPa and unquenchable nature at the ambient conditions, studies on analogue ABX_3 perovskites which transform to quenchable post-perovskite at lower pressure than $MgSiO_3$ would provide valuable insights on the post-perovskite transition. The ABX_3 post-perovskites have also been received considerable attention from materials science, because they may potentially have interesting physical properties derived from the low-dimensional structure. Using multianvil apparatus, we have studied on high-pressure behaviors of $\mathrm{Na}M^{2+}\mathrm{F}_3$ and $\mathrm{Ca}B^{4+}\mathrm{O}_3$ perovskites where M and B were transition metals.

Both $NaNiF_3$ and $NaCoF_3$ perovskites transform to post-perovskite at 16-19 GPa and 1200-1500 K with transition boundaries of approximately P(GPa) = -2.0 + 0.014T(K). Both $NaNiF_3$ and $NaCoF_3$ post-perovskites are quenchable at ambient conditions. At 19-20 GPa, $NaCoF_3$ post-perovskite further dissociates into two unquenchable Na-rich and Co-rich fluorides. $NaMnF_3$ perovskite does not transform to post-perovskite but dissociates into two fluorides. $NaNiF_3$ perovskite and post-perovskite exhibit antiferromagnetic transitions at 156 and 22 K, respectively. $\mathrm{Ca}B\mathrm{O}_3$ perovskites (B = Ru, Rh, Ir) transform to post-perovskite below 25 GPa. However, above 1500 K at 5-20 GPa, $CaRhO_3$ perovskite first transforms to a new-structured phase which further changes to post-perovskite. In the new structure, six chains of edge-sharing RhO_6 octahedra make a unit which stacks alternatively with CaO_8 polyhedra. The new structure is different from stacking-fault structures proposed by Oganov et al. (2005) as the intermediate structures between perovskite and $CaIrO_3$-type post-perovskite, and might suggest presence of another family of post-perovskite.

Combing these results with the previous ones on ABX_3 post-perovskites, several common features are suggested as follows. The perovskite–post-perovskite transition in $MgSiO_3$ is consistent with these features. In most of ABX_3 compounds, the perovskite–post-perovskite boundaries have dP/dT slopes of 10-17 MPa/K and volume change of -1.2–-1.8 %. ABX_3 perovskites with octahedron tilting angle (Φ) larger than about 15^{o} at ambient conditions transform to post-perovskite with increasing Φ with pressure. Also, covalent character of B-X bonds may play an important role to stabilize the post-perovskite structure. Our results suggest that $NaNiF_3$ is a good quenchable low-pressure analogue for $MgSiO_3$.

Inelastic X-Ray Spectrometer at the Advanced Photon Source for High Pressure Research Community*

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Measurements of thermo-elastic properties like elastic moduli, anisotropy in sound velocity, and dynamical viscosity under extreme conditions of pressure and temperature are essential in understanding material properties that is interest in mineral physics and geophysics. Inelastic x-ray scattering (IXS) spectrometer has emerged as technique of choice since it directly probes the collective excitations as a function of energy and momentum transfer space in entire Brillouin zone providing information on the thermo-elastic properties. Combined with seismic waves and ultrasound measurements, IXS measurements can provide new insight for geophysically relevant materials.

There are two dedicated IXS beamlines at the APS, 3-ID and 30-ID, with overall energy resolution of 2.1 and 1.6 meV. Currently, we are in process of improving their capabilities for the high pressure experiments. Both spectrometers are instrumented with micro focusing and diffraction capabilities to facilitate diamond-anvil-cell studies.

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Toward obtaining the experimental constraints on the role of water on melting under the lower mantle conditions

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Water and other volatile components (such as carbon dioxide) are known to have important influence on the melting behavior of silicates. The role of these components on melting under upper mantle conditions is now reasonably well understood. Recent experimental studies in our lab as well as some of the previous studies show that water has an important influence on the melting relationship under the lower mantle conditions. The influence of water is not only to reduce the solidus but also to change the composition of the melt to be (Mg,Fe)O rich.

Quantifying these observations is essential to understanding the chemical evolution of the Earth and other planets. However, there are several challenges in performing these experimental studies. In this presentation, we discuss the issue of quantifying the water effects with special attention to the capability of preserving water content during the high pressure-temperature experiments.

The issue of the preservation of water is important firstly because water can escape from the capsule during an experiment, and secondly because the melt is unquenchable in commonly used processes under lower mantle conditions. A commonly used practice is to identify the deficit of EPMA measurements from 100% to the water content, but there is no sound basis for this practice. In this presentation, we will show some preliminary results of our new approach to quantify the water content from the high-pressure run products containing melt.

Diamond anvil cell, its invention and 54 years of development

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The Diamond anvil cell (DAC) is a remarkable small device capable of subjecting samples to very high pressures by using small sample area rather than large applied force. The DAC was born when Alvin Van Valkenburg looked right through one of the diamond anvils and saw a sample while it was at high pressure. He and his colleagues, Charles E.Weir, Ellis R. Lippincott, Elmer N. Bunting, Gasper Piermarini, and Stanley Block at the National Bureau of Standards (later NIST) went on to develop many applications and to produce numerous studies taking advantage of the DAC's extraordinary features. Because of its very simple principle, the DAC has morphed into many diverse designs to take advantage of the large number of analytical techniques capable of analyzing micron-size samples. Virtually all of these applications start with the ability to look through the cell at the sample. The ability to subject fluids to high pressures in the DAC became possible when Van Valkenburg found he could encapsulate a fluid in a small metal gasket, a technique that also allowed more hydrostatic pressure in solids and also led to the ability to subject samples to extremely high static pressures. Early methods developed for determining sample pressure included equation of state applied to lattice parameter measurements of NaCl and the shift in fluorescence emissions of ruby. Several methods subsequently developed for heating high-pressure samples in DACs include external resistance heating, internal resistance heating, and laser heating. New techniques and new applications continue to be developed today, especially at synchrotron sources. Diamond anvil cell research continues to benefit from the complementary capabilities of multi-anvil devices and other high-pressure techniques.

Deformation of forsterite and enstatite aggregates at mantle PT

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A good knowledge of the mechanical behavior of olivine and enstatite at high pressure and high temperature is essential to model Earth upper mantle dynamics. In this study, we have performed deformation experiments on forsterite and on iron-free enstatite polycrystalline aggregates at upper mantle pressures and temperatures. Fine-grained forsterite powders were obtained by crushing a commercial forsterite in WC or zirconia grinders. Enstatite powders were synthesized by solid state reaction between mixed fine-grained powders of silica and forsterite in a conventional furnace. The powders were sintered by Spark Plasma Sintering (SPS) at 1000-1300°C and 100 MPa. We obtained aggregates with very low porosities (>99% dense) and well equilibrated microstructures with mean grain sizes of a few microns.

Compression deformation experiments were conducted on both types of aggregates in a D-DIA apparatus coupled with synchrotron white X-ray beam at the X17-B2 beamline at the National Synchrotron Light Source (Brookhaven National Laboratory, NY, USA). Strain and stress were measured in situ during deformation. Macroscopic strains were determined by measurements of sample shortening on X-ray radiographies. Stress and pressure were determined from the X-ray diffraction patterns of forsterite or enstatite collected on detectors arranged in different orientations with respect to the maximum principal stress.

Experiments were performed at pressures between 3 and 11 GPa and at temperatures ranging from 1100 to 1300°C. The samples were deformed to total strains of up to 30% with deformation rates ranging from 10^{-5} to 10^{-4} s^{-1}. We will discuss the effect of pressure, temperature and grain size on the rheology of forsterite and enstatite and compare our results with previous studies on forsterite and Fe-bearing olivine.

Deformation of polyphase aggregates, forsterite+MgO, at pressures and temperatures of the Earth's upper mantle

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Modelling the solid-state flow of the upper mantle requires a thorough understanding of its rheology and therefore necessitates to perform deformation experiments on mantle rocks (or analogues) at very high pressures and temperatures. Minerals other than olivine constitute up to 40 vol% of upper mantle rocks and may have a significant effect on the rheological behavior of these rocks. Nevertheless, most experimental studies to date have focused on the deformation properties of olivine single crystals or monomineralic olivine aggregates. In this study, and as a first step before focusing on more realistic mantle-like compositions, we have performed deformation experiments on polymineralic model aggregates of forsterite and MgO, at upper mantle pressures and temperatures. Commercial powders of Mg_2SiO_4 and MgO were mixed and ground in WC grinders and dried in a one-atmosphere furnace at 1000°C. Powders with different volume proportions of the two phases were sintered by spark plasma sintering (SPS) at temperatures of 1300–1400°C and 100 MPa for a few minutes, resulting in dense pellets 8 mm in diameter and 3–4 mm in length. Microstructural analysis by SEM reveals equilibrated microstructures with forsterite and MgO grain sizes of a few microns. Deformation experiments on samples 1.2 mm in diameter and $\sim$ 1.2 mm in length were performed at 3–8 GPa and 1000–1300°C in a D-DIA apparatus coupled with synchrotron X-ray radiation. The technique permits *in situ* measurement of macroscopic strain rates as well as stress levels sustained by different subpopulations of grains of each phase. Typically, two specimens, respectively a monomineralic and a polyphase aggregate, were deformed concurrently in order to minimize the relative uncertainties in temperature and pressure and to facilitate the comparison of their rheological properties. The samples were deformed to total strains of 15-25%. As expected, the harder phase, forsterite, sustains much higher stress levels than MgO, in agreement with numerical models for two-phase flow. Results on stress and strain partitioning in polyphase aggregates and on the effect of the presence of a weak phase on the rheology of forsterite-rich aggregates will be discussed.

Valence and Magnetic State in Europium at Extreme Pressures

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Changes in the valence state and magnetic properties of Eu metal under extreme pressures have been studied using x-ray absorption near-edge spectroscopy, x-ray magnetic circular dichroism and synchrotron Mössbauer spectroscopy to understand the pressure-induced superconductivity of Eu metal at pressures above 75 GPa. Contrary to previous reports about Eu's valence state, Eu is found to remain nearly divalent to the highest pressures reached (87 GPa) with magnetic order persisting up to 50 GPa at low temperatures. Density functional theory has been applied to give insight into the pressure-induced changes in both Eu's electronic structure and Mössbauer isomer shift.

Volatiles and Deep Earthquakes

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An alternative to metastable olivine is proposed in explanation of deep (>300 km) earthquakes. Essential in this pressure regime, where frictional sliding is inhibited, is local stress changes associated with either dehydration reactions and pore pressurization or anti-crack development due to metastable preservation of olivine. Based on high-pressure phase equilibria, temperatures near subducting slab interfaces, where volatile content is highest, exceed dehydration limits at depths shallower than several hundred kilometers. However, focal mechanisms for intermediate and deep earthquakes (to depths of 700 km) are associated with faults extending tens of kilometers from the interface into the colder core of downgoing slabs. Volatiles (including water, carbon dioxide, and methane) released in the high temperature zone can travel along faults to colder regimes where either freezing or reactions with rocks occur. The new volatile reservoir, in a colder and previously "drier" region, will be transported with the subducting slab. As conductive heating raises temperatures, further volatile release at greater depth is possible with fault orientations again allowing volatile transport still deeper into the colder slab core where freezing and back reactions are again possible. A series of earthquakes, triggered by devolatilization reactions, thus occur sequentially at increasing depths.

Support for metastable olivine as essential for deep earthquakes has been based on a correlation between earthquake locations and thermal models for specific subduction zones. The detection of elastic anisotropy in the putative metastable wedge regions is also an important observation. The current model is consistent with the thermal correlation for the distribution of earthquakes. Since small amounts of water in nominally anhydrous olivine suppress metastability, either deep earthquakes are not associated with metastable olivine or downgoing slabs are extremely dry. In contrast, the current model provides for deep transport of volatiles that can either be "leaked" into the deep mantle above subducting slabs or can be circulated into the deepest reaches of the mantle.

Equations of State for Aqueous Solutions

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We report accurate equations of state for several aqueous chemical system at pressures to several GPa from -20°C to 250°C. Sound velocities as a function of pressure, temperature, and composition were measured by the method of impulsive stimulated scattering. Densities were determined from thermodynamic relationships. Volumes of mixing were inferred with respect to the equation of state for water. A key improvement in the current analysis is the use of generalized geophysical inverse theory to determine thermodynamic parameters and their uncertainties. The methods outlined here have general utility in most high pressure and high temperature equation of state investigations.

Thermodynamic properties of aqueous solutions are described within a conceptual framework based on the properties of water and its interactions with molecular species. Such equations of state are used in the study of chemical, biological and fluid dynamic processes important to the fields of engineering, medicine and environmental and natural sciences. Aqueous systems are relevant for terrestrial studies ranging from the chemistry of seawater to chemical processes in hydrothermal systems. An understanding of processes occurring in icy moons in the outer Solar System also requires knowledge of the thermodynamic properties of solutions. Missing in prior studies has been sufficient high pressure data to constrain aqueous system behavior under pressures relevant to planetary interiors.

Microstructural Observations and Elastic Plastic Self Consistent (EPSC) Modeling of Plastic Deformation in San Carlos Olivine

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We report on in-situ synchrotron x-ray diffraction from high pressure deformation experiments conducted using polycrystalline San Carlos olivine. The experiments were conducted using the D-DIA apparatus at beamline X17B2 at the NSLS. We observe the diffraction behavior of x-ray reflections for lattice planes oriented nearly perpendicular to compression and at several other orientations including the transverse orientation for which lattice planes are nearly parallel to the plane that contains the beam direction and the compression direction. Sample strain is measured by periodically taking a radiograph of the sample (which is bounded by metal foils) and comparing the length of the sample to a radiograph taken immediately before the start of the deformation experiment. We used electron backscatter diffraction (EBSD) to evaluate the degree of preferred orientation development as well as the level of plastic strain within various subpopulations of grains within the samples. We also compare our diffraction data with elastic plastic self-consistent (EPSC) models of olivine deformation. As with previous studies, the models do not match the diffraction well. This can be explained in part by the fact that in our orientation image maps we observe some grains with low Schmidt factors that contain significant internal deformation and some grains with high Schmidt factors that remain comparatively undeformed. Thus the assumption made in the EPSC model that the Schmidt factor controls deformation is an oversimplification. These observations point to the importance of grain-to-grain interactions in the deformation of polycrystalline olivine.

Mineral Physics Educational Modules for Advanced Undergraduates and Graduate Students

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We have created a group of web-based educational modules on mineral physics topics and used them to teach on on-line course entitled “Introduction to Mineral Physics” during the spring 2012 semester. More than 20 students and postdocs as well as 15 faculty and senior scientists participated in the course which met twice weekly as a webinar. Recordings of faculty lectures and student-led discussions of journal articles are now available upon request and edited versions of the lectures will be incorporated into the educational modules. The modules reside in the On Cutting Edge, Teaching Mineralogy collection on the SERC website. Although the modules are designed to function as part of a full semester course, each module can also stand alone. Potential users of the modules include COMPRES members teaching “bricks and mortar” classes at their own institutions, COMPRES members teaching in a distance education setting, mineralogy teachers interested in including supplementary material in their mineralogy class, undergraduates doing independent study projects and graduate students and colleagues in other sub-disciplines who wish to brush up on a mineral physics topic. Our experience in creating the modules and the course indicates that the use of 1) community generated internet-based resources and 2) webinars to enable shared teaching between faculty at different universities, has the potential to both enrich graduate education and create efficiencies for university faculty.

The iron alloys of the Earth's core

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We estimate the necessary amount of several light elements - C, S, P, O, Si - as major alloying components to match the observed seismic properties of the Earth's inner core. For this we compute the elastic constants tensors and determine the seismic properties of Fe$_3$X compounds, with X = C, S, P, O and Si, using first-principles calculations. Assuming linear relations and similar temperature corrections of velocities, we obtain as most reasonable silicon and oxygen.

Then we compute the electrical conductivity of iron and iron alloys at Earth's core conditions from electron-phonon coupling in the ABINIT implementation. We find an excellent agreement with experimental results for pure hcp iron below 1 mbars. We confidently use our results up to core pressure conditions. We show that the conductivity exhibits saturation at high pressures. We treat in detail the effect of Si on hcp iron and show a decrease of conductivity with Si concentration, an increase in anisotropy and a strong dependence with the substitution pattern.

Electronic Spin Transition of Iron in Dense Hydrous Magnesium Silicate Phase D at High Pressure

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Among dense-hydrous magnesium silicates potentially transporting water into Earth's deep interior, phase D ($MgSi_2H_2O_6$) exhibits the widest stability range extending into the lower mantle along very-cold slab geotherms. Phase D contains nearly 12 wt% H_2O yet has a dense structure (ρ_0 = 3.3 g/cm^3) featuring Si in six-coordination with OH. Because subducted sediments in oceanic lithosphere include significant Fe and Al, the influence of these chemical substitutions on the structure and physical properties of phase D are under investigation, especially the possibility of pressure-induced spin transitions of Fe. Gem-quality single crystals of phase D were synthesized at 25 GPa and 1400C that are rich in Fe^{3+} (ferric iron) and Al with approximate composition: $MgSi_{1.5}Fe^{3+}_{0.15}Al_{0.3}H_{2.5}O_6$. Ferric iron occupies only octahedral sites in the crystal, allowing us to isolate the electronic spin transition of Fe^{3+} in octahedral coordination. Synchrotron X-ray emission spectroscopy was carried out to 90 GPa at the 16-IDD beamline (HPCAT) of the Advanced Photon Source. The compressibility of this sample was studied by X-ray diffraction in separate experiments to 85 GPa in a neon pressure medium. The experimental results reveal a gradual pressure-induced spin transition of Fe^{3+} between 35-70 GPa, which is also observed as a discontinuity in the compression curve. Because Fe^{3+} is isolated on one 6-coordinate site of this structure, results from this study are also useful in interpreting the nature of spin transitions of mixed-valence iron at multiple sites in other mantle phases such as silicate perovskite.

Magneto-elastic coupling in compressed Fe_7C_3 supports carbon in Earth's inner core

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The nature of light element(s) in the core holds key to our understanding of Earth's history of accretion and differentiation but the core composition remains poorly constrained. Carbon is among the leading candidates of the light elements in the core, which also include oxygen, sulfur, silicon and hydrogen. To test the hypothesis that carbon is a major constituent of the inner core, we investigated the structure, elasticity, and magnetism of the iron carbide Fe_7C_3 up to 167 GPa, well into the pressures of the core, using synchrotron-based single-crystal X-ray diffraction and Mössbauer spectroscopy techniques. Two discontinuities were detected in the compression curve up to 167 gigapascals (GPa), the first of which corresponds to a magnetic collapse between 5.5 and 7.5 GPa and is attributed to a ferromagnetic to paramagnetic transition. At the second discontinuity near 53 GPa, Fe_7C_3 softens and exhibits Invar behavior, presumably caused by a high-spin to low-spin transition of iron. Considering the magneto-elastic coupling effects, an Fe_7C_3-dominant composition can match the density of the inner core, making the core potentially the largest reservoir of carbon in Earth.

Texturing in Stressed Nickel Nanocrystals

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The plastic behavior of coarse-grained (usually referred to as micrometer-sized or coarser) metals is mainly controlled by the nucleation and motion of lattice dislocations. Plastic deformation due to dislocation glide results in crystallite rotations, generating lattice preferred orientation (LPO) or texture. How plastic deformation occurs in nanocrystalline materials is a long-standing and controversial topic. Whether plasticity in ultrafine nanocrystals is still generated by dislocations, how pressure affects the deformational regimes of nanoparticles, and how structural anisotropy is affected by size reductions are all questions that remain unresolved. Our goal is to examine the interplay between stress and particle size in determining when dislocation-mediated deformation processes predominate within nanoparticles and, correspondingly, when likely grain-boundary associated processes may be critical.

The size of nanocrystals provides a limitation on dislocation activity and associated deformation. Dislocation-mediated plastic deformation and texturing are expected to become inactive below a critical particle size, which is often thought to be between 10 and 30 nm based on computer simulations and TEM analysis. Through *in-situ* observations of texturing in stressed polycrystalline nickel of various mean particle sizes, we find that dislocation activities are still operative in 3 nm crystals. In radial diamond anvil cell (rDAC) x-ray diffraction experiments, significant texturing is observed at pressures above 3.0 GPa for 500 nm nickel and at >11.0 GPa for 20 nm nickel. Surprisingly, texturing is also seen in 3 nm nickel when compressed above 18.5 GPa. The observations of pressure-promoted texturing indicate that under high external pressures, dislocation activities can be extended down to a few-nanometer length scale.

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COMPRES Facility Report: Developments at Beamline 12.2.2 at the Advanced Light Source

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Beamline 12.2.2 at the Advanced Light Source is designed to offer a wide range of high-pressure/high-temperature capabilities for the geosciences community. These include readily interchangeable axial and radial diffraction geometries, two-sided laser heating and associated spectroradiometry, external heating capabilities, combined resistive and laser-heating designed to minimize temperature gradients, and recently implemented single-crystal diffraction capabilities. Advanced sample preparation capabilities include a computer-controlled laser mill and a soon-to-be-delivered high-pressure gas-loading apparatus using the GSECARS design. Recent scientific achievements at 12.2.2 include recognition of the separate effects of transformation and deformation on preferred orientation in magnesium-silicate post-perovskite, deformation experiments on iron-rich oxides under external heating conditions approaching 2000 K (Miyagi and co-workers), and deformational results constraining size constraints on dislocation mobility in nanocrystals. The hardware and software of our laser-heating system continues to undergo ongoing improvements with improved alignment diagnostics and better spectroradiometric characterizations of temperature gradients. In terms of new capabilities, S. Clark (ALS), P. Dera (GSECARS) and O. Tschauner (UNLV) and co-workers have recently set-up high-pressure single-crystal diffraction capabilities, with excellent initial validation results. Our future directions during COMPRES-III include: further improvements and upgrades in our laser-heating system; benchmarking and refining our external heating achievements; augmenting our successes in radial diffraction by exploring mixed-heating (laser- plus external-) approaches within these experiments; interfacing single-crystal capabilities with our heating approaches; and enhancing interactions between users of the 12.2.2 high pressure beamline and those of other beamlines at the ALS, such as the microdiffraction beamline 12.3.2 for chemical characterizations of different phases present in quenched samples: for example, these are capabilities taken extensive advantage of by Friedrich and Winkler's groups in their studies of binary transition metal nitrides, carbides and borides.

Water in the mantle: a key to reconcile fine structures between mineral physics and seismology models

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Study of mechanical properties of mantle minerals has unveiled many mysteries of Earth's interior alluded through seismic events. However, some details of seismic models remain unexplained. For instance, magnitude of seismic discontinuity at 410 km depth in seismic models is significantly larger than that derived from elastic properties of dominant minerals at such depth. For another example, although the attenuation of seismic anisotropy in the upper mantle at about 200-220 km depth can be attributed to switchover of active dislocation slip system in the dominant mineral, olivine, the depth and its variation are discrepant from that derived from the pressure at which such switchover is observed in the deformation experiment of mineral plasticity study. We have investigated influence of water on elastic and plastic behaviors of olivine through equation of state and rheological creep experiments using synchrotron x-rays at the X17C and X17B2 beamlines of the NSLS. Results indicate water significantly weakens the mineral. Elastically, 0.4 wt% H_2O in olivine results in a 5% reduction in bulk modulus (*i.e.* from 130 GPa for anhydrous sample to 123 GPa for hydrous sample). Plastically, structural H_2O in olivine influences different dislocation slip system very differently, and therefore alters the pressure of active slip system switchover with respect to "dry" sample. Even 30 ppm H_2O in weight may lower down the pressure for transition from [100](010) slip to [001](010) slip by 2 GPa (*i.e.* from 8 GPa in dry condition to 6 GPa in wet condition). Implications of these results will be discussed in this presentation. Together with previously reported results on elastic and plastic properties of mantle dominant minerals, we are able to reconcile the discrepancies between mineral physics and seismology models for the magnitude of 410 km discontinuity and for the attenuation of seismic anisotropy at about 200-220 km respectively.

TEM studies on olivine and diamond samples deformed in the D-DIA

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As part of the high pressure deformation studies, we examine dislocation microstructure of polycrystalline olivine and diamond samples deformed in the deformation DIA (D-DIA), to gain physical insights into deformation mechanisms. Here we report our preliminary results based on observations using Transmission Electron Microscopy (TEM).

Polycrystalline samples of San Carlos olivine were deformed at high-pressure (2.81–5.09 GPa), high-temperature (1458 to 1626 K), and strain rates between 0.98×10^{-5} and 1.35×10^{-5} s^{-1}[1]. The microstructure of deformed olivine is dominated by free dislocations and sub-grain boundaries. The density of free dislocations is about 3.1×10^{9} cm^{-2} within the studied area, corresponding to a differential stress of ~780 MPa [2]. This agrees well with the x-ray determined stress of 897 MPa for the last deformation cycle [1]. Slip under these conditions was dominated by dislocations with the Burgers vector **b** = [001], as all dislocations are out of contrast with g = 040 and g = -110 in TEM observations. Most of the straight dislocations in this area are screw dislocations. On the other hand numerous curved dislocations are present, indicating high-temperature induced dislocation climb.

The polycrystalline diamond sample studied here was from GE (with Co as binding material) and was deformed at 8 GPa, 1273 K, and strain rates about 10^{-5} s^{-1}[3]. Maximum axial strain was ~15%. The sample contained certain amount of porosity. High density of dislocations was observed in bright-field images, indicating significant plastic deformation, consistent with stress-strain curves determined in the D-DIA. Dislocation distribution appears heterogeneous, presumably due to the presence of voids.

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The Diamond Anvil Cell X-ray Facilities (X17-DAC) at NSLS

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As the first dedicated high-pressure beamline in the world, the diamond anvil cell X-ray (X17-DAC) facilities at National Synchrotron Light Source (NSLS) have made significant contributions in high-pressure research in Earth Science, Physics, Chemistry and Materials Sciences for more than two decades. The X17-DAC beamlines (X17C and X17B3) have high photon flux (4×10^{14} photons/second/0.1%BandWidth @ 30 keV) from a superconducting wiggler source. The facilities have served 30 research groups (over 90 users) from 28 institutions since 2009, resulting in 30 peer-reviewed articles annually. The two supporting laboratories for sample preparation and pressure measurement house newly updated equipment and serve users from more than 7 beamlines at NSLS.

Both X17C and X17B3 beamlines are available for energy dispersive (EDXD) and angle dispersive (ADXD) x-ray diffraction. Monochromatic beams with 10-20 micron diameter beam size and high flux are provided through the use of Sagittally-bent double Si crystal Laue mode monochromators. At X17C, both monochromatic beam (30keV) and white beam (5-80keV) can be focused by Kirkpatrick-Baez mirrors for x-ray diffraction at the extreme conditions of pressure and temperature for powder, single crystal and nanocrystalline samples. At X17B3, in addition to EDXD, two modes of monochromatic beam for ADXD are available: A focused 30 keV beam combined with in-situ laser heating provides high-pressure and high temperature capabilities; an unfocused 80keV beam can measure ADXD up to a very high Q range for a total scattering experiments.

The time-dependent volume relaxation in vitreous silica characterized by Gigahertz ultrasonic interferometry

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Noted nearly a century ago by Bridgman (1925, 1939, 1948) vitreous SiO_2 (v-SiO_2) displays anomalous compressibility up to 3 GPa. The mechanism causing the anomalous compressibility has been subject of much debate with recent work indicating the presence of a polyamorphic transition (Clark et al., in prep; Deschamps et al., 2009; El'kin et al., 2002; Huang and Kieffer, 2004; Walker et al., 2007). Here we explore the utility of gigahertz ultrasonic interferometry to probe the timescales for this polyamorphic transition as reflected by time-dependent changes in P- and S-wave velocities. We conducted experiments on v-SiO_2 with 76 ppm OH from 0 to 6 GPa compressed in the DAC. The wavelength of a microns eliminates unwanted diffraction making gigahertz ultrasonic interferometry well suited to the study of glasses. The time-dependent velocity changes within the region of anomalous compressibility displays complex behavior with the percent change between the initial and stabilized velocity increasing with pressure up to a maximum percent change at 0.5 and 0.9 GPa for P- and S-waves, respectively, both within the region of maximum volume reduction. At pressures above the maximum the percent change decreases with increasing pressure up to 6 GPa. Gigahertz ultrasonic interferometry provides the first observation of this time-dependent behavior for v-SiO_2 in the region of anomalous compressibility. This time-dependent behavior is likely related to large scale density fluctuations resulting in the slowing down of relaxation near the phase transition.

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Measurement of activation volume and determination of preferred slip systems for creep of dry olivine at upper mantle pressure

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Olivine is the most abundant and probably weakest phase in the upper mantle, and thus its rheological properties have a critical role in controlling convective flow in this region. A persistent obstacle to understanding the behavior of olivine in the mantle has been the difficulty of determining activation volume (*V**), the influence of hydrostatic pressure on high-temperature creep. The bulk of previous studies examining *V** were conducted at low pressure (<300 MPa) and over small pressure ranges in gas-medium deformation machines, limiting precision and raising questions about application to relevant geological conditions. For this study, we conducted numerous deformation experiments on dry polycrystalline olivine in the D-DIA apparatus to pressures 1.5 to 9 GPa at 1373 K. Stress and strain were measured in-situ with synchrotron x rays. Refinement of diffraction technique has allowed stress resolution of ±0.02 GPa. For the pressure range in this study, we have measured an average activation volume of about 15 cm^3/mol for dry polycrystalline San Carlos olivine. This is a substantial pressure effect, representing a pressure-induced viscosity increase of nearly 7 orders of magnitude from the base of the lithosphere to the bottom of the upper mantle. The diffraction technique used for stress measurement in these experiments also illuminates the relative strength of differently oriented grains in our polycrystalline sample, providing new experimental evidence for preferred dislocation slip systems in olivine at high pressure.

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Deuterium-Hydrogen Exchange in Olivine: Implications for Point Defects and Electrical Conductivity

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Hydrogen diffusion in mantle minerals is important for understanding hydrogen transport, mixing, and reservoirs in the mantle. Knowledge about hydrogen self diffusion (D_H) is critical for determining mantle hydrogen distribution and understanding point defects. Additionally, chemical diffusion of hydrogen in olivine, such as redox exchange with polarons (D_{Redox}), depends on D_H. We measured D_H in olivine from deuterium (^2H) - hydrogen (^1H) exchange experiments performed at 2 GPa, between 750-900 °C. Samples were sectioned so that ^2H profiles could be measured for each principle orientation of olivine by secondary ion mass spectrometry. The resulting ^2H profiles were fit to obtain the hydrogen self diffusion coefficient for the [100] orientation, $D_{H,[100]} = 10^{(-4.9 + 1.4)} * e^{(-140+ 30kJ/mol)/(RT)}$ m^2/s. This is ~1 log unit lower than $D_{Redox, [100]}$, with similar activation enthalpy. By comparison of $D_{H,[100]}$ and $D_{Redox, [100]}$, we estimate the small polaron diffusion coefficient and D_H in the [010] and [001] orientations. These D_H values were used with the Nernst-Einstein relation to estimate the contribution of hydrogen to electrical conductivity in olivine, $\sigma_H = 10^{1.1} * e^{(-130kJ/mol)/(RT)}$ S/m for 10^{-2} wt% H_2O. We calculate σ_H that are lower in magnitude than previous measurements, and suggest that hydrogen cannot account for high electrical conductivity anomalies observed at asthenospheric depths (~10^{-2} to ~10^{-1} S/m). The maximum anisotropic variation of D_H and σ_H in olivine is ~2 log units between 750-900 °C, and increases with extrpolation to higher temperature. This indicates that hydrogen could account for the range of electrical conductivity anisotropies observed in the upper mantle if hydrogen was abundant.

Reaction time scale of partial melting of KLB-1 peridotite and temperature gradient effect

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To address the question whether partial melting is related to seismic low velocity zone in the Earth's mantle, it is crucial to quantify the reaction time scale between melt phase and the solid phase. If the time scale is close to or lower than the seismic period, then partial relaxation of P-wave and S-wave velocity will occur. If the time scale is much larger than seismic wave period, seismic wave velocity will not be softened by the melting process. During a partial melting process, two variables need to be considered, one is the time required to generate certain amount of melts; two is the time to reach chemical equilibrium among melts and solid phases. The former directly relates to the volume reduction and elastic modulus of the bulk sample.

Thermodynamic models using MELTS software were carried out to quantify melts and the composition of residue solid phases and to generate the relation between density-composition-P-T, plus the effect of water. Two series of melting experiments were carried on KLB-1 peridotite in multi-anvil apparatus at pressure around 1.5-2.0 GPa. Compositions of solid residues of these experiments were determined by electron microprobe.

Three melting experiments were carried out at same pressure and three different temperatures to produce different amount of melt of KLB-1 peridotite. During each experiment, the sample was heated for 30 minutes at the highest temperature and then quenched. The three experiments produced 4%, 20%, and 40% melts respectively. Comparison of $Mg^{\#}$ of olivine from melting residue with thermodynamic data from MELTS algorithm shows: (1) KLB-1 peridotite starts to melt at ~1300 °C; (2) at ~1600 °C, there's almost no clinopyroxene left and melting percent is as high as 40%. Using the electron probe data obtained for the recovered samples, we find that there is ~ 100 K temperature gradient in the hottest region and coolest region in the sample.

Two more experiments were carried out under same pressure and temperatures ~1500°C but with different heating time 1 minute and 10 minutes. The experiment at the same conditions but heated for 30 minutes generated 20 % melts. Backscatter electron images show clearly that melting occurred in triple junctions in KLB-1 samples in all three heating experiments. Visible Compositional zoning is clearly visible in large olivine and orthopyroxene grains in the 1-minunte and 10 minutes heating runs. We find that (1) heating time longer than half an hour is necessary to achieve complete equilibrium between solid and liquid phases at this temperature and pressure domain; (2) the highest $Mg^{\#}$ of olivine from these three experiments agree well with each other, which indicates that similar percentage of melting is approaching; (3) Melting of KLB-1 peridotite that happened in seconds at 1.5~2.0 GPa and ~1500 °C will cause tangible volume reduction.

Resistively-heated diamond-anvil cell to temperatures greater than 1300 K

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Generation of homogeneous high temperatures in the diamond-anvil cell (DAC) has been a great challenge over the past several decades. To overcome disadvantages of small heating areas (< 30μm) and large temperature gradients (> 100K/μm) inherent in the laser-heated DAC as well as the limited pressure range (< 60 GPa, but routinely < 25 GPa) in multi-anvil press experiments, we have developed an externally resistive-heated DAC to produce uniform high temperatures at high pressures. In this study, we modify a previous design employing graphite sheets (Liermann et al., *RSI*, 2009) with a ring-shaped graphite heater. In a pilot run, we successfully generated temperatures of ~1300 K at a nominal pressure of 40 GPa. To test this newly designed heater, we investigated the well-determined melting curve of water up to 30 GPa. This technique will open doors to studying material properties, particularly melting, transport and elasticity at high temperatures and pressures.

Deformation of olivine-orthopyroxene aggregates: Implications for the strength of the lithosphere

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In this study we aim to address the long-standing discrepancy between strong olivine rheology and the weak lithosphere required for plate tectonics to occur. We deformed a realistic upper mantle mineral assemblage composed of 75 wt. % olivine and 25 wt. % orthopyroxene under (nearly) lithospheric conditions to study the role of a second phase on bulk rheology and evolution of microstructure. We use synthetic hot-pressed aggregates as starting materials. The recovered specimens typically demonstrated relatively coarse-grained (10-50 μm) equilibrated microstructures. We carried out successive constant strain rate ($\sim 10^{-4}$ s^{-1}) simple shear deformation experiments on ~0.4 mm thick slices of the hot-pressed material for a range of temperatures (1173-1553 K) and shear strains (0.7 – 3.0). Below 1273 K, specimens showed continuous hardening until the strength exceeded the confining pressure. At 1373 K and higher, "yielding" occurred at stresses less than the confining pressure. Both at 1373 K and 1473 K substantial weakening occurred after the first "yield", and the strength at the higher strains was smaller than that of a pure olivine aggregate. The microstructural observations show high strain deformation of some orthopyroxene grains and resultant dynamic recrystallization. Small orthopyroxene grains, formed by dynamic recrystallization, mixed with recrystallized olivine grains. Therefore, we suggest that dynamic recrystallization of orthopyroxene promotes rheological weakening of the olivine and orthopyroxene mixture. However, we do not observe such weakening at ~1550 K. By comparing the recrystallised grain-size stress relationship with the deformation mechanism map (of olivine), we interpret that at this temperature, average recrystallized grain size is too large to deform the rock within the grain size sensitive creep domain. Our experimental observations show that rheological weakening is enhanced by the presence of orthopyroxene via mechanisms such as grain boundary pinning with possible weak orthopyroxene rheology and that the weakening occurs only in a limited temperature window. Possible implications of these observations to the presence of plate tectonics on terrestrial planets will be discussed.

Single-Crystal X-Ray Diffraction of Pyrope to 50 GPa

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Aluminosilicate garnets are some of the most common and accommodating crystal structures, and are major constituents of the interiors planets, potentially remaining metastable to high pressures in subducting slabs. However there are few previous studies on their cold compression behavior, especially at higher pressures. We have collected preliminary single-crystal X-ray diffraction data on the high-pressure behavior of near end-endmember natural pyrope (Dora Maira pyrope) to ~50 GPa. Up to this maximum pressure we observe only smooth compression of the pyrope, and no discernable phase transitions. This work extends the pressure for which crystallographic information has been collected on single-crystal pyrope by ~20 GPa, as previously, the highest pressure single-crystal work had been limited to ~30 GPa. Our data is consistent with previous diffraction studies of pyrope and provide improved constraints on the stability, equation of state, and structural evolution of this fundamentally important mineral.

Equations of state and phase diagrams of iron-silicon alloys

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The outer core of the Earth contains several weight percent of one or more unknown light elements, which may include silicon. Therefore it is important to understand the high pressure, high temperature properties and behavior of iron-silicon alloys with geophysically relevant compositions in the iron-rich Fe-FeSi system. We have studied Fe-9Si, Fe-16Si, and FeSi to megabar pressures and high temperatures using synchrotron X-ray diffraction in a laser-heated diamond anvil cell. Room temperature compression experiments were conducted at beamlines 12.2.2 of the Advanced Light Source and X17C of the National Synchrotron Light Source, and laser-heating experiments were performed at beamline 13-ID-D of the Advanced Photon Source. We determined the equations of state, subsolidus phase diagrams, and melting temperatures for each iron-silicon alloy that we studied (Fischer et al., submitted; Fischer et al., in prep). For Fe-9Si and Fe-16Si, a B2+hcp mixture is stable at the pressures relevant to the Earth's outer core, so eutectic melting should be expected under these conditions. Comparing our new equations of state to that of iron (Dewaele et al., 2006) and the seismologically determined density of the core (Dziewonski and Anderson, 1981), we find that for an Fe-Ni-Si outer core, 11.3 ± 1.5 wt% silicon would be required to match the core's observed density at the core-mantle boundary, making this the maximum amount of silicon that could be in the outer core.

New development of spin transition problems of iron in lower mantle minerals

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It is now widely recognized that spin transitions of iron in lower mantle minerals largely affect the various phenomena of the lower mantle. Nevertheless, spin transitions of iron in lower mantle minerals have been unclear except for ferropericlase. In particular, the spin transition of iron in Mg-perovskite (Pv) have been an issue of much controversy, because there are large discrepancies among the previous reports. However, very recently there is a sign that those discrepancies will be resolved through two approaches: one is the theoretical studies on quadrupole splitting of Fe^{2+} in Mössbauer spectra of Pv and post-Mg-perovskite (PPv), and the other is the experimental studies on cation exchange reaction of Fe^{3+} and Al between the A- and B-sites in Pv and PPv. Here we report our recent experimental results on the effects of Fe^{3+} and Al exchange reaction by annealing on the spin states of Fe^{3+} in Pv and PPv at pressures up to 200 GPa (Pv) and 165 GPa (PPv), respectively, using synchrotron X-ray emission spectroscopy and X-ray diffraction. These results strongly indicate that the large discrepancies of the spin state of Fe^{3+} in the previous reports of the lower mantle minerals will be resolved by the Fe^{3+} and Al exchange reaction between the A- and B-sites in Pv and PPv. Based on these results combined with the recent theoretical studies on Fe^{2+}, the spin transitions of iron in whole lower mantle minerals are discussed.

Earth and Planetary science at the high pressure beamline ID27, ESRF

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In the last decade, we have witnessed an unprecedented surge in high-pressure research that has greatly improved our fundamental understanding of materials under high compression. The X-ray investigation of matter under extreme conditions has became one of the major activities at the ESRF and other 3rd generation synchrotron sources. The array of techniques that was initially restricted to structural measurements using X-Ray diffraction is now extended to include many other X-ray techniques such as IXS, NIS, EXAFS, XMCD, X-ray Compton scattering and X-ray magnetic scattering. As a direct consequence, many scientific breakthroughs have been achieved across fields ranging from Earth and planetary sciences to fundamental physics, chemistry and materials research, and extending into biophysics/biochemistry including questions concerning life and biological function under extreme conditions.
In this context, ID27 is a major resource for the European physics, structural chemistry, materials science, Earth science and structural biology communities.
Beamline ID27 is one of the most recently upgraded high pressure, high energy beamline worldwide and operates in user mode since 2006 in replacement of ID30, an instrument which was mostly dedicated to white beam experimentation. This instrument is fully optimised for monochromatic high resolution XRD in order to address the most exciting and challenging questions related to science at very high pressures. The beamline can easily accommodate very complex sample environments such as the double-sided laser heating system (CO_2 and YAG), the Paris-Edinburgh press and the HP helium cryostat. These techniques are powerful tools to explore a very wide pressure ($P>2$ Mbar) and temperature domain ($5<T<5000$ K). The beamline components (source, optics and detectors) are entirely designed to give the best possible response to these very demanding conditions. In this presentation, I will give an overview of the beamline capabilities in the field of Earth and planetary science.

Aseismic Deformation of Dehydrating Serpentinite

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Serpentinite is one of the main constituents of the subducting slabs. Its dehydration is believed to be responsible for triggering earthquakes at intermediate depths (i.e., 60-300 km). Based on experimental results, some authors have proposed mechanisms that explain how brittle deformation can occur despite high pressure and temperature conditions [1]. However, reproducing microseismicity in the laboratory associated with the deformation of dehydrating serpentinite remains challenging. A recent study showed that, even for fast dehydration kinetics, ductile deformation could take place rather than brittle faulting in the sample [2]. This latter study was conducted in a multi-anvil apparatus without the ability to control differential stress during dehydration.

We have since conducted controlled deformation experiments in the deformation-DIA (D-DIA) on natural serpentinite samples at sector 13 (GSECARS) of the APS. Monochromatic radiation was used with both a 2D MAR-CCD detector and a CCD camera to collect, respectively, full diffraction rings and radiographies of the samples. The data allow us to determine stress and the strain of the sample during the deformation process [3]. In addition, an Acoustic Emission (AE) recording setup was used to monitor the microseismicity from the sample, using piezo-ceramic transducers glued on the basal truncation of the anvils.

The samples were deformed at strain rates of 10^{-5}-10^{-4} s^{-1} under confining pressures of 3-5 GPa. Dehydration was triggered during the deformation by heating the samples at rates ranging from 5 to 60 K/min. Before the onset of the dehydration, X-ray diffraction data showed that the serpentinite sustained significant amounts of stress which plummeted when dehydration occurred. No AE events ever accompanied this stress drop, suggesting ductile deformation of the samples. Hence, unlike many previous studies, we document here an aseismic interplay between deformation and dehydration of natural serpentinite. These results suggest that dehydration of serpentinite alone cannot be responsible for intermediate earthquakes. The presence of other phases with greater strength (capable of sustaining higher stress levels) is required to reconcile the experimental data with the seismological observations.

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X-ray powder diffraction facility at NSLS-II

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An X-ray Powder diffraction (XPD) beamline is being built at the new synchrotron X-ray source (NSLS II) at Brookhaven National Laboratory, USA. The optical scheme takes full advantage of the high flux of a 7m long 1.8T damping wiggler, and uses techniques and instrumentation pioneered or under development at the NSLS (*e.g.*, Laue optics and Ge strip array detectors). Using three experimental hutches, the XPD powder diffraction beamline will become a multi-instrument facility with the ability to collect diffraction data at high monochromatic x-ray energies (30 keV-80 keV), offering rapid acquisition (milli-second) as well as high angular resolution capabilities. XPD is designed to combine sample-limited Q-space-resolution diffraction measurements and sample-limited real-space-resolution Pair Distribution Function measurements. The beam size is adjustable to match the graininess and heterogeneity scales of the samples above the micron scale. XPD addresses future scientific challenges in *e.g.*, hydrogen storage, CO_2 sequestration, advanced structural ceramics, catalysis, and materials processing. Such materials of high technological value often are complex, nanostructured and heterogeneous. The scientific grand challenge is to obtain robust and quantitative (micro)structural information, not only in the ground state at ambient conditions, but also *in situ* or *in operando* with varying temperature, pressure, magnetic, electric or stress field, chemical environment, *etc*. The beamline will be fully commisioned by March 2014 and full user operation mode begins by June 2015.

Effect of water on olivine single crystals deformed under high pressure and high temperature

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Earth's interior is full of mysteries. While seismic waves, the only probe accessible to the Earth's interior, give abundant information about Earth's structure to us, they also bring many puzzles for us to solve. For example, anisotropy of the seismic velocity shows a significant attenuation at about 220km, but the cause of such attenuation has been long under a debate. It is believed that lattice preferred orientation (LPO) of mineral may give rise to seismic anisotropy (e.g., Wenk et al., 1985). An early model illustrates the attenuation through a change in deformation mechanism from dislocation creep to diffusion creep in the mantle (Karato et al. (1993). As dislocation creep may result in LPO and diffusion creep does not, such a change in deformation mechanism will cause disappearance of seismic anisotropy. Nevertheless, considering the observation that a xenolith from depth deeper than 220 km shows strong LPO (Jin, 1995; Ben Ismaïl et al., 1998; Ben Ismaïl et al., 2001) and the evidence for strong seismic anisotropy in the deep continents (Gung et al., 2003, Karato, 2010) recently pointed out "the hypothesis of transition from dislocation creep in the shallow upper mantle to diffusion creep in the deep upper mantle does not have strong support.". This research takes the advantage of brilliant high energy synchrotron x-rays to study the influence of the major volatile in Earth's mantle (i.e. water) on dislocation slip systems of mantle minerals. A single crystal deformation technique is developed at the X17B2 beamline. Significant weakening effect due to the presence of hydroxyl in San Carlos olivine, $(Mg, Fe)_2SiO_4$ is observed. Study on olivine indicates that water reduce the pressure required to change the active slip system from [100](010) to [001](010), which could make dislocation slip switchover an excellent origin for the seismic anisotropy attenuation, solving this long standing puzzle in Earth science community.

This research extends the experimental capability for the single crystal deformation up to 10 GPa integrating a multianvil deformation DIA apparatus with the high energy x-ray beam at the X17B2 beamline. Deformation experiments were carried out in uniaxial compression along $[110]_{c,}$ $[011]_c$ and [101]c crystallographic directions, at pressure ranging from 4 to 8GPa and temperature between 1373 and 1473K. These three orientations mainly activate respectively [001](010), [100](010) and simultaneously [100](001), [001](100) slip systems. Talc sleeves about the annulus of the single crystals were used as source of water in the assembly. A polycrystalline stress sensor is introduced to the deformation column so that the sample stress can be measured through diffractions by multiple detectors at different azimuth angles while the sample strain in response to applied stress is directly monitored using radiography imaging of the sample. In addition, micro-focused IR beam at the U2 beamline enables an accurate mapping of water content cross the deformed single crystal samples through FTIR spectroscopy.

TEM investigation reveal numerous dislocation dipoles in annihilation process indicating that the presence of protons might favor the dislocation glide assisted by climbing during the deformation, essential for the annihilation of dipole. This may explain the hydrolytic weakening observed in this study. By direct comparison of single crystals strain rates, we observed that [110]c deforms faster than [011]c below 5GPa. However above 6GPa [011]c deforms faster than [110]c. This indicates that [100](010) is the dominant slip system below 5 GPa, and above 6 GPa [001](010) becomes dominant. The study of [101]c orientations shows a large sensibility to temperature between 1273-1473K. With around 4-5GPa, [101]c is weaker than [110]c and [110]c at 1473K, and [110]c becomes weaker than [101]c at 1273K. According to our results, the slip system transition, which is induced by pressure, occurs at 6 GPa. Water influences the pressure where the switchover occurs, by lowering the transition pressure from 8 GPa (corresponding 260 km in depth) to 6 GPa (corresponding 220 km in depth). The pressure effect on the slip systems activity has been quantified and the hydrolytic weakening has also been estimated for each orientations. This result supports Mainprice (2005) and Karato (2010), and shows that in presence of water, slip systems transitions occurs between 5-6GPa (200-250Km depth) where the seismic anisotropy attenuation is observed.

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Stabilities of $Fe_3(P_xS_{1-x})$ in Earth and planetary interiors: experimental studies with multi-anvil apparatus and diamond anvil cell

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The Earth's and planetary cores are composed of iron, nickel and a certain amount of light elements, among which sulfur is widely accepted as a possible candidate. In the iron-rich side, Fe_3S could be a dominant phase in planetary interiors. Phosphorus is also unambiguously a candidate light element in planetary cores due to the common occurrence of iron phosphides in differentiated iron-rich meteorites, which are generally believed have been derived from molten cores of parent bodies. The coexistence of iron phosphides minerals with iron sulfur nodules is often observed in enstatite chondrites and iron rich meteorites. Since many Fe-P minerals are isostructural with Fe-S minerals, phosphorus is found to dissolve in crystalline iron at 23 GPa in Fe-S-P system up to 4 wt% and are likely to form extensive solid solutions with sulfur. The high pressure behaviors of $Fe_3(S,P)$ solid solutions will provide valuable insights into the structural evolution of Fe_3S and implications for phosphorus storage in planetary cores. Here we explored the phase boundary of $Fe_3(S,P)$ up to 25 GPa, 2000 K by multi-anvil facilities and synthesized $Fe_3(P_xS_{1-x})$ solid solutions (x=0.25, 0.5, 0.75) under elevated pressures and temperatures. The homogeneity of synthesized samples was exanimated using Scanning Electron Microscope and Energy Dispersive X-Ray Spectrometer (SEM/EDS), and their structures were confirmed by X-ray diffraction at Geophysical lab. We presented new synchrotron X-ray diffraction data of Fe_3P in diamond anvil cell up to 30 GPa, 1600 K at APS and compared the X-ray diffraction patterns of high-pressure polymorph with our previous calculations.

The Elasticity of Synthetic Polycrystalline Grossular and Almandine Garnet at High Pressures and High Temperatures.

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Acoustic wave velocities for synthetic polycrystalline grossular [$Ca_3Al_2(SiO_4)_3$] and almandine [$Fe_3Al_2(SiO_4)_3$] were measured up 8 GPa and temperatures up to 1000K by ultrasonic interferometry in conjunction with energy-dispersive synchrotron X-ray diffraction in a cubic anvil deformation DIA-type apparatus (D-DIA). Travel times of compressional (P) and shear (S) waves and X-ray diffraction data were collected after heating and cooling at high pressures to minimize effect of non-hydrostatic stress on the measurements. Elastic bulk (K_s) and shear (G) moduli data obtained at the end of the cooling cycles were fitted to functions of Eulerian strain to third order, yielding pressure derivatives of the elastic moduli $(\partial K_s/\partial P)_T = 4.4$ (2); $(\partial G/\partial P)_T = 1.28$ (5) for grossular and $(\partial K_s/\partial P)_T = 4.3$ (1); $(\partial G/\partial P)_T = 1.13$ (3) for almandine. Both $(\partial K_s/\partial P)_T$ and $(\partial G/\partial P)_T$ are identical within mutual experimental uncertainties for both garnet compositions. Moreover, the new pressure derivatives of the elastic moduli are also equal within experimental uncertainties, to those of end-member pyrope garnet Py_{100} (*Gwanmesia et al., 2006*), and of $Py_{60}Mj_{40}$ and $Py_{50}Mj_{50}$ (*Gwanmesia et al., 2009*), from previous ultrasonic studies, thus indicating that the effect on the pressure derivatives of the elastic moduli due to cation (Ca, Na, Fe) substitutions in the garnet structure, observed in this study is minimal or insignificant.

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New High-Pressure Neutron Beamline PLANET in J-PARC

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The PLANET is the new high-pressure neutron beamline constructed at the intense pulsed neutron source J-PARC. This beamline aims at revealing the effect of water on dynamics of the interior of the Earth and planets by using the neutron character that is sensitive to hydrogen. The most characteristic feature of this beamline is to possess the huge 6-axis press with the maximum load of 500ton/axis, which can simultaneously generate high-pressure and high-temperature condition of 20GPa and 2000K (see Sano *et al.*). Coupled with the state-of-the-art techniques in neutron diffraction and radiography, the beamline offers the microscopic and macroscopic information of materials at high-PT condition.

The PLANET has the specification which can analyze the structures of both crystalline and non-crystalline materials to satisfy the wide demands of various users. The beamline enables us to obtain data with the *d*-value of 0.21-4.09A with the resolution, Δd/d of less than 0.5%. The neutron beam emerged from the moderator is cut with choppers and collimators, and is transferred into the experimental hutch using supermirror guides. The beam is injected into the compressed sample through the anvil gap and the neutrons scattered along 90 degree direction are detected with ^{3}He position sensitive detectors. The neutrons passing through the sample are detected with the neutron color image intensifier. To reduce the background, the detector banks are equipped with radial collimators which reject the parasitic scattering from sample surrounding materials. For high-pressure generation, 6-axis press is mainly used, in which the 6-6 or 6-8 compression method is applied for quick replacement of the radiologically activated samples and anvils.

We stared designing at 2008 and finished the construction on the last March. We received the first beam on March 7, 2011. During the first beam ceremony celebrating it, we experienced the terrible earthquakes on March 11. In spite of unbelievably strong shaking, the damage for beamline was smaller than expected. Now we received the beam again and started the beam commissioning. The beamline is used by the project members before the next March, and then it will be opened for general users.

Changes in structure of hydrogen hydrate under low temperature and high pressure above 5 GPa

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Low-temperature and high-pressure experiments were performed with filled ice Ic structure of hydrogen hydrate in an unexplored conditions of 5 to 50 GPa and 30 to 300K using diamond anvil cells and a helium-refrigeration cryostat. *In-situ* X-ray diffractometry revealed that cubic filled ice Ic structure transformed to a tetragonal structure at low temperatures and high pressures, and that the axis ratio c/a of the tetragonal phase changed depending on pressure and temperature. These results were totally consistent with a theoretical prediction by first principle calculation. And, the existing region of the tetragonal phase was determined to be above 20GPa at 300 K, above 15 GPa at 200K, and 10 Gpa at 100K, respectively.

Recent progresses in high-pressure research with diamond-anvil cell at SPring-8

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Synchrotron radiation has become an important tool for high-pressure science. Recent advances of synchrotron-based X-ray techniques in diamond anvil cells (DAC) have greatly enhanced our ability to study condensed matters physics under extreme conditions, including Earth and planetary sciences and materials science. Techniques such as X-ray diffraction (XRD), X-ray spectroscopy (absorption, X-ray Raman, emission), nuclear resonant scattering, inelastic X-ray scattering, and SR infrared spectroscopy are used for understanding structural, electronic, magnetic, chemical, and phonon properties of materials at high pressure. Research under extreme conditions is being performed at many beamlines at SPring-8. Among them, high-pressure research beamline BL10XU at SPring-8 is a dedicated to high-pressure powder XRD study in a DAC. To enhance the high-pressure DAC research program, the developments in high-pressure synchrotron radiation techniques has been carried out constantly at BL10XU: X-ray optics, diffraction system, and DAC instrumentation. One of recent focus in high-pressure facility upgrade is to integrate multiple techniques and then establish simultaneous measurement system, for understanding various high-pressure phenomena. Specific examples as integration of multiple techniques in combination with XRD include (1) XRD and optical Raman spectroscopy for studying dynamics of atoms and characterization of materials, (2) XRD and Brillouin scattering for simultaneous measurement of both density and sound velocity, (3) XRD and electrical conductivity measurement for determining electronic properties, and (4) XRD and SR Mossbauer spectroscopy for studying magnetic properties and charge states. In this paper, several current developments will be discussed.

Time dependence of phase transformations under high pressure and temperature

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Kinetics of phase transition has been studied effectively in materials science and related technology, such as metallic heat-treatment and food process etc. In these fields temperature-time-transition (TTT) diagram is widely applied for understanding the temperature-induced phase transition. On the other hand, kinetic behavior of pressure-induced phase transitions is still not notably researched, although many equilibrium phase diagrams and stability relations for varied materials have been found up to very high pressure. To face this challenge, experimental developments are expectant at least in two directions, one is the pressure-drives with precise control and tenability across a broad range of pressures, temperatures and compressions rates, another is the precise time resolved diagnostic probes with high pressure cells.

In fact, kinetic behavior of diamond nucleation and growth from C-H-O systems could be well known through characterization of the recovered samples. The results are suggesting time-dependent conditions of pressures and temperatures. A notional kinetic phase diagram was established by previous experimental and geological data.

In recent years, we made a pressure-jump apparatus with the compression rate from several GPa/s to 0.5GPa/ms within 10GPa, such rates range lies in the gap between conventional static and dynamic time scales at high pressure experiments. By using it, some novel metastable phases (e.g., amorphous) were solidified through the rapidly compressing their melts, including alloys, non-metallic elements and polymers. In comparison to characterization of the recovered phases and their experimental conditions, dependence of phase transition on the compression rates could be clearly showed, and also in considering of heating or cooling rates at high pressure the kinetic phase diagrams with three factors (pressure, temperature and time) could be established.

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Titanium, Zirconium and Hafnium at high pressures and temperatures

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We measured the volumes of titanium, zirconium and hafnium at several pressures up to 65 GPa and at temperatures between 300 to 2000 K using a resistively heated and laser heated diamond anvil cell (DAC) with synchrotron X-Ray diffraction at the Advanced Photon Source and Cornell High Energy Synchrotron Source. The measured data allows us to determine the P-V-T equation of state (EOS) and improved the experimental constraint on the high P-T phase boundaries between high pressure and high temperature phases in these metals. High pressure phase diagrams, isothermal room temperature bulk modulus and its pressure derivatives as well as thermal expansion at high pressure for Ti, Zr and Hf have been re-assessed. Interestingly, for hafnium, the previously described phase transition[1] from hcp (α) to simple hexagonal (ω) phase at 38 GPa at room temperature was not observed even up to 51GPa [2]. The ω phase in hafnium was only observed at elevated temperatures. Thermodynamic equations of state based on the Gibbs free energy formulation at high pressure were derived for titanium, zirconium and hafnium.

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Equation of state of synthetic garnet solid solution along Almandine-Pyrope join

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Abstract:

The Pressure-Volume relations of three synthetic garnet samples along Almandine-Pyrope join, $Alm_{80}Py_{20}$, $Alm_{50}Py_{50}$, and $Alm_{20}Py_{80}$ were measured at ambient temperature in diamond anvil cell using in situ synchrotron X-ray diffraction at the COMPRES beamline (X17C) of NSLS. The obtained P-V data were fitted to the third order Birch–Murnaghan equation of state (EOS). The ambient cell volumes V_0 of the three samples were measured to be 1511(1) Å^3, 1513 (2) Å^3, and 1529(1) Å^3 respectively. With fixed pressure derivative of the isothermal bulk modulus K'_{T0} at 4, isothermal bulk moduli K_{T0} of the three samples were determined to be 173(4), 175(4), and 181(3) GPa respectively. These results show that the bulk modulus of garnet along the Alm-Py join is proportional to the mole fraction of almandine. While there was no bulk modulus data measured on synthetic garnet sample along Alm-Py join (references showed in fig.1 were measured from natural samples, expect Zhang's end members), these data can be used to establish a relationship of compositional dependence of bulk modulus in the solid solution system as shown in fig.1 and applied to model the composition of the Earth mantle.

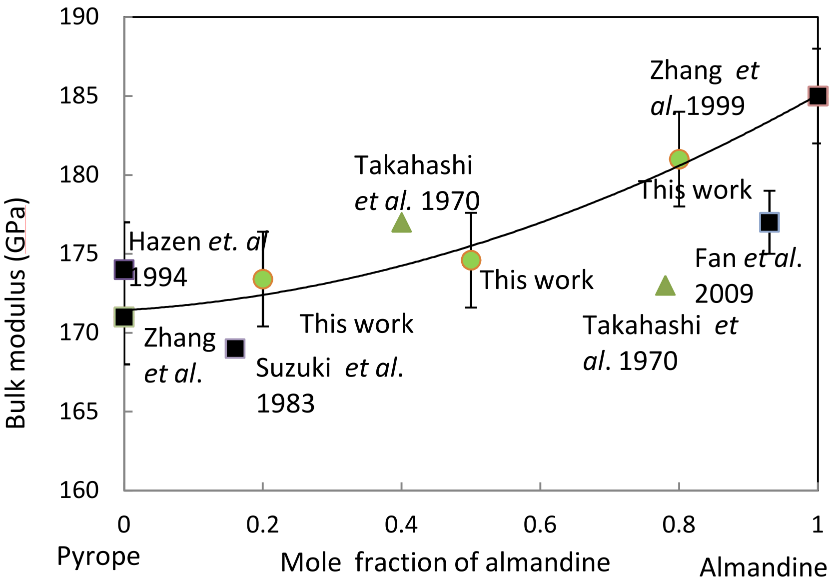

Fig.1 Bulk modulus almandine-pyrope joint

Effect of hydrogen on melting temperature of iron at high pressure and hydrogen partitioning between FeHx and hydrous mantle minerals

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Earth's inner and outer core is approximately 2.5-5% and 5-10% less dense than pure iron, respectively. Hydrogen is considered to be one of the light elements which cause the density deficit of Earth's core. Fukai (1984) suggested that hydrogen can dissolve into metallic iron under high pressure, thus hydrogen is possibly contained in Earth's core. In order to discuss the possibility of hydrogen existence in Earth's core and process of core formation, it is important to investigate hydrogen partitioning between metallic iron (FeHx) and silicates. In this study, we performed in-situ X-ray experiments at 15 and 20GPa using single crystal diamond capsule and the effect of hydrogen to melting temperature of pure iron and hydrogen partitioning between metal and hydrous mantle minerals were investigated. We used powder mixtures of metallic iron and hydrous silicates which contain up to 4.5 wt.% H_2O as starting materials. Amounts of hydrogen (x) in FeHx were estimated from volume expansion of lattice volume by in-situ X-ray diffraction pattern using multi anvil apparatus SPEED-Mk2 installed at BL04B1 in SPring-8 synchrotron, Japan. In these results, hydrogenations of iron coexisting with wadsleyite, ringwoodite, and super hydrous phase B were observed above 1273K. The amounts of hydrogen x increased with temperature to solidus temperature and decreased along with solidus line to liquidus temperature. Increase of x with temperature can be explained by decomposition of hydrous phase and the decrease of x at further higher temperatures can be explained by coexistence of hydrogen-rich FeHx melt above solidus temperatures. In some runs at which silicates contain less than 2 wt.% H_2O, super hydrous phase B was not observed at low temperature and the x values were unchanged at sub-solidus temperature. Above solidus temperature, x values decreased with temperature and a broad peak was appeared, indicating that FeHx solid and FeHx melt coexisted. The solidus temperatures (ex. 1723K at 15GPa and x=0.6,) were much lower than the melting temperature of pure iron (ex. ~2200K at 15GPa). Coexisting wadsleyite and ringwoodite in recovered samples contained very low water (judging from nearly 100% total concentrations of major elements by EPMA), We are now analyzing water contents in wadsleyite and ringwoodite using FT-IR in order to discuss hydrogen partitioning between iron and hydrous silicates. Our observations are generally consistent with pioneering study by Shibazaki et al., (2009) who determined hydrogen partitioning at 1273K at 15 and 20GPa. Our results indicate that most of water in the Earth building material should have dissolved in the core rather than remained in the Earth's mantle. If Earth's outer core contained substantial amount of hydrogen, its melting temperature should be much lower than current estimate.

The water storage capacities and the effect of water on the high pressure phase transformation boundaries of mantle minerals

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Water is the most important volatile component in the Earth, and affects the physicochemical properties of mantle minerals, e.g. density, elastic property, electrical conductivity, thermal conductivity, rheological property, melting temperature, melt composition, element partitioning, etc. So many high pressure experiments have been conducted so far to determine the effect of water on mantle minerals.

In our laboratory, we have been clarifying the effect of water on mantle minerals, especially the water storage capacity in nominally anhydrous mantle minerals, the stability region, the effect of water on the high pressure phase transformation, the water partitioning, the melting temperature, the composition of hydrous magma, etc.

The water storage capacities of mantle minerals are supposed to be significantly coupled with Al by a substitution with Mg^{2+}, Si^{4+} or $Mg^{2+} + Si^{4+}$, because Al^{3+} is the trivalent cation, and H^{+} is the monovalent cation. To clarify the degree of the substitution, the water contents and the chemical compositions of Al-bearing minerals in the mantle transition zone and the lower mantle were determined in the Al-bearing systems with H_2O. In addition, to clarify the effect of water on the high pressure phase transformation boundaries, the high pressure experiment on pyrolite (lherzolite), harzburgite and MORB with H_2O systems have been conducted. We will introduce the recent results on the effect of water on mantle minerals.

A new 6000-ton Kawai-type multianvil apparatus and its application to synthesis of ultrahard nano-polycrystalline diamond and other novel materials

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A Kawai-type multianvil apparatus (KMA) with a ram capacity of 6000 tonf, named as BOTCHAN-6000, was constructed in 2009 at the Geodynamics Research Center of Ehime University. A pair of guide blocks with split-cylinder type first-stage anvils was adopted, where the assemblages of the second-stage tungsten carbide anvils with edge lengths of either 52, 65, or 75 mm can be accommodated by replacing the hardened steel spacers with adequate dimensions. The initial target of producing pressures up to 15 GPa and temperatures to 2600K to synthesize consolidated ultrahard nano-polycrystalline diamond (NPD) has been achieved, and the NPD rods with dimensions up to 1 cm in both diameter and length can now successfully be produced on a routine basis, under the press loads of up to about 5000 tons. We have tested various types of tungsten carbide anvils for stable generation of such pressures and temperatures with minimum anvil damages, and found some of them can be repeatedly used for more than 20 times under such severe P-T conditions. The BOTCHAN has mainly been used for synthesis of the NPD rods with 7-10 mm dimensions for applications to anvils for new classes of high-pressure apparatus. Synthesis of other consolidated nano-micro polycrystalline materials has also been successfully made using the BOTCHAN, which should be a potential tool to produce novel ultrahard materials and high-pressure minerals for some industrial and scientific applications.

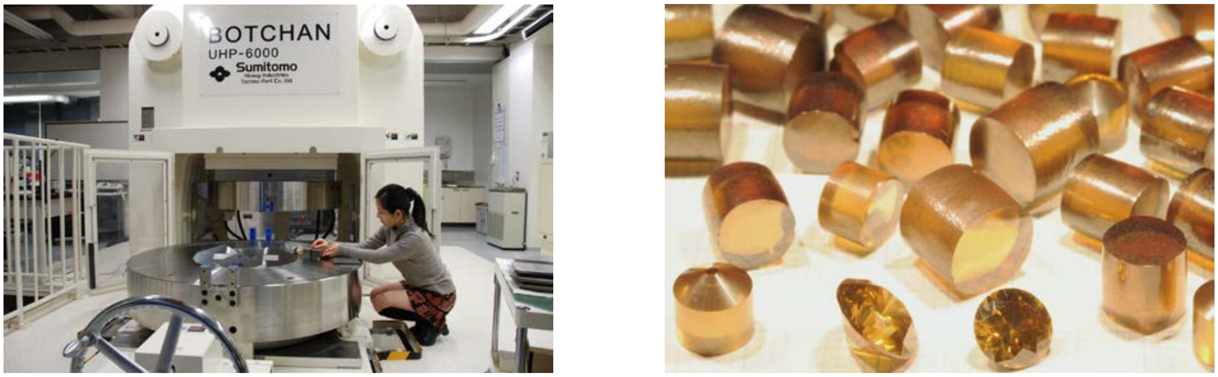

Fig.1. BOTCHAN-6000 (left) and some examples of the NPD samples with various shapes.

High pressure study of transition metal monoxides using the Kawai-type apparatus with sintered diamond cubes

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By adopting sintered diamond (SD) cubes as the second stage anvils, the maximum attainable pressure of the Kawai-type multi anvil have largely been extended and recently reaching 100 GPa without substantial reduction of the specimen volume [1]. Based on the innovation of the experimental technology, we have studied structure and electrical resistance of MnO and CoO at 300 K by means of in situ X-ray diffraction using synchrotron radiation at SPring-8.

Reagent grade of MnO and CoO powders were used as the starting materials. Both were mixed with 5% (in weight) of Au powder. The powder sample was put into the central part of a small octahedron of MgO+5%Cr_2O_3 and was sandwiched by Re electrodes. The sample configuration was similar to those for study of high P-T phase equilibria [1]. The Kawai-cell of the SD cubes was squeezed in the DIA type press SPEED mkII at the beam port BL04B1. Energy dispersive method was employed using polychromatic X-ray. Pressure was determined from the measured volume of Au based on the Matsui et al. [2].

MnO: Onset of rhombohedral distortion of the B1 type MnO observed at ca. 35 GPa in which the hexagonal c/a of the rhombic cell was less than 2.45 (the value corresponding to the B1 structure) and decreased to 2.27 at 72 GPa. The results are in accord with the previous work using the DAC [3]. Measured electrical resistance decreased from the 10^7 Ω order at 10 GPa to ca. ca. 2x10^6 Ω at 70 GPa.

CoO: The rhombohedral distortion was also recognized at pressures higher than 40 GPa. However, onset of the distortion was not clear, because the distortion was quite small compared MnO. The direction of distortion was opposite to that in MnO: i.e., the c/a increased with pressure reaching 2.51 at 88 GPa. These observation is consistent with Guo et al. [4] but not with Noguchi et al. [5]. The transition at around 80 GPa shown by the preceding works [4, 5] was not observed in the present study. The electrical resistance which started at order of 10^7 Ω demonstrated very conspicuous changes with pressure; the minimum of ~10^4 Ω at 15 GPa and the maximum of ~10^6 Ω at 49 GPa, and then decrease gradually to few tens ohms at 88 GPa. The feature in change of electrical resistance with pressure is in sharp contrast with Atou et al. [6].

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Phase Transition of Potassium Azide Under High Pressure

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Alkali azides are the stable members among metal azides. Their insensitivity towards impact makes them suitable subjects for high pressure studies. Previous studies disclosed pressure-induced phase transitions of sodium azide and cesium azide at low pressures (<1 GPa). A high pressure study of potassium azide (KN_3) would be useful to probe new phases, while at the same time providing valuable data which could greatly benefit theoretical studies of such a model compound with symmetrical cations and linear molecular anions. Additionally, such a study would help further the understanding of forming polymeric nitrogen with high pressure treatments. Thus, we studied KN_3 by in-situ synchrotron powder X-ray diffraction (XRD) and Raman spectroscopy using a diamond anvil cell at room temperature up to 55.0 GPa. In the ambient phase, an anisotropic compressibility dominated by repulsions between azide anions was observed. The bulk modulus (18.7 GPa) and mode Grüneisen parameters were also calculated. A pressure-induced phase transition was identified at 15.5 GPa and 13.6 GPa through the XRD and Raman data, respectively. In the Raman spectrum, the onset of the phase transition was characterized by the hardening of a previously soft lattice mode. Compression beyond the phase transition onset pressure induced a symmetry reduction manifested by the splitting of the librational modes, the development of infrared active vibrational modes (loss of inversion center), and the appearance of other new modes in the lattice mode region. The high-pressure phase, with azide ions still in a molecular state, was preserved down to 1.2 GPa.

In-situ measurements of sound velocity of Fe-S, Fe-Si, and pure Fe liquids at high pressures

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Liquid Fe-light element alloys are likely present in the Earth's outer core and the cores of other terrestrial planets such as the Moon, Mercury, and Mars, suggested by geophysical and geochemical observations. In order to determine the abundances of light elements and their effects on the structure, dynamics, and evolution of planetary cores, it is crucial to determine the equation of state for Fe-light element alloying liquids under core conditions. However, the equation of state for liquid Fe-light element alloys is poorly constrained at planetary core pressures due to the scarcity of density data and the absence of sound velocity data for these liquids at high pressures. As a result, density and sound velocity data on solid Fe alloys are often used in the literature to compare with seismological observations by making rough corrections for the volume of melting. In this study, we have developed techniques to directly measure the ultrasonic sound velocities in Fe-light element alloying liquids at high pressures using both a multianvil apparatus and a Paris-Edinburgh cell. The sound velocity was determined by measuring the travel time difference between the sample echo and the buffer rod echo using a waveform generator and a digital oscilloscope and by measuring the sample thickness using X-ray radiographic images. Using this technique, we have successfully obtained sound velocities for three Fe-S liquid compositions (Fe-10wt%S, Fe-20wt%S, and Fe-27wt%S), two Fe-Si liquid compositions (Fe-17wt%Si and Fe-25wt%Si), and pure Fe liquid at high pressure and temperature conditions up to 8 GPa and 2073 K. Results show that the presence of sulfur significantly reduces the sound velocity of liquid Fe, but the presence of silicon increases the sound velocity of liquid Fe. The sound velocity data can provide tight constraints on the bulk modulus and hence the equation of state of Fe-light element liquids at high pressures. In addition, these data can be compared to geophysical observations such as the lunar seismic data to provide constraints on the composition and structure of planetary cores.

Single crystal elasticity of natural Garnet at high pressures and room temperature by Brillouin scattering

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Mg-perovskite (Pv) is a key mineral of the lower mantle. There are many studies on elasticities of Mg-Pv (e.g., Sinogeikin et al., 2004; Murakami et al. 2007a) and a higer pressure phase of Pv, i.e., post perovskite (pPv) (e.g., Murakami et al., 2007b). Although Mg end member of perovskite has been studied, Fe bearing perovskite has not been studied well. In adittion, Al is also an impotant element in the lower mantle. Therefore, Fe-bearing natural pyrope was investigated in this study to discuss Fe and Al effects on elasticities of perovskite.

Brillouin scattering measurements on a natural pyrope and almandine were performed up to 20 GPa and room temperature using a diamond anvil cell. An argon ion laser (514.5 nm) and a six-pass tandem Fabry-Perot interferometer were used for the Brillouin scattering measurements. The chemical compositions of the natural pyrope and almandine were $(Mg_{0.943}Fe_{0.047}Ca_{0.013})_3Al_{2.002}Si_{2.993}O_{12}$ and $(Mg_{0.302}Fe_{0.498}Ca_{0.185})_3Al_{1.976}Si_{2.992}O_{12}$, respectively. The densities at ambient pressure were determined by X-ray diffraction. Pressures were determined based on ruby fluorescence (Dewaele et al., 2004). A single crystal of the garnets was loaded into a sample chamber with typical thickness of 50 μm. Pressure media were NaCl and MEW for pyrope and almandine respectively.

Elastic moduli of pyrope and almandine were determined at room pressure and temperature. The moduli are 277.4 GPa of C_{11}, 99.6 GPa of C_{12}, and 86.6 GPa of C_{44} for pyrope and those are 295.4 GPa of C_{11}, 106.9 GPa of C_{12}, and 90.1 GPa of C_{44} for almandine. We will discuss the elasticity of garnets up to higher pressures.

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Self-diffusion coefficients of the mantle minerals

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The mantle is under high temperature conditions, and the strain rate in it is low. Hence the rate-limiting processes of both dislocation and diffusion creeps are expected to be diffusion of the constituent elements. In this study, we measured Si and Mg self-diffusion coefficients (D_{Si} and D_{Mg}) of the important minerals to understand mantle dynamics, namely, stishovite (*St*), $MgSiO_3$ perovskite (*MPv*) and forsterite (*Fo*).

Stishovite. D_{Si} of single crystal *St* in the [110] and [001] directions was measured at 1700-2100 K and 14 GPa. D_{Si} of *St* ranges from 10^{-22} to $10^{-19.5}$ m^2/s in these conditions, which is the lowest D_{Si} among those of the minerals so far measured. The activation enthalpy (H_a) is small, 320 kJ/mol. D_{Si} in the [110] direction is slightly (40 %) faster than in the [001] direction.

$MgSiO_3$ Perovskite. D_{Si} and D_{Mg} of single crystal *MPv* were measured at 1600-2100 K and 25 GPa. The present results agree to those obtained using polycrystalline samples [Yamazaki *et al.*, 2000; Dobson *et al.*, 2008], suggesting the reliability of these measurements. D_{Si} and D_{Mg} are almost identical, which implies Mg and Si cations diffuse together by forming defect clusters. The low D_{Mg} is probably because of Mg occupancy in the position of closed packed oxygen and no interstitial site available for hopping in the *MPv* structure. H_a is 310 kJ/mol and almost identical to that of *St*. The 6-coordinated Si seems to have smaller H_a than 4-coordinated Si. The D_{Si} in *MPv* is by 2 orders of magnitude higher than *St*, maybe because of the largely distorted O packing in *MPv*.

Forsterite: D_{Si} of synthetic single crystal *Fo* was measured at 0-13 GPa and 1600-1800 K under dry conditions. The absolute values obtained in this study are about 2 orders of magnitude higher than those by Jaoul *et al*. [1981]. The present high values of D_{Si} well explain the results of deformation experiment. The activation volume is found to be very small, 2 cm^3/mol. H_a is 410 kJ/mol. This H_a is smaller than those obtained by the deformation experiments (about 500 kJ/mol) D_{Si} was also measured as a function of water content (C_{H_2O}) at 8 GPa and 1600 K. It is found that the C_{H_2O} exponents of D_{Si} is 1/3, respectively, which is much smaller than that given by deformation experiments, 1.2 [Hirth & Kohlstedt, 2004]. Thus, the *T*, *P* and C_{H_2O} dependences of self-diffusion coefficients are all small. Viscosity should not vary largely in the upper mantle.

Integration of elastic wave velocity and viscosity measurement with liquid structure measurement in a Paris-Edinburgh press at the sector 16-BM-B, HPCAT, APS

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Correlation between structure and physical properties is fundamental for understanding the behavior of materials. For crystalline materials, both structure and physical properties have been widely studied at high pressure and high temperature conditions by integrating high-pressure apparatus (e.g. large volume press, diamond anvil cell) with various measurement techniques. In contrast, those of liquids and amorphous solids have been much less studied due to experimental difficulties. Some efforts have been made to investigate structure, physical properties such as density, viscosity, and elastic wave velocities. However, these results were often based on individual techniques, and the discussions were made by comparisons with results from other techniques. Integration of structure and physical property measurements of liquids and amorphous solids should promote a comprehensive understanding of the behavior of amorphous materials. The sector 16-BM-B, HPCAT at the APS is capable of amorphous and liquid structure measurement by using multi-angle energy dispersive x-ray diffraction measurement at high pressure and high temperature conditions in a Paris-Edinburgh cell. In addition to the structure measurement capability, we recently developed elastic wave velocity and viscosity measurement. Elastic wave velocity measurement was conducted by ultrasonic technique in conjunction with x-ray radiography measurement. The first simultaneous structure and elastic wave velocity measurement was carried out for SiO_2 glass at pressures up to 6.8 GPa at around 500 °C. The results indicated a strong correlation between the intermediate range order variations and compressional wave velocity or Poisson's ratio, but a complicated behavior for shear wave velocity. In contrast, viscosity was measured by falling sphere viscometer using x-ray radiography measurement. The first viscosity measurement test on albite showed a consistent result with previous studies. These setups open a new way to investigate the link between microscopic structure and macroscopic elastic wave velocity or viscosity at high pressure and high temperature conditions. We acknowledge the supports of COMPRES on facility development, of GSECARS for the Paris-Edinburgh press system, and of HPCAT for elastic wave velocity and viscosity measurement developments.

Recent topics using a Kawai-type large volume press at SPring-8/BL04B1

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SPring-8/BL04B1, white X-ray beamline from a bending magnet is designed to conduct research on the structure and properties of minerals in the Earth's interior using the Kawai type large volume press (SPEED-1500 and SPEED-type D). Especially SPEED-type D is the largest deformation apparatus (D-DIA), having the hydraulic control unit of upper and lower 1st-stage anvils aside from main ram, and then, high horizontal to vertical ratio of a sample chamber is maintained up to high press load. Deformation experiments using SPEED-type D have been carried out using a multianvil 6-6 cell assembly (MA6-6). In this presentation, we present several experimental techniques and results at BL04B1. For example, we have developed newly-designed nano-polycrystalline diamond (NPD) anvils for 2nd-stage anvils of MA 6-6 system. The new anvil is composed three parts – NPD anvil top, stainless anvil guide and sintered diamond backing base plate. As optimization of cell assembly, we have achieved the pressure as high as ~ 30 GPa. Further, We set-up an ultrasonic measurement system, so that simultaneous ultrasonic and in situ X-ray measurements can be conducted at ~27 GPa and ~1873 K, equivalent to the conditions of the lower mantle region. Moreover, we have developed the new anvil material, diamond/SiC composite, which provides high X-ray transparency. Combination of diamond/SiC composite anvils in the Kawai-type LVP and synchrotron X-rays enables to obtain entire Debye-Scherrer rings and expand the radiographic image through the anvils. We will give details of our recent results in the presentation.

Single crystal diffraction tools for Earth materials at the ALS

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The need for accurate experimental structural characterization of Earth materials lies at the center for most problems in mineral physics. Structure refinement against powder diffraction data is in many cases the method of choice, despite its reduced content of information compared to single crystal data. This is thanks to the high tolerance of powder diffraction experiments to restricted sample mobility and/or access, both of which is often encountered when dealing with samples under non-ambient conditions. Powder diffraction, however, reaches the limit of its applicability when dealing with very small sample volumes, the need for accurate interatomic distances or the need for high spatial resolution. We believe that in such cases, a return to one of the various flavors of single crystal diffraction is beneficial.

The Advanced Light Source (ALS) at Lawrence Berkeley National Laboratory (LBNL) is a relatively low energy (1.9 GeV) synchrotron operating at 500 mA in top-off mode. Combining this low energy ring with 5 Tesla super conducting bending magnets delivers an X-ray spectrum which is ideal in flux and spectral width for single crystal X-ray diffraction techniques. In addition the brightness of the ALS will be increased by x3 in the next ALS shutdown (Jan-March 2013) with the addition of new hexa-poles in the ring lattice.

With beamlines 11.3.1, 12.2.2 and 12.3.2, the ALS is now offering three complementary single crystal diffraction tools to the mineral physics community:

11.3.1 (optimal energy ~ 17 keV, spot size ~ 200 um) is one of the few dedicated chemical crystallography synchrotron beamlines in the world. Besides the ability to solve difficult structures from difficult crystals, it is of special interest for projects involving non ambient conditions thanks to its non-ambient gas environment capabilities plus the integration of a diamond anvil cell, optimized for moderate pressure ranges (<10 GPa). The planned move of this beamline onto a superbend source will expand its spectral range up to 25 keV, which expands its high-pressure applicability.

12.2.2 (energy range ~ 5 – 35 keV spot size ~ 10 um) is a dedicated high-pressure beamline with an emphasis on resistive and laser-heated powder diffraction. Various recent upgrades now allow continuous monochromator energy scans, which enables energy dispersive single-crystal diffraction on a stationary sample. Thus diffraction from laser-heated single crystal samples can be viewed as practical.

12.3.2 (energy range ~ 5 – 25 keV, spot size ~ 1 um) is a dedicated Laue microdiffraction beamline. White beam Laue single crystal diffraction allows the recording of a significant portion of reciprocal space within a single exposure of a few seconds. This combined with a (sub)-micron beam allows the extraction of information on elastic and plastic deformation as well as structural details with high spatial resolution. This can be used e.g. to probe specific regions within the restricted volume of a pressure cell.

Single-crystal elasticity and structural behavior of $MgSiO_3$ perovskite at pressure of the lower mantle

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Seismic wave velocities determined as a function of depth in the Earth's interior impose strong constraints on the chemical and thermal state of the mantle. Such profiles can still be interpreted, however, with a range of different geophysical models. Precise measurements of the pressure dependence of acoustic wave velocities of mantle minerals are, therefore, essential in order to assess which model is appropriate for the Earth's lower mantle. $MgSiO_3$ perovskite has been the subject of several studies, since it is likely the primary lower mantle mineral phase. Several high-pressure experiments have been performed to constrain both the density and bulk modulus of both pure $MgSiO_3$ perovskite and perovskite containing iron and aluminum in different proportions. However, knowledge of its full elastic constant tensor is limited either to room pressure conditions, due to the challenge of synthesizing and recovering high-quality single-crystals of this high-pressure mineral, or to first-principle calculations.

We have collected Brillouin scattering spectra from single-crystals of $MgSiO_3$ perovskite at different pressures in the diamond anvil cell employing He as a pressure transmitting medium. Simultaneous density measurements were made using X-ray diffraction. The spectra are used to obtain the full set of elastic constants of $MgSiO_3$ perovskite. The elastic behavior of perovskite can be understood through a comparison with its structural variation with pressure, obtained from single-crystal structural refinements that have been carried out up to 90 GPa.

Fe_4O_5 a new high-pressure iron oxide

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Iron oxides have broad geophysical and geochemical relevance due to their abundance, electronic properties and role in redox equilibria. While investigating the stability and decomposition products of siderite ($FeCO_3$) at conditions of the Earth's mantle we obtained, at 10 GPa and about 1800 K, a single crystal of unknown phase. Structural solution and refinement suggest the phase to be a new iron oxide of formula Fe_4O_5. The phase has orthorhombic symmetry where iron is 6-coordinated in layers of trigonal prisms and edge-sharing octahedra.

We performed first-principle calculations in order to examine the viability of Fe_4O_5 in terms of its energetics and stability under pressure. The calculated lattice constants, equation of states and atomic coordinates are in excellent agreement with experimental data, confirming the synthesis of Fe_4O_5. To examine the relative stability of Fe_4O_5 with respect to its possible breakdown products (FeO and Fe_3O_4 in the ambient- and high-pressure structures) enthalpies were calculated as a function of pressure. We found that Fe_4O_5 becomes energetically more favorable than the breakdown products above ~ 8 GPa, providing complementary evidence that the synthesis of Fe_4O_5 is energetically plausible at high pressure. Calculations show that Fe_4O_5 is ferrimagnetic at 11 GPa.

With the aim of confirming the stability in a pure system, we performed several HP-HT synthesis starting from mixtures of pure $Fe+Fe_3O_4$. Fe_4O_5 was synthesized in the range 10-20 GPa readily upon heating in the range 1500-2500K. Upon decompression and further heating we observed the decomposition of Fe_4O_5 at ~ 5GPa and 1800K, to wüstite and magnetite. The new oxide can be retrieved to ambient conditions if it is pressure quenched at ambient temperature.

The finding has a broad significance, spanning planetary science, physics and materials science. A recoverable, magnetic iron oxide with multiple non-equivalent short Fe-Fe distances is likely to show a complex electronic structure and might find technological applications. It is difficult to predict the occurrence of the new iron oxide in the deep Earth's interior as many factors, including P, T, oxygen fugacity, composition and element partitioning among coexisting phases, contribute to the stability of a mineral in a rock. Fe_4O_5, being a relatively reduced phase, is a plausible accessory phase of the deep interior, particularly considering the high heterogeneity of the upper mantle.

Cell assemblies for reproducible multi-anvil experiments (the COMPRES assemblies)

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The multi-anvil high pressure technique is an important tool in high pressure mineralogy and petrology, as well as chemical synthesis, allowing the treatment of large (mm-size) samples of minerals, rocks and other materials at pressures of a few GPa to over 25 GPa and simultaneous uniform temperatures up to 2500 °C and higher. A series of cell assemblies specially designed and implemented for interlaboratory use is described here. In terms of the size of the octahedral pressure medium and the anvil truncation size, the five sizes of assemblies developed here are an 8/3, 10/5, 14/8, 18/12 and 25/15 assembly. As of this writing, these assemblies are in widespread use at many laboratories. The details of design, construction, and materials developed or used for the assemblies are presented here.

History of High-Pressure Mineral Physics Seminars: 1976-2007

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Under the auspices of the U.S.-Japan Cooperative Science Program between the National Science Foundation and the Japan Society for the Promotion of Science, the first U.S.-Japan joint seminar on High Pressure Research Applications in Geophysics was held in Honolulu, Hawaii, in July 1976. Originated as bilateral U.S.-Japan seminars on high-pressure research applications in geophysics by Murli Manghnani and Syun-iti Akimoto, this series continued at 5-year intervals under those auspices from 1976 to 1996 with venues alternating between Hawaii and Japan [1981 in Hakone, 1986 in Turtle Bay, 1991 in Ise, and 1996 in Maui],

As a result of growing international interest in high-pressure mineral physics, the seminar series was re-envisioned with a broader focus than the original US-Japan bilateral series. The first of the new meetings entitled “High Pressure Mineral Physics Seminars” [HPMP-6] took place in Verbania, Italy on August 26-31, 2002. More than 90 scientists from Asia, Australia, the Americas and Europe attended. HPMPS-7 took place in Matsushima, Japan on May 8-12, 2007 and was attended by 134 scientists from throughout the world.

Each of these seminars has been followed by a published volume of scientific and technological papers; many of the papers in these volumes have not appeared elsewhere. Consequently, these volumes have served as archival repositories of progress and development in high-pressure mineral physics for the past 35 years, and have been read by more than two generations of young and senior scientists.

Furthermore, this seminar series has an excellent record of fostering the professional careers of young scientists in the world of mineral physics; see evidence in the group photos of each seminar in the poster.

Multi-anvil, high-pressure apparatus: A half century of development and progress

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In 1958, Tracy Hall invented the first multi-anvil, high-pressure apparatus: a tetrahedral anvil device capable of attaining simultaneous pressures of 10 GPa and temperatures above 3000K. In the past half century, multi-anvil apparatus have evolved progressively and can now reach pressures close to 100 GPa at high temperatures. Many of these high-pressure devices have been utilized in conjunction with in situ X-ray diffraction, especially with the advent of synchrotron radiation facilities in the early 1980s. There are a variety of technological approaches to generating high pressures in the laboratory, primarily motivated by the desire to study the behavior of materials at elevated pressures and temperatures; many of these approaches have been developed in the Earth science community due to the desire to replicate in the laboratory the P-T conditions of the Earth's deep interior. In addition to the dynamic techniques of shock-wave experiments, there have been two complimentary static techniques to achieve these goals: the diamond-anvil cell and the multi-anvil apparatus. Although these two static techniques have occasionally been viewed as competitive, they are both useful and very complimentary. The purpose of this paper is to review the development and progress in multi-anvil apparatus.

A Career Path for African-American Students from HBCUs to National Laboratories

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The Department of Geosciences at Stony Brook University has established a new M.S. program in Geosciences Instrumentation, primarily for African-American Students from Historically Black Colleges and Universities. This new program is directed to building on the evolving relationship between professors from Historical Black Colleges and Universities [HBCUs] and the National Synchrotron Light Source [NSLS] of the Brookhaven National Laboratory [BNL]. An outcome of this relationship has been the creation of an Interdisciplinary Consortium for Research and Educational Access in Science and Engineering [INCREASE], an organization to promote research in HBCUs and other minority-serving institutions [MSIs], involving utilization of national user facilities, such as the NSLS at BNL.

The goal of this new initiative is to recruit undergraduate science and engineering students from underrepresented groups into the graduate program in the Department of Geosciences at Stony Brook University, to educate them via formal courses and research projects to the M. S. degree in geosciences, and to position them for employment as science associates in national user facilities of the U. S. Department of Energy [DOE], such as the National Synchrotron Light Source [NSLS] at the Brookhaven National Laboratory [BNL]

This 2-year MS program in Geosciences will include both formal courses in the Departments of Geosciences, Chemistry, and Physics & Astronomy and internship research at the X-ray and ultraviolet beamlines operated at the NSLS of BNL by COMPRES [Consortium for Materials Properties for Research in Earth Sciences]. Full-time students in this program will receive financial stipends of $28,000 per year as well as tuition and fees and health care coverage. Support provided by the National Science Foundation [NSF], Brookhaven National Laboratory and Stony Brook University.

This program will be overseen by Robert Liebermann, Professor of Geosciences at Stony Brook, Lars Ehm, Research Assistant Professor in the Mineral Physics Institute [MPI] at Stony Brook, and Gabriel Gwanmesia, Professor in the Department of Physics and Pre-Engineering at Delaware State University.

As of April 2012, three students have been recruited into this M. S. program: Ashley Thompson from Delaware State University enrolled in January 2012 and is now working on her research project with Lars Ehm at the MPI and NSLS. Melissa Sims from the University of South Carolina and Adaire Heady from Delaware State University will enroll in September 2012.

Students interested in this program should contact Robert Liebermann [Robert.Liebermann@stonybrook.edu] and Yvonne Barbour, Graduate Secretary in the Department of Geosciences [betty.barbour@stonybrook.edu] for instructions on the procedures for applying to the Graduate School at Stony Brook.

Sound Velocities of Iron Alloys in the Earth's Core

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Enigmatic properties of the Earth's inner core have recently been discovered, including differential super-rotation, seismic anisotropies, and fine-scale seismic heterogeneities. Deciphering these observations requires solid knowledge about the composition of the Earth's inner core and therefore the thermoelastic properties of candidate iron alloys. Compressional wave velocity-density relations of candidate iron alloys at relevant pressure-temperature conditions of the Earth's core are critically needed to evaluate the composition, seismic signatures, and geodynamics of the planet's remotest region. Specifically, comparison between seismic velocity-density profiles of the core and candidate iron alloys provides first-order information on the amount and type of potential light elements - including H, C, O, Si, and/or S - needed to compensate the density deficit of the core.

To address this issue, here we have surveyed and analyzed the literature results in conjunction with newly measured compressional wave velocity-density results of hexagonal closest-packed (hcp) Fe and Fe-Si alloys using *in situ* high-energy resolution inelastic x-ray scattering and x-ray diffraction in an externally-heated diamond cell up to megabar pressures and high temperatures at Sector 3 of the Advanced Photon Source. The nature of the Fe-Si alloy, where Si is readily soluble in Fe represents an ideal solid-solution case to better understand the light-element alloying effects. The obtained phonon dispersion curves were fitted to a sine function within the framework of the Born–von Karman lattice-dynamics theory to derive the compressional wave velocity of iron alloys, whereas in situ X-ray diffraction patterns allowed direct measurements of the densities of the samples. These results are combined with a thermodynamic modeling and seismic models of the inner core in order to decipher high P-T effects on our current understanding on the amount of light element(s) in the core. These new datasets, together with literature results, allow us to build a reliable velocity-density model of Fe alloys in order to determine the chemical composition of the core. Our models show that the V_P–ρ profile of Fe with approximately 8 wt.% Si at 6000 K matches well with the seismic models of the inner core. We will also discuss future challenges and research opportunities in the studies of the chemical composition, seismic velocity profiles, and high-pressure techniques under extreme environments of the Earth's core so as to stimulate participating scientists in the COMPRES community to explore this new frontier collaboratively.

Sound Velocity of $Fe_{0.85}Si_{0.15}$ in Earth's inner Core

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The Earth's core is mainly composed of Fe-Ni alloy with a few weight percent candidate light elements (e.g., Si, S, and O). Knowing the elasticity of iron and its alloys is thus critical to understanding the physics and chemistry of the Earth's core. In particular, the alloying effects of light elements on the elasticity of iron are of great importance to establishing satisfactory geophysical and geochemical models of the Earth's core. For example, comparison between compressional wave velocity-density (V_p-ρ) relation of candidate iron-light element alloys and seismic velocity profiles at pressure-temperature (P-T) conditions relevant to the core provides a direct constraint on the composition of the Earth's core. According to previous studies, different light elements can have distinct effects on the elasticity of iron under high P-T. It should be noted that high temperatures can also affect the V_p behavior of iron-light element alloys under the pressures. Thus the experimental V_p-ρ results of iron alloys at simultaneous high P-T conditions are indispensable to establish a more reasonable composition model of the Earth's core. Here we have measured the Vp-ρ relation of Fe-Si alloy under high pressures using in situ high-energy resolution inelastic X-ray scattering (HERIX) and X-ray diffraction in a diamond anvil cell (DAC). Compared with previous V_p studies on iron, these results allow us to derive the alloying effect of silicon on the V_p of hcp-Fe. Our data are also used to critically evaluate seismic and geochemical models of the core, providing new insights into understanding seismic observations and chemical composition of the Earth's inner core.

(C) Description

Stability of the α- and β-Co_2P at high pressure and temperature and the implications for allabogdanite-barringerite transition in planetary cores

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Meteoritic metal phosphide minerals orthorhombic allabogdanite and hexagonal barringerite $(Fe,Ni)_2P$, which can be used as proxy of pressure/temperature conditions inside the meteorite parent body, typically contain around 1 wt. % of cobalt. Presence of Co was suggested to have a potential strong effect on the allabogdanite/barringerite transition. While thermochemical arguments and fast-temperature quench experiments at ambient pressure indicate high-temperature stability of the hexagonal β-Co_2P, recent high-pressure in situ study did not find evidence of formation of the barringerite-like β-phase at 15 GPa and high temperature. We studied the high-pressure and high-temperature behavior of allabogdanite and barringerite structured Co-end members in a laser-heated diamond anvil cell up to 50 GPa and 2200 K, utilizing single-crystal x-ray diffraction approach. The hexagonal phase was successfully synthesized on heating above 2000K at 5 GPa and remained untransformed on further compression without heating up to 50 GPa. Unheated orthorhombic phase also remains untransformed on compression without heating to 42 GPa, indicating strong metastability and large activation barrier of the transformation. Equations of state for both phases, as well as the compression mechanism of the othorhombic α-phase, established from single crystal refinements are in excellent agreement with first principles calculations.

Infrared Diamond Anvil Cell Facility (IR-DAC) at National Synchrotron Light Source

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The study of the effects of pressure on materials is fundamental to understanding a broad range of problems in Earth and planetary sciences, physics, biology, chemistry, materials science, and high-technology. Increasing pressure on materials can radically alter the physical and chemical properties of materials. Tremendous progress and breakthroughs in the development of ultrahigh-pressure diamond-anvil cell techniques have been made in the past several decades. For example, the behavior of materials at pressures comparable to those found near the Earth's inner core (*i.e.*, roughly to >300 GPa) can be studied under sustained conditions with unprecedented accuracy, precision, and sensitivity. Such experiments are crucial for developing a comprehensive understanding of the structure, dynamics, and evolution of planetary interiors. High-pressure spectroscopy provides essential and often unique information about the properties of materials under these conditions. For instance, vibrational infrared (IR) spectroscopy provides detailed information on bonding properties of crystals, glass, and melts, thereby yielding a microscopic description of thermochemical properties. Infrared measurements also provide information on electronic excitations including crystal-field, charge-transfer, excitonic spectra of insulating and semiconducting materials, interband and intraband transitions in metals, and pressure-induced metallization of insulators.

Using synchrotron radiation for infrared studies substantially improves our ability to probe microscopic samples including *in situ* measurements under extreme conditions due to its high brightness, broad-spectrum distribution, and diffraction-limited performance. The development of synchrotron infrared spectroscopy as a whole is fast becoming a leading technique in synchrotron radiation research. The National Synchrotron Light Source (NSLS) has the unique capability to provide synchrotron radiation over a broad range from hard x-ray down to the very far-infrared. The infrared radiation at the VUV ring of the NSLS has world-class performance with up to ~10^4 times the brightness of a conventional thermal (lamp) source. As one of the six infrared beamlines at the NSLS, U2A is the first dedicated high-*P* synchrotron infrared facility in the world, with many unique capabilities compared to high-pressure x-ray beamlines. The high-pressure infrared program at the NSLS has been very successful in terms of beamline development, outreach of user community, and scientific productivity. A number of important scientific and technical challenges are being addressed at the facility, making it a highly attractive complement to x-ray sources for high-pressure studies. Here, we highlight the beamline capabilities and progress on the CO_2 laser heating system.

The NaCl pressure scale from simultaneous sound velocity and density measurements at high temperatures and pressures

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The temperature-pressure-volume (*T-P-V*) equation of state (EOS) of the B1 phase of NaCl developed by Decker(1971) has been widely used as a practical pressure scale at temperatures and pressures up to 1200 K and 25 GPa. The NaCl EOS was called into question, however, by Li et al., who directly determined the pressure by simultaneously measuring the sound velocities and densities of Mg_2SiO_4 wadsleyite (Li et al., 2005) and MgO (Li et al., 2006) at 300 K and high pressures. The samples examined experimentally by Li et al. (wadsleyite or MgO) were surrounded by a mixture of NaCl and boron nitride (BN) (NaCl:BN = 10:1 by weight), and the X-ray diffraction patterns from both the sample and the NaCl were collected to obtain the respective densities simultaneously at a given pressures. The directly determined "absolute" pressure from the wadsleyite sample at 20 GPa and 300 K was 12(1) % (2.3 GPa) higher than the pressure in NaCl predicted by the Decker EOS. The *T-P-V* EOS of NaCl is important, and many high-pressure studies rely on the pressures estimated based on the EOS of NaCl. Thus, direct pressure measurements of NaCl at 300 K and high temperatures are strongly required.

In the present study, the elastic compressional (P) and shear (S) wave velocities in NaCl were measured up to 12 GPa at 300 K, and up to 8 GPa at 473 and 673 K, by combining ultrasonic interferometry, in situ synchrotron X-ray diffraction, and X-ray radiographic techniques in a large-volume Kawai-type multi-anvil apparatus. The simultaneously measured sound velocity and density data at 300 K were used to estimate the 300 K EOS by a least squares fit to the fourth-order Birch-Murnaghan finite strain equation, without pressure data. The adiabatic bulk modulus K_S was converted to the isothermal one K_T using the usual relation. For a fixed isothermal bulk modulus K_{T0} of 23.7 GPa at 0 GPa and 300 K, as determined from previous data, we obtained the first and the second pressure derivatives of K_{T0}, $K_{T0}' = 5.14 \pm 0.05$ and $K_{T0}'' = -0.392 \pm 0.021$ GPa^{-1}, respectively. A high-temperature and high-pressure EOS of NaCl was then developed using the Mie-Grüneisen relation and the Debye thermal model. The simultaneously measured sound velocity and density data at both 473 and 673 K, as well as previously reported volume thermal expansion data of NaCl at 0 GPa were used for fitting in the present analysis, to yield $q = 0.96$, holding the Grüneisen parameter and the Debye temperature, both at 0 GPa and 300 K, fixed at 1.56 and 279 K, respectively, from previous studies. Our EOS model accurately reproduced not only the present measured K_T data at pressures up to 12 GPa and temperatures between 300 and 673 K, but also the previously reported volume thermal expansion and the temperature dependence of K_T, both at 0 GPa. We found that the present and Decker EOS's agree within 1 GPa over the temperature and pressure ranges up to 1200 K and 30 GPa. The new *T-P-V* EOS for NaCl, developed here, provides a pressure-independent primary pressure standard at high temperatures and high pressures up to 1200 K and 25–30 GPa.

Correlation between acoustic emission and mechanoluminescence of rock cores under quasistatic compression

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When a rigid solid undergoes mechanical deformation, locally accumulated strain energy can be released through multiple avenues including acoustic emission (AE) and light emission known as mechanoluminescence (ML). While AE is used extensively for rock evaluation in geophysics, civil engineering, and mining, ML by comparison has received little attention from the geoscience community. ML from stressed and fracturing rock has been observed in mines, earthquakes, and the laboratory, but the underlying mechanism behind ML is poorly understood. Possible candidates include defect movement, creation of charged surfaces during fracturing of piezoelectric grains, and triboluminescence. Observing whether a correlation exists between ML and AE will help determine the source of ML. ML may prove to be a useful tool for studying brittle fracture under laboratory conditions. An apparatus for AE and ML detection of rock cores under quasistatic compression was designed and constructed. Using photomultiplier tubes and piezoelectric transducers, AE and ML events were spatially and temporally observed and correlated. AE and ML in Berea sandstone were measured, and a strong correlation was observed. Unexpectedly, preliminary experiments showed the ML events occurred many microseconds after the AE events.

Dislocation textures and aluminum diffusion in majoritic garnet. ATEM study.

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Plastic deformation by dislocations and atomic diffusion by vacancies of minerals at high pressures are important for the rheology of the Earth's mantle. Because those processes are controlled by moving of two agents (line and point defects) in deformations at high temperature, the post-mortem examination by analytical transmission electron microscope is indispensable for evaluating those agents (carriers). Majoritic garnet (MajGt) and magnesium silicate perovskite (MgPv) are major constituents in the mantle transition zone and the lower mantle, respectively. Diffusivity differences in these mantle minerals are very important to understand the changes of the nature of chemical heterogeneity, viscosity through those creep raw, and other various transport properties across the upper and lower mantle boundary.

Here I report an Al = Si + Mg interdiffusion in MajGt, in comparison to Miyajima et al. (2006). The diffusion couples using a multi-anvil press are pre-synthetic $Mg_3Al_2Si_3O_{12}$ pyrope and majoritic garnet ($Maj_{76}Prp_{26}$). The annealing condition is at 18.5 GPa and 1750 ºC for 300 min, corresponding to a MajGt-single phase region in the binary system $MgSiO_3$-Al_2O_3. Diffusion profiles of the recovered sample were examined with scanning electron microscopes and scanning transmission electron microscopes equipped with energy dispersive X-ray spectrometers. Dislocation microtextures were also observed in weak-beam dark-field images using the thickness-contour fringe method (Ishida et al., 1980; Miyajima & Walte, 2009).

MajGt displays <100> and 1/2 <111> free dislocations and subgrain textures consisting of a dislocation array, suggesting that climb of dislocations was relatively easy during diffusion annealing. The obtained Al = Mg + Si interdiffusion coefficient (D_{Al}) of MajGt at 18.5 GPa and 1750 °C is 6.2(4) x 10^{-19} (m^2/s), which is comparable with those of Mg and Si self-diffusion coefficients in MgPv under lower mantle conditions (Xu et al., 2011) and Si diffusions in the other high pressure silicates with 6-coordinated silicon at a homologous temperature. However, the D_{Al} is significantly smaller than ~2 × 10^{-16} (m^2/s) in MgPv and ~5 × 10^{-15} (m^2/s) in MajGt at 25 GPa and 1950 °C (Miyajima et al., 2006). Comparisons with Al diffusion in MgPv are given to highlight the slowest species of Si and the effect of trivalent cations on diffusion rates. Considerations for further diffusion experiments in MajGt and aluminous MgPv are discussed toward the rheology from the transition zone from the lower mantle. I thank the generous support from BGI colleagues for commissioning of this study.

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Melting properties of iron alloys under high pressure

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It is well established that the Earth's liquid outer core is less dense than a pure Fe-Ni liquid alloy. The so-called "core density deficit" is currently estimated around 5-10 wt %[1] and is attributed to the presence of light elements dissolved in an iron-rich liquid alloy. Recent studies have suggested that Mars, Ganymede and even the Moon could have a liquid metallic outer core together with a solid inner core, like the Earth [2, 3]. Hence, it is important to investigate the evolution of the Fe-FeX phase diagram and the physical properties of the liquid iron alloys in respect to pressure, temperature and light element content.

We studied the melting properties of various iron alloys (Fe-S, Fe-Si, Fe-C) using different high pressure tools. Firstly, we investigated the melting properties of several alloys up to megabar pressures: Fe-5%wtNi-15%wtSi; Fe-5%wtNi-10%wtSi; Fe-5%wtNi-12%wtS using in situ laser heated diamond anvil cell[4]. Scrupulous attention in the synthesis and characterization of the starting material is fundamental to accurately control the chemical composition in the laser-heated spot. The appearance of a diffuse signal around 30 nm^{-1} has been used to determine the onset of melting. Furthermore, density values for these liquid alloys have been determined up to 1 Mbar, using diffraction data analysis similar to[5]. Model for S and Si content of the Earth's liquid outer core have been derived from this data set.

Secondly, melting properties of Fe-S, Fe-Si and Fe-C samples were investigated up to 6 GPa and 2200 K by *in situ* X-ray diffraction using the Paris Edinburgh press, thanks to the new multichannel collimator set-up [6] designed on the High Pressure Beamline ID27, ESRF.. After a careful data analysis, structural properties and densities have been extracted for different light element content and different P-T conditions. Such data set could be used to model small planetary cores, such as Ganymede and the Moon.

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Water, iron, redox environment: Effects on the Wadsleyite-Ringwoodite phase transition

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The transition zone of the Earth's upper mantle is characterized by three discontinuities in seismic wave velocity profiles. The one around 520 km depth is assigned to the transformation of wadsleyite ($\alpha-$) to ringwoodite (β-$(Mg,Fe)_2SiO_4$) (e.g.[1]). The exact location, width, and other properties of that discontinuity are affected by a multitude of parameters. The present study specifically focuses on the influence of water, iron content, and redox conditions on the phase transition.

Both wadsleyite and ringwoodite are nominally anhydrous minerals but can incorporate up to 3 wt.% H_2O as hydroxyl in their structures (e.g. [2]). Former studies have shown that there is a shift of the transition of about 2 GPa to higher pressures in the presence of water [3, 4]. The reason for that, however, remained unclarified as the maximum solubility of water in both phases is comparable. Therefore we performed high pressure experiments using a rotating multi-anvil apparatus at 1200°C with variation of Mg-Fe-compositions ($0.10 < x_{Fe} < 0.24$), water-contents ($0 < x_{H2O} < 2$ wt.%) and the redox environment (using different buffers: Fe/FeO (reducing), Re/ReO_2 (oxidizing). EMPA (composition), FTIR spectroscopy (water content), Mössbauer- and EELspectroscopy (Fe^{3+}-content) on all samples provide new insights into the phase relations compared to previous studies. Under hydrous and reducing conditions the wadsleyite-ringwoodite boundary shifts by 0.5 GPa to higher pressures accompanied by a broadening of the region of coexisting wadsleyite and ringwoodite. In contrast, under oxidizing conditions the field of coexisting phases gets narrower and the shift of the phase transition to higher pressures is amplified. Thus, the stability field of wadsleyite is extended to higher pressure most likely due to the higher water and Fe^{3+} content compared to ringwoodite. Based on Mössbauer spectroscopy we infer that Fe^{3+} is incorporated as a spinelloid component.

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High Pressure Deformation and Imaging Techniques in Experimental Geophysics

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Deforrmation processes have extraordinary importance for Geosciences. Mountainbuilding, i.e. orogenesis, slab subduction, continent-continent collision and penetration of the Earth's mantle transition zone are examples of such processes. There is also a strong correlation between mineral content, phase transitions and structural properties of natural rocks. Ductile rock deformation is a typical property for Earth's mantle conditions. Nevertheless most of experimental rock deformation was conducted under crustal conditions in the past. So, it was a revolutionary event when the first Deformation-DIA was introduced about a decade ago. Today this technique is indispensable not only for rock deformation under unextrapolated Earth's mantle conditions but also for attenuation measurements in the seismic frequency range and attaining of lower mantle conditions in Large Volume Presses. In principle all these techniques require the installation of the high pressure device at a 3rd generation light source.

Thermoelasticity of hcp-Fe to 1.7 Mbar

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Abstract

Iron is thought to be the main constituent in Earth's core, along with ~5 to 10 wt% nickel and some light elements (e.g., H, C, O, Si, S). Current estimates for the light element component of the core are poorly constrained, due in part to uncertainties in the high-pressure thermoelastic and thermodynamic properties of both pure iron and iron-alloys. It is important to first firmly establish the high-pressure properties of the end-member composition of pure iron, which will then provide a solid baseline against which to compare measured properties of iron-alloys. In addition, such measurements will help to better constrain the composition of Earth's core via comparison with seismic observations and models of this remote region.

To further our understanding of iron at core pressures, we directly probed its total phonon density of states (DOS) at 300 K and up to 1.7 Mbar using nuclear resonant inelastic x-ray scattering (NRIXS) and *in situ* x-ray diffraction (XRD) experiments, performed at beamline 3-ID-B of the Advanced Photon Source at Argonne National Laboratory. Our unique analysis and *in situ* determination of sample volume with XRD distinguishes this study from previous similar works. In addition, we collected between 8 and 21 NRIXS scans at each of our eleven compression points—a subset of which were conducted with a neon pressure medium—making our high-pressure phonon DOS the most accurate reported to date to 1.7 Mbar.

From the phonon DOS, we determined a number of high-pressure thermoelastic parameters for iron, such as the vibrational entropy (S_{vib}) and sound velocities. From the volume-dependence of S_{vib}, we determined iron's ambient-temperature thermal expansion coefficient up to 171 GPa. This parameter serves as a self-consistent check on our previously determined vibrational thermal pressure and Grüneisen parameter, and provides a means for determining adiabatic bulk moduli from reported values for the more experimentally accessible isothermal bulk moduli at 300 K. We use this adiabatic bulk modulus to determine the Debye sound velocity of ε-Fe from the low-energy region of the phonon DOS and in turn, establish a new tight constraint on the density (pressure) dependence of ε-Fe's compressional and shear sound velocities. Based on comparisons between our measured sound velocities for iron and those inferred for Earth's solid inner core, we will discuss the presence of light elements in Earth's core.

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Lithium amidoborane at high pressure up to 19 GPa in diamond anvil cell

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One of the major obstacles to the use of hydrogen as an energy carrier is the lack of proper hydrogen storage media. Lithium amidoborane has attracted significant attention as hydrogen storage medium due to its low dehydrogenation temperature. It releases ~10wt% hydrogen, which is beyond the DoE target, at around 90^{o}C. It is essential to study the structural behavior of this potential material to improve its dehydrogenation behavior further and also to make rehydrogenation possible. We have studied the phase stability of this material at high pressures in a diamond anvil cell using in-situ Raman spectroscopy. Two phase transitions were observed at 3.5 GPa and 12.5 GPa based on the changes in the Raman spectrum. These changes appear with splitting of the N-H and B-H stretching modes, which indicates the structural complexity increases with applied pressure. Also, N-H stretching modes do not show redshift with pressure, indicating that there is no dihydrogen bonding in this material. The absence of the dihydrogen bonding in this material is an interesting phenomenon, as the dihydrogen bonding is the dominant bonding feature in its parent compound ammonia borane. This observation provides guidance to improvement of the hydrogen storage properties of this material.

Ferrous iron partitioning between magnesium silicate perovskite and ferropericlase

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(Mg,Fe)SiO_3 perovskite (Mg-Pv) and (Mg,Fe)O ferropericlase (Fp) are comprise more than 90% of the Earth's lower mantle. The chemical and physical properties of these phases are essential, therefore, for understanding the structure and dynamics of the lower mantle. We have investigated the exchange of Fe and Mg between the dominant lower mantle phases at 25 GPa and 2400 to 2600 K using a Kawai-type multianvil apparatus. $(Mg,Fe)_2SiO_4$ olivine with different compositions (Fo#70–95) were used as starting specimens. Each experiment was performed with coexisting metallic Fe, which buffered the oxygen fugacity at the lowest possible level. As the system was Al-free, the presence of metallic Fe ensures low ferric iron (Fe^{3+}) levels in all phases and allows ferrous iron (Fe^{2+}) partitioning to be characterized. The results are used to extract thermodynamic data to describe Fe^{2+}-Mg partitioning. A thermodynamic assessment and modeling of the available high pressure partitioning data indicates that the influence of an Fe-spin transition in Fp on Fe-Mg partitioning may be more subtle than previously proposed. Furthermore, we demonstrate that a comparison between Mg-Pv Fe^{2+} contents predicted by the thermodynamic model and previously reported perovskite analyses can be used to estimate Mg-Pv Fe^{3+} concentrations of both Al-free and Al-bearing phases in the previous studies. These estimates show that the Fe^{3+} content of Al-free Mg-Pv depends strongly on oxygen fugacity, and varies accordingly with the capsule materials used in experiments. On the other hand, the Fe^{3+} content of Al-bearing Mg-Pv shows no clear dependence on oxygen fugacity, however, it depends apparently on Al and bulk Fe contents. The relationship between Fe^{3+} and Al concentrations indicates that the dominant substitution mechanism of Fe^{3+} and Al into Mg-Pv changes with Al content, i.e. from $MgFe^{3+}O_{2.5}$/$MgAlO_{2.5}$ oxygen vacancy substitution at low Al contents to $Fe^{3+}AlO_3$ coupled substitution at high Al contents. The currently prevailing view is that the lower mantle may be dominantly of a peridotitic composition, however, it plausibly also contains chemical heterogeneities arising from the presence of recycled depleted lithosphere and oceanic crust. Variations in rock composition in the lower mantle will result in the formation of Mg-Pv with quite differing Al and bulk Fe concentrations, which will cause differences in Fe^{3+} contents and the corresponding substitution mechanisms. These changes are probably associated with differences in geophysical properties for different materials in a heterogeneous lower mantle.

Effect of sulfur on the sound velocity in liquid iron at high pressure

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The Earth's outer core is thought to be composed of Fe–Ni alloy with about 10% of light elements which affect to increase the sound velocity of pure iron (Anderson and Ahrens, 1994). Since sulfur is a major candidate of the light elements, it is important to understand the effects of S on the sound velocity of liquid iron at high pressure. Although the effect of S on V_P of liquid Fe has been reported based on the calculation from density and bulk modulus, there is no direct measurement of Fe-S liquid at high pressure. Here we report the results of P-wave velocity (V_P) measurements of liquid Fe–S up to 5.4 GPa and 1550 °C.

High pressure experiments were performed using a 1500 ton Kawai-type multi-anvil apparatus (SPEED-1500) at BL04B1 beamline, SPring-8, Japan. V_P measurements were carried out using the pulse echo overlap method. P-wave and S-wave signals with a frequency of 37 MHz and 21 MHz, respectively, were generated and received by 10° Y-cut $LiNbO_3$ transducer. S-wave signals were used to verify the sample melting.

The V_P of liquid Fe–S showed negligible temperature dependence and it decreases linearly with increasing S content at 2.5 GPa and 1300 °C. The V_P of liquid $Fe_{57}S_{43}$ increases almost linearly from 3048 ± 13 m/s to 3845 ± 9 m/s with increasing pressure from 1.3 to 5.4 GPa. Although the V_P of liquid $Fe_{57}S_{43}$ is smaller than that of pure liquid Fe up to 5.4 GPa, the V_P of liquid $Fe_{57}S_{43}$ is expected to exceed that of liquid Fe over 7 GPa because the pressure derivative of V_P of liquid $Fe_{57}S_{43}$ is larger than that of liquid Fe. This result suggests S is effective in increasing the V_P of liquid Fe over 7 GPa. Therefore, S is considered to be a possible light element of the Earth's outer core.

The Large Volume Press Beamline at the extension of PETRA III using high energy X-rays

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PETRA III is currently the brightest 3rd generation strage ring in the world. During the initial PETRA III project, 14 beamlines were constructed occupying one octant of the 2.3 km storage ring. One of these beamlines is the Extreme Conditions Beamline (ECB, P02.2) that is dedicated to extreme conditions research in the DAC using hard X-ray diffraction techniques with a strong focus on time resolved capabilities. In order to compliment and expand on the existing extreme conditions research capalilities, DESY decided to develop a new LVP instrument at the extension of PETRA III (Hall North) that will be located at one of the 100 m long damping wiggler section of the storage ring. It offers the unique opportunity to develop one of the most intense hard X-ray LVP beamlines in the world. Construction of the experimental halls, Hall North and Hall East, will start in March 2013 and beamline constructions are scheduled to begin in 2014. Here we report the conceptual design of this new beamline. X-rays from the damping wiggler will be filtered by an absorber to cut X-rays below 40 keV because the power and heat of the damping wiggler beam will be too high for optical components. The pink beam will be used for energy dispersive X-ray diffraction experiments under high pressure. We believe that time-resolved X-ray diffraction study can be performed using this setup. We are planning to install double crystal monochromator in Laue configuration to obtain mono-beam with 45-120 keV. We are planning to have a 6-axis press in this beamline with capability of deformation of samples under confining pressure similar to the DDIA capability. The combination of the high energy mono-beam and the 6-axis press will allow us to carry out stress/strain-analysis based on distortion of Debye rings as well as study of lattice preferred orientations. We are also planning to install a multi-channel collimator system for powder X-ray diffraction studies under high pressure. This technique will enable us to perform structure refinement using the Rietveld method in order to refine atomic positions of materials in wide range of pressures and temperatures range up to 30 GPa and 2500 K using WC anvils. Since the 6-axis press can keep the pressurized space cubic under high load, use of sintered diamond anvils with the 6-axis press may allow us to extend the reachable pressure range beyond 1 Mbar. We believe that the instrumentation of the new LVP beamline will be very powerful for geosciences and material sciences. PETRA III is an open access facility and beamtime will be distributed based on the scientific merit of the proposed research.

Partitioning of Potassium into the Earth's Core

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The possible presence of potassium in the Earth's core as a radioactive heat source can have a significant influence on the thermal evolution of the Earth[1]. Our recent study[2] showed the experimental evidence on the gravitational stability of basal magma ocean[3] against the crystallized solid silicate at >76 GPa conditions. This dense, incompatible element-rich reservoir would evolve in equilibrium with the liquid core at the core-mantel boundary pressure conditions during the early cooling history of the Earth and constrain the content of potassium in the Earth's core.

The partitioning of K between liquid Fe, Fe-FeS (2wt%) alloys and KLB-1 compositional silicate melt has been determined up to Mbar and 5000K using laser-heated diamond anvil cell and FE-EPMA (JEOL JSM-7000F). The results show small effect of pressure but large effect of temperature on partitioning behaviors of K, suggesting the effect of Fe-K alloying[4] at >25GPa was not significant at high temperature conditions. The clear correlation between partitioning coefficient of O and K was observed as suggested by Corgne et al., (2007)[5]

The results are interpreted in respect to the thermal history of the Earth in the presentation.

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Elasticity of Diamond at High Pressures and Temperatures

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Abstract

We combine density functional theory within the local density approximation, the quasiharmonic approximation, and vibrational density of states to calculate single crystal elastic constants, and bulk and shear moduli of diamond at simultaneous high pressures and temperatures in the ranges of $0-500$ GPa and $0-4800$ K. Comparison with experimental values at ambient pressure and high temperature shows an excellent agreement for the first time with our first-principles results validating our method. We show that the anisotropy factor of diamond increases to 40% at high pressures and becomes temperature independent.

Thermoelastic Properties of Ringwoodite $(Fe_x,Mg_{1-x})_2SiO_4$: Its Relationship to the 520 km Seismic Discontinuity.

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Abstract

We combine density functional theory (DFT) within the local density approximation (LDA), the quasiharmonic approximation (QHA), and a model vibrational density of states (VDoS) to calculate elastic moduli and sound velocities of ringwoodite $[\gamma-(Fe_x,Mg_{1-x})_2SiO_4]$, the most abundant mineral of the lower Earth's transition zone (TZ). Comparison with experimental values at room-temperature and high pressure or ambient-pressure and high temperature shows good agreement with our first-principles findings. Then, we investigate the contrasts associated with the $\beta \rightarrow \gamma(Fe_x,Mg_{1-x})_2SiO_4$ transformation at pressures and temperatures relevant to the TZ. This information offers clearly defined reference values to advance the understanding of the nature of the 520 km seismic discontinuity.

Thermoelastic Properties of Olivine and Wadsleyite $(\mathrm{Fe}_x,\mathrm{Mg}_{1-x})_2\mathrm{SiO}_4$: Their Relationship to the 410 km Seismic Discontinuity.

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Abstract

We combine density functional theory (DFT) within the local density approximation (LDA), the quasiharmonic approximation (QHA), and a model of vibrational density of states (VDoS) to calculate elastic moduli and sound velocities of $\alpha-$ and $\beta-(\mathrm{Fe}_x,\mathrm{Mg}_{1-x})_2\mathrm{SiO}_4$ (olivine and wadsleyite), the most abundant minerals of the Earth's upper mantle (UM) and upper transition zone (TZ). Comparison with experimental values at room-temperature and high pressure or ambient-pressure and high temperature show good agreement with our first-principles findings. Using our results, we investigate the discontinuities in elastic moduli and velocities associated with the $\alpha \rightarrow \beta-(\mathrm{Fe}_x,\mathrm{Mg}_{1-x})_2\mathrm{SiO}_4$ transformation at pressures and temperatures relevant to the 410 km seismic discontinuity. We find the compressional velocity contrast to be smaller than the shear velocity contrast, in agreement with the preliminary reference earth model (PREM).

Experiments on diamond transformation to graphite in the presence of fluid

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Graphite and diamond occur in mantle and crustal ultrahigh pressure metamorphic (UHPM) rocks they play an important role for understanding deep carbon reservoirs and global carbon cycling. Though the stability fields of carbon are well studied experimentally, the mechanisms of diamond-to-graphite and graphite-to-diamond transformation are not well constrained yet. Observations on natural samples from UHPM terranes show that diamond reaction to graphite was accompanied by the formation of disordered graphite which was recrystallized to "normal" graphite during exhumation and tectonic uplift. The disordered graphite was characterizied by HRTEM studies due to the presence of d_{002} =0.346, = 0.373 and =0.447 nm interplanar and lattice fringe spacings. Because the structures of diamond and graphite are very dissimilar, it is unlikely that a direct phase transformation would occur during the graphitization process. Such an assumption is supported by higher enthalpy (1.897) and lower entropy (2.38) of C-diamond with respect of those of C-graphite (enthalpy = 0, entropy=5.74). At least three different mechanisms of diamond-to-graphite transformation have been proposed. We conducted a series of anhydrous and hydrous experiments in a piston-cylinder apparatus at P=1 GPa and T=1300^{o}C using 20 micron size synthetic diamonds as the starting material. $Mg(OH)_2$ was added to the starting material in all hydrous experiments as the H_2O supply according to the breakdown reaction: $Mg(OH)_2 = MgO + H_2O$. The duration of all experiments was 5 hrs. Run products were studied with SEM, 3-D Raman spectroscopy combined with SEM, and FIB assisted TEM. The anhydrous experiments show that diamond crystals are "integrated" together producing deformed diamond polycrystallites with complicated textural patterns occurring at the grain boundaries. The hydrous experiments show the formation of spheroidal nanometric (20 nm) carbon particles, and tiny flakes of graphite at the {100} and {111} diamond surfaces. The latter was confirmed to be a disordered graphite (TEM studies) which is characterized by 1580-1560 cm^{-1} Raman bands. The Raman spectrum of the starting materials was 1332-1331 cm^{-1} what corresponds to the sp^{3} bonding of diamonds. The spheroidal carbon particles were distributed at the surfaces of the starting diamond material in the run products of the hydrous experiments and were characterized by Raman bands at 1321, 1446, 1620 and 1647 cm^{-1}. These new spheroidal carbon materials exhibit the presence of sp^{2} cyclic structures, and have high sp^{2}/sp^{3} ratios. However, a Raman spectroscopy method alone cannot always be used to uniquely assign a spectrum to a given carbonaceous material because Raman lines are sensitive to temperature, pressure, and stress or strain (Chiapra et al., 2011). New studies await to clarify and coordinate two different measurements arrived from TEM electron difraction patterns analyses and Raman spectroscopy of carbon-polymorphs produced at high PT hydrous and anhydrous experiements.

Experimental study on the martensite-like phase transition mechanism of graphite to hexagonal diamond

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Hexagonal diamond (lonsdaleite) is a metastable polymorph of carbon and consists of ABAB… stacked sp3-bonded (tetrahedral) carbons. It occurs as microscopic crystals associated with graphite and cubic diamond in carbonaceous meteorites such as the Canyon Diablo meteorite and impact craters and can also be synthesized from well-crystalline graphite by high pressure experiments (e.g. Bundy and Kasper, 1967; Yagi et al., 1992). The phase transition of graphite to hexagonal diamond is considered to be a martensite-like process, where [100] of hexagonal diamond is located parallel to [001] of the host graphite. However, we recently found a variety of coaxial relations between graphite and hexagonal diamond based on TEM observations of samples synthesized by high-pressure experiments. Based on these results, here we discuss the nature of the phase transition mechanism.

We performed a series of high *P*-*T* experiments using a laser-heated diamond anvil cell (DAC) at pressures of 25 and 50 GPa and temperatures ranging up to 3300 K. Highly oriented graphite was compressed in a DAC without using a pressure transmitting medium and rapidly heated to a target temperature 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 phases occurred under those *P*-*T* conditions. 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 obvious cubic diamond peak centered at 1332 cm^{-1}. TEM observation revealed that the transparent area in each recovered sample consists mainly 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 some reciprocal patterns in which [100] and [001] of hexagonal diamond are both arranged in a coaxial relation with graphite [001]. This suggests that the martensitic phase transition from graphite to hexagonal diamond proceeds as a result of $1/2\sqrt{3}a$ or $1/\sqrt{3}a$ layer shifts of graphene layers along graphite [100].

Pressure-induced metallization in FeO with rocksalt-type structure

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FeO is one of the fundamental components in the Earth's interior as the iron endmember of ferropericlase, the second most common mineral in the Earth's lower mantle. It is likely to keep the rocksalt (B1) type structure throughout Earth's lower mantle according to recent x-ray diffraction studies [*Ozawa et al.*, 2008]. Over two decades ago, the existence of a high-pressure metallic phase of FeO was first suggested based upon measurements of resistivity under shock loading [*Knittle et al.*, 1986], and the observed metallization has long been considered to be due to a structural transition to the NiAs structure [*Fei and Mao*, 1994].

We report the electrical conductivity of B1 FeO measured up to 141 GPa and 2480 K in a diamond-anvil cell. We found that B1-type structured FeO metallizes at around 70 GPa and 1900 K without any structural phase transition. In addition, we computed fully self-consistently the electronic structure and the electrical conductivity of B1 FeO as a function of pressure and temperature, and found that although insulating as expected at ambient condition, B1 FeO metallizes at high temperatures, consistent with experiments. The observed metallization is related to spin crossover.

Presence of such highly conductive FeO at the core-mantle boundary region can enhance the electromagnetic interaction between mantle and core, which would induce the anomalous features in observed Earth's rotation. Since we know that the MgO endmember of magnesiowüstite is insulating throughout the Earth, the existence of metallic FeO requires a two-phase field for the MgO-FeO binary system. This will modify the MgO-FeO-SiO_2 ternary for iron rich compositions, so that phase relations in the deep Earth could be more complicated than assumed.

Sound velocity measurement of hcp-iron and iron-light-element alloys at high pressure and temperature

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We measured the sound velocity of hcp-iron by the inelastic X-ray scattering (IXS) method using DAC at high pressure and temperature. Inelastic X-ray scattering spectra were taken at BL35XU, Spring-8. The dispersion of longitudinal acoustic phonons of the sample was measured by three spectrometers for 4 values of the momentum transfer. The average acoustic sound velocities were fitted by the sine curve using the equation with free parameters of Vp and Qmax (1). We used the symmetric diamond anvil cell for the measurements at high pressure and room temperature, whereas the Basset type external heating cell was used for the measurements at high temperature up to 1000 K. We also measure the sound velocity and density of FeH up to 70 GPa and Fe_3S up to 85 GPa and room temperature.

We made the inelastic X-ray scattering measurements of hcp-iron at pressures up to 167 GPa at room temperature. Sound velocity measurements at high pressure and temperature were made in the pressure range from 53 GPa to 91 GPa and the temperature range from 300 K to 1000 K using the Bassett type external heating diamond anvil cell. The experimental pressure was evaluated by the cell volume determined by XRD at the same beamline BL35XU before and after the IXS measurement and the equation of state of the sample calibrated by the Pt scale by Matsui et al.(2). When we plot sound velocity and density of hcp-Fe measured in this work in the Birch's diagram, we found that the Vp-density relation at 300 K is consistent with the trend reported by Mao et al.(3) measured by NIRIXS, and slightly higher in velocity compared to that reported by Fiquet et al.(1) using IXS method. The temperature dependency obtained in this work shows almost no temperature effect, i.e., the Birch law is applicable up to the temperature at least 1000 K up to c.a. 91 GPa in contrast to the previous NIRIXS work by Lin et al.(4). This is consistent with the recent result by Antonangeli et al.(5).

We estimated the inner core compositional range of the light elements of H and S which can explain the PREM density and sound velocity based on the present IXS measurements of the inner core candidate materials.

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

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Deformation experiments on hydrous melt-bearing dunite (olivine + 4 vol.% orthopyroxene + 4 vol.% clinopyroxene with less than 2.5 vol.% of the melt phase) were conducted at pressures of 1.3−5.7 GPa and temperatures of 1270−1490 K in order to explore the effect of intergranular fluids on the plastic flow of olivine in Earth’s upper mantle. The strain rate was proportional to steady-state creep strength to the 2.1 power, and the creep strength markedly increased with increase in grain size. Developments of the crystallographic preferred orientation of olivine and flattening of olivine grains were hardly observed even after 33−55 % shortening of the samples. These observations show that grain boundary sliding (GBS) dominated the deformation of olivine (i.e., superplasticity). The creep strength of hydrous melt-bearing dunite was 2−5 times lower than that of melt-free dunite. The dependence of creep rate on melt fraction is known to be expressed empirically as $\dot{\varepsilon}(\phi) = \dot{\varepsilon}(0) \cdot \exp(\alpha\phi)$, where α is a constant and ϕ is the melt fraction. The experimentally obtained value of α was in the range of 150−230, corresponding to 5−7 times the reported values for the olivine−basalt system at 0.3 GPa (i.e., creep strength of dunite was efficiently reduced by the hydrous melt). Superplasticity is the dominant creep mechanism of olivine in fluid-bearing fine-grained peridotites under low-temperature and high-stress conditions (i.e., peridotite shear zones in the upper mantle). Superplasticity induced by geological fluids would play an important role in the shear localization (and thus initiation of subduction) in the upper mantle.

Morphological Instabilities and the Chemical Interaction at the Core-Mantle Boundary of the Earth

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Abstract

The core-mantle boundary is a region where molten iron-rich materials interact with oxides/silicates in the mantle. A range of geophysical and geochemical observations suggest that iron-enrichment might occur at the bottom of the mantle leading to low seismic wave velocities and high electrical conductivity. However, physical processes of iron-enrichment have been poorly understood. First, diffusion-controlled iron enrichment is too inefficient. Second and more fundamentally, the recent experimental studies show that the core is under-saturated with oxygen implying that the mantle next to the core should be depleted with FeO. Here, we show, based on the high-pressure experiments, that iron-depletion in oxide ((Mg,Fe)O) in contact with molten iron leads to the morphological instability by which efficient penetration of liquid iron blobs occurs into (Mg,Fe)O. Iron-rich melt could be transported into the mantle by ~50 km by this process providing an explanation for the iron-rich regions at the core-mantle boundary.

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High *P*-*T* XRD experiments using fabricated controlled-geometry metal/oxide/metal double hot-plate samples

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To model and predict the structure, dynamics, and composition of Earth's deep interior and to properly interpret density profiles generated from seismic data, accurate and precise measurements of thermal expansion and compressibility are required. These measurements need to be made for constituent Earth materials at extreme pressures and temperatures. The laser-heated diamond anvil cell (LHDAC) coupled with synchrotron-based x-ray diffraction (XRD) is a powerful tool to determine pressure-volume-temperature relationships. However, LHDAC experiments may be hampered by an offset of the x-ray beam with respect to the center of the laser-heated hotspot. This offset can result from small misalignments on the order of a few microns. Such misalignments cause systematic errors which result in flawed predictions of the material's density at the conditions of Earth's core and mantle. Fabrication of controlled geometry samples for use in the LHDAC eliminates the uncertainty in the relative alignment of the laser and x-ray beam. We have designed samples of Ni and SiO_2 that are mass manufactured ($>10^7$ samples) using a combination of physical vapor deposition (PVD), photolithography, acid etching, and plasma etching. The controlled-geometry samples have dimensions of 15 μm x 15 μm x 0.3 μm thick and are arranged as a double hot-plate. The dimensions of the samples are similar in size to that of a synchrotron x-ray beam and smaller than the laser-heating hotspot. The size of these samples ensures that the volume of the sample probed by the x-ray is at the peak hotspot temperature. This guarantee allows for more precise measurements of thermoelastic properties which will lead to more accurate models of the Earth's deep interior. The fabricated samples were compressed in a LHDAC to $P = 24 – 70$ GPa and heated to $T = 1100 – 2000$ K by double-sided laser heating. We present initial results from *in situ* XRD experiments conducted at HPCAT beamline 16-ID-B. These preliminary experimental results show a need to solve the difficulties of Ni layers spreading during laser heating when the layers are less much less than 500 μm. We present progress made toward fabricating thicker samples in order to address the metal migration complications. The thicker samples are fabricated with dimensions of 5 μm x 5 μm x 2 μm thick and have a $Ni/SiO_2/Ni$ chemistry.

Diamond Anvil Cell Facilities at GSECARS

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The main goal of the diamond anvil cell (DAC) program at GSECARS is to address key geochemical and geophysical problems across the entire pressure-temperature range of the Earth and other terrestrial planets. The DAC experiments cover all the major aspects of in-situ studies of minerals and non-crystalline materials at extreme conditions utilizing the wide range of experimental techniques available at GSECARS: micro-x-ray diffraction with monochromatic radiation, inelastic x-ray scattering (x-ray Raman), x-ray emission spectroscopy, x-ray fluorescence microprobe, x-ray absorption and radiography. To maintain the GSECARS high pressure instrumentation as state-of-the-art research tools we are continuously investing in the latest technological tools to help the high pressure user community perform their research at the highest potential. This talk summarizes current capabilities and future directions in the diamond anvil cell facilities at GSECARS, sector 13 at the Advanced Photon Source [1-4], including new developments:

On-line techniques.

- New capabilities at the 13-IDD station for time-resolved experiments - in-situ measurements of x-ray diffraction and temperature in the pulsed laser heated DAC with optional APS storage ring bunch modes synchronization
- Fast high pressure single-crystal step-scan measurements (on fly) supported with analytical software are possible now at 13-IDD and 13-BMD stations with new PerkinElmer detector
- Single crystal sample screening at ambient pressure at 13-BMC
- The on-line Brillouin spectroscopy at 13-BMD station combined with high resolution x-ray diffraction techniques and in-situ Raman, fluorescence (Ruby), and absorption spectroscopy in the range of 500 – 1600 nm, including spectroscopic measurements of relatively low temperatures (550-1000 K) in the externally heated DAC
- A new time-domain (from nanoseconds to minutes) on-line optical spectrometer at 13-IDD station for in-situ fluorescence (Ruby x-ray or laser excited), absorption spectroscopy in the range of 500 – 1600 nm and spectroscopic measurements of relatively low temperatures (550-1000 K) and very high T (above 4,000 K) in the externally and laser heated DAC

Off-line techniques available for all APS and ANL users.

- Continuous and pulsed off-line one side laser heating with T measurements
- Gasket and sample pulse laser cutting technique
- Gas-loading of CO_2, Ar and N_2 in addition to Ne and He
- Raman and optical spectroscopy

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Magnesite synthesis from olivine and CO_2

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Magnesite ($MgCO_3$) is regarded as the primary carbon-bearing mineral in the Earth's mantle, and its stability has been verified experimentally [1,2] and computationally [3,4] along typical mantle geotherms. Explanations for magnesite's presence in the mantle include transport via subduction of ocean slabs, as well as exsolution from dolomite at ~200 km depth [5]. However, the presence of solid CO_2 in mantle xenolith outcrops [6] suggests a third explanation - chemical synthesis within the mantle via the reaction:

$$Mg_2SiO_4\ (s) + 2\ CO_2\ (s) = 2\ MgCO_3\ (s) + SiO_2\ (s) \qquad (1)$$

Since Mg_2SiO_4 phases, such as olivine, wadsleyite and ringwoodite, are abundant in the mantle, this reaction may lead to the formation of magnesite. However, the very presence of free CO_2 in mantle xenoliths suggests the possibility of kinetic barriers to this reaction. In order to investigate the formation of magnesite within the mantle, we have investigated the reaction in Eqn. (1) experimentally, using a laser-heated diamond-anvil cell (DAC) to simulate mantle conditions, using synchrotron powder X-ray diffraction to monitor *in situ* chemical reactions and phase transitions. We observed the formation of magnesite over a range of mantle conditions between 8-35 GPa and 1300-2000 K, suggesting that this mechanism may account for magnesite in the mantle.

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Synthesis and mechanical properties of polycrystalline gamma-boron

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Boron is one of the nonmetal elements that have been widely studied due to its complex polymorphism and fascinating chemical and physical properties. Among elemental boron polymorphs, only α-rhombohedral (α-B), β-rhombohedral (β-B), γ-orthorhombic (γ-B), and β-tetragonal (T-192) have been currently established as pure phases. However, the relative stability of boron phases is still experimentally unresolved at high pressure and high temperature (HPHT), and the mechanical properties of polycrystalline γ-B are also not clear. Here, we report the phase relations in boron at HPHT and mechanical properties of polycrystalline boron.

The phase relations in boron have been investigated at HPHT using a multianvil apparatus, and the quenched sample has been analyzed by x-ray diffraction, Raman spectra and transmission electron microscopy. We demonstrate that γ-B_{28} can be synthesized over a wide P-T range, and T-B_{50} is obtained at higher temperatures and similar pressures. The phase boundary of the β-B_{106}, γ-B_{28} and T-B_{50} is determined at pressures between 7 and 18 GPa and the temperatures of 500-2200 °C. The results suggest that T-B_{50} might be an intermediate phase-formed for kinetic reasons (Ostwald rule) on the way from β-B_{106} to T-192 and γ-B_{28} to T-192.

The Vickers hardness of polycrystalline γ-B was measured using a diamond indentation method. The elastic properties of polycrystalline γ-B were determined using ultrasonic measurement method. Under the loading force up to 20 N, our test gave an average Vickers hardness in the asymptotic-hardness region of 30.3 GPa. The average fracture toughness was measured as 4.1MPa $m^{1/2}$. We also measured the hardness and elastic properties of polycrystalline β-B and PcBN for comparison. The hardness and elastic properties for polycrystalline γ-B was found to be very close to that of PcBN. Our results suggest that the polycrystalline γ-B could be a superhard polycrystalline material for industrial applications.

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Phase relations, melting and thermoelastic properties in FeS to 65 GPa and 2650 K

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The phase stability and equations of state of the NiAs superstructured FeS IV phase and the high-temperature NiAs structured FeS V phase (space group $P6_3/mmc$) have implications for the structure and dynamics of planetary cores, such as that of Mars. A suite of high-pressure, high-temperature X-ray diffraction data were collected on stoichiometric FeS at pressures ranging from 15 to 65 GPa and temperatures reaching 2650 K using a laser-heated diamond anvil cell. Experiments were performed at beamline 13-ID-D (GSECARS) at the Advanced Photon Source. We refined the high-temperature, high-pressure phase boundaries of the FeS IV and FeS V polymorphs. The phase transition from FeS IV to FeS V is indicated by the disappearance of some or all of the following FeS IV reflections: 110, 002, 210, 211, 220, 311. Fixing K'_0 at 4 and q at 1, we find $V_0 = 7.79$ cc mol^{-1} and 8.04 cc mol^{-1}, $K_{0T} = 98.7$ GPa and 91.6 GPa, and $\gamma_0 = 2.15$ and 1.47 for the FeS IV and FeS V phases, respectively. Our suite of data on FeS polymorphs also included *PVT* measurements on the FeS VI phase (space group Pnma), and for this phase we find $V_0 = 8.27$ cc mol^{-1}, $K_{0T} = 89.1$ GPa and $\gamma_0 = 1.36$. At conditions of the Martian core, we find that the stable phase at the core-mantle boundary is FeS IV, with a possible transition to FeS V occurring deeper in the Martian core. At ~ 40 GPa, we observe melting of FeS V consistent with the melting curve of Boehler (1992).

GSECARS Canted Undulator Upgrade

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GSECARS is in the final stages of an upgrade of the undulator beamline to a canted geometry. This project adds a new experiment station, 13-ID-E which provides a dedicated x-ray microprobe that runs 100% of the time, and is completely independent. The 13-ID-C station now houses only the large Newport 6-circle diffractometer, while 13-ID-D station continues to house the laser-heater diamond anvil cell and 1000-ton multi-anvil press. Because the microprobe will now have its own independent station, the time available for high-pressure experiments will increase by about 33%.

As part of this upgrade the undulator that feeds the 13-ID-C and 13-ID-D stations was replaced. The previous device had a 3.3 cm period, while the new device has a 3.0 cm period, which produces a factor of 2 more x-rays at the energies used for high-pressure experiments. This undulator was installed and commissioned in Jan. and Feb. 2012. In the middle of March 2012 the beamline was shut down to install new optics for both the ID-C/D and ID-E branches. The monochromator for the C/D branch will now have both Si 111 and 311 crystals, allowing it to reach at least 65 keV, significantly higher than the previous limit of 45 keV, and important for high-pressure experiments.

The ID-E microprobe has a 3.6 cm period undulator, capable of going down to the sulfur K-edge, and up to about 28 keV. It will have a compound x-ray focusing system capable of reaching sub-micron spot size.

The new optics for the ID-C/D branch will be commissioned starting June 1, 2012 and high-pressure users will have access to the beamline beginning a few weeks later. The new ID-E microprobe is expected to begin commissioning sometime later this summer.

Novel microfocus x-ray sources for high-pressure crystallography

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Diamond anvil cells (DACs) are widely used for examining the crystal structure of materials under high pressure. The area of reciprocal space accessible in a high-pressure X-ray diffraction experiment is primarily restricted by the geometry of the DAC. For a typical high pressure experiment using Mo radiation, only a small fraction of all reflections can be collected. This can be as low as 30% for triclinic crystal structures. Using radiation with a shorter wavelength, such as Ag Kα, a larger portion of the reciprocal space is accessible, thus increasing the number of observations and the resolution of the data. However, because of the low intensity of conventional Ag sealed tubes,
Ag sources are rarely used for high-pressure studies in the home lab. Microfocus sealed tube sources have proven to deliver flux densities beyond that of traditional X-ray sources when combined with 2D focusing multilayer mirrors [1, 2]. The sharp beam profile of these sources produces a high flux density at the sample position, thus leading to strong diffracted intensities. Furthermore, the small beam cross-section significantly reduces the background that usually results from scattering at the gasket of the DAC. Therefore, this type of source presents a promising alternative to classical sealed tube sources currently being used in high-pressure crystallography. We will be reporting on the latest developments on microfocus X-ray sources (Ag and Mo anodes) which enable a clear increase in intensity compared to other sealed tube sources. Selected results on the use of these sources in high-pressure crystallography will be presented.

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A new high-pressure research facility at the Australian Synchrotron

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The development of large volume, multi-anvil experimental techniques in the United States and their use with synchrotron sources has brought new technology to the experimental community. These facilities have allowed us to significantly improve our understanding of the physical properties of Earth and planetary materials under high pressures and temperatures. In 2011-2012, this technology is being brought to the Australian Synchrotron, and will provide the Australian scientific community an opportunity to significantly improve understanding of materials under extreme conditions. The high-pressure deformation system located at the Australian Synchrotron is comprised of a D-DIA deformation apparatus and a 250T hydraulic press together with the necessary x-ray definition and detection systems. The development of a synchrotron-based, high-pressure and temperature deformation facility is an exciting new opportunity and a major advance in experimental capability for Australia. We will build into the facility a unique addition, the accompanying AC conductivity system that will allow conductivity measurements at the same time as x-ray diffraction and imaging. This is important for the projects where interconnectivity of fluids is being investigated; as it will provide us with an in-situ measure of melt connectivity.

Equation of state of Fe-Ni alloy at multi-megabar pressure

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Equation of state of Fe-Ni alloy is fundamentally important to understand the Earth's inner core. Especially, the uniaxial stress effect is important to discuss the effect of nickel on the density. Here we report the equation of state of $Fe_{0.9}Ni_{0.1}$ alloy up to 262 GPa with the laser annealing technique.

We used a symmetric-type diamond anvil cell with the diamond anvils of culet size of 40, 130 and 300 μm for high pressure generation. $Fe_{0.9}Ni_{0.1}$ reagent (99.99% pure, Rare Metallic Ltd.) was used as the starting material. The sample was sandwiched between layers of sodium chloride and loaded into a sample hole that had been drilled in a precompressed tungsten gasket. The unit cell volume of the sample was determined by the synchrotron X-ray diffraction experiment at the SPring-8 BL10XU beamline, Japan. The experimental pressure was determined by the 3rd order Birch-Murnaghan equation of state (B-M EoS) of NaCl-B2 phase as reported by *Sakai et al.*, 2011. The sample was annealed by a double-sided laser-heating method using a fiber laser at the BL10XU in order to minimize the deviatoric stress in the sample.

$Fe_{0.9}Ni_{0.1}$ was compressed to a pressure of 262 GPa at ambient temperature. The data sets were fitted using the 3rd B–M EoS. The density of $Fe_{0.9}Ni_{0.1}$ is 1.0% and 1.5% denser than that of pure iron (*Dewaele et al.*, 2006) at 200 GPa and 330 GPa, respectively.

Sound velocity and structure measurement of silicate glasses under pressure

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The degree of polymerization in silicate melt/glass is one of the most important parameters to understand the magma behavior. For silicate melts at ambient pressure, the degree of polymerization is highly related to composition, which is quantitatively described by a ratio of non-bridging oxygen (NBO) to tetrahedrally cation (T). In particular, the NBO/T is widely used to obtain viscosity information of various silicate melts and discuss the magma mobility in the Earth's interior. Several viscometry studies reported that polymerized melts showed much higher values of viscosity than those of depolymerized ones. Interestingly, it should be noted that the pressure dependence of the high viscosity of polymerized melts was shown to be negative. This gives important questions of the compression effect on the degree of polymerization and its effects on properties of silicate melts. In this study, we have measured the sound velocity of polymerized glass (jadeite and albite glass: NBO/T=0) and depolymerized glass (diopside glass: NBO/T=2) at pressures up to 10 GPa by using ultrasonic technique and synchrotron radiation with a Paris-Edinburgh press. We have also obtained the X-ray structure factor, $S(Q)$, of these glasses by using energy-dispersive X-ray diffraction method in order to understand structural changes in the intermediate-range order with pressure.

All experiments were conducted using a Paris-Edinburgh press, which is installed at the HPCAT 16-BM-B beamline, Advanced Photon Source (APS). High pressure sound velocity measurements were carried out using the ultrasonic pulse-echo-overlap method. The outer pressure media consisted of machinable zirconia pallets and sintered boron-epoxy. Graphite cylinder was used as a sample container, with a gold foil placed on top and bottom of the capsule as markers for sample length measurement. Radiography images taken by CCD camera allowed us to calculate the sample length under high pressure. Pressure was determined by the equation of state of gold, which was located below the sample. The scattered X-rays were detected using a Ge solid state detector (Ge-SSD) with a 4096 multi-channel analyzer. Alumina above the sample was used as a buffer rod. The glass sample and the alumina buffer rod were polished with 1 μm diamond paste. Ultrasonic signals were generated and received by a $LiNbO_3$ transducer. The signals were collected with a sampling rate of 5×10^9 point/second.

Structure measurements were performed using the energy dispersive X-ray diffraction technique. The 16-BM-B is a bending magnet beamline which provides white X-rays (5-120 keV) with high brightness. The incident X-ray was collimated by two sets of vertical (0.1 mm) and horizontal (0.1 mm) slits. The diffracted signal was collimated with a 0.1 mm gap scattering slit 80 mm downstream from the sample and a 0.1 mm × 5.0 mm receiving slit 400 mm further downstream from the scattering slit. The Ge-SSD was mounted on a two-theta arm on a large Huber rotation stage, which allows accurate control on two-theta angle. The diffraction patterns were collected for 9 fixed diffraction angles (2θ = 3°, 4°, 5°, 7°, 9°, 11°, 15°, 20°, 25°). Collecting time varied with the diffraction angles, as intensities decreased with increasing angle. All patterns were collected until the maximum intensity reached at least 2000 counts. Structure factor, $S(Q)$, was obtained by combining X-ray diffraction profiles collected for 9 diffraction angles.

Pressure dependence of sound velocity of jadeite, albite and diopside glasses will be presented, along with structure factor S(Q) of the glasses at high pressure. We would like to discuss a direct correlation between the intermediate-range order structure and sound velocity in these glasses, and the influence of the degree of polymerization.

Effect of Al content 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 a key role 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 change considerably with concentration of OH. Although water partitioning has been studied by many workers under water saturated concentration, 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, 0.1)). Powdered minerals were enclosed in metal foil capsule (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 1300ºC. Oxygen fugacity was controlled by 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.

Water partitioning coefficient between Ol and Al free Opx are $D_{(\text{Al free Opx/Ol})} = 1\sim2.3$. On the other hand, that between Al bearing Opx and Ol are $D_{(\text{Al bearing Opx/Ol})} > 4.1$. Thus $D_{(\text{Opx/Ol})}$ becomes larger with Al content of Opx. 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 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 density of dry and hydrous silicate melts in the upper mantle

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The density of silicate liquids is a key control on many magmatic processes, including magma chamber dynamics and volcanic eruptions, melt extraction from residual rocks during partial melting, fractional crystallization processes and crystal settling. However, the experimental data on the density and compressibility of silicate melts at relevant pressure and temperature conditions remain scarce, especially for volatile-bearing compositions.

In this contribution we report *in situ* investigations of the density of silicate liquids, including dry and hydrous granitic (5 and 10wt% H_2O) and alkaline (phonolite) compositions, at upper mantle conditions (3.5 GPa and 2000 K). The experiments were performed using a panoramic Paris-Edinbugh press at ID27 beamline of the ESRF. The samples were contained in a cylindrical diamond capsule, capped with a platinum disk on either side, surrounded by hexagonal boron nitride (hBN) and placed inside a graphite heater and boron epoxy gasket. Density was determined from the X-ray absorption contrast between the samples and a diamond capsule and pressure and temperature calibrated from the X-ray diffraction patterns of hBN and Pt using the double-isochor method. The molten state of the sample at the condition of the density measurements was verified by X-ray diffraction. The run products were analyzed by electron microprobe, infrared spectroscopy and SIMS to verify the chemical composition and volatile content of the samples.

The results, combined with literature data on silicate melt density at atmospheric pressure, provides the first experimentally derived equation of state for dry and hydrous granitic and phonolitic liquids at crustal and upper mantle conditions. The resulting equation of state for granitic liquids allows constraining pressure and compositional effects on the compressibility of dissolved water in silicate melts. Although the hydrous granitic liquids are buoyant compared to their dry counterparts at all investigated conditions, the density contrast decreases with pressure above 1 GPa, due to changes on the compressibility of dissolved water. Additionally, a comparison of the partial molar volume of dissolved water derived from this study with literature for MORB and peridotite melts suggests that the compressibility of dissolved water in silicate melts strongly depends on melt composition at upper mantle conditions (< 4 GPa). The implications of these results for the ascent rate of slab melts in subduction zones, the dynamics of magma chambers and crystal-liquid buoyancy relations and crystal settling velocities in the upper mantle will be discussed.

6-rams multi anvil press for neutron diffraction experiment at J-PARC

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Hydrogen is considered to play an important role in the deep mantle since it has a significant effect on the physical properties of minerals and rocks. Taking advantage of neutron diffraction that can detect light elements, beamline “PLANET” at J-PARC is aimed at investigating hydrogen in minerals and rocks in the earth’s interior. In the beamline, 6-rams press “ATSUHIME” is installed to generate high pressure and high temperature conditions in the earth’s mantle. 6-rams press is suitable for neutron diffraction experiment because it has a wide window surrounding the sample compared to other multi anvil press using guide block. It allows us to place the insertion device and radial collimator close to the sample, and to cover wide diffraction angle and azimuth angle.

The press consists from six hydraulic rams mounted on a flame with maximum load of 500-ton in each ram. The anvil stroke is measured by magnetic scale fixed on the flame. The measured stroke value is collected to eliminate the effect of the elongation of the flame at high pressure. Each ram is controlled independently by six plunger pumps so as to retain the deviation of the anvil stroke within 10 μm.

Three types of experimental mode are available: (1) hydrostatic experiment that compresses the cubic sample space uniformly, (2) uni-axial experiment using top and bottom rams and (3) deformation experiment that deform the sample using two opposed rams at horizontal direction. In general hydrostatic experiment, 6-6 type anvils are used for the second stage anvils. The detail of design and performance of the 6-rams press will be presented.

The impact of microstructure defects on the high pressure/high temperature phase transitions of boron nitride

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The microstructure of the boron nitride (BN) precursors used for the catalyst-free conversion of hexagonal BN (h-BN) into its dense wurtzitic and/or cubic form (w-BN/c-BN) is known to have a significant impact on the kinetics of the high-pressure/high-temperature (HP/HT) phase transformation and consequently on the microstructure and properties of the resulting product. Still, a fast optimization of the parameters of the HP/HT synthesis and the microstructure design of the synthesized product require a deeper understanding of the interplay between the microstructure defects and the kinetics of the phase transition.

For this reason, three different h-BN precursors containing different predominant microstructure defects have been used to synthesize BN (nano)composites in HP/HT experiments with nearly identical process parameters, which were accompanied by in situ energy dispersive X-ray diffraction (EDXRD) measurements. These in-situ experiments were performed at the MAX200X multi-anvil press located at beamline W2 at DESY/HASYLAB in Hamburg, Germany. The kinetic parameters of the conversion process of h-BN into w-BN and/or c-BN have been extracted mainly from the time and temperature dependent phase composition of the samples being converted; the evolution of the microstructure defects was concluded from the XRD line broadening . The in-situ HP/HT investigations benefited from an improved design of the high pressure cell and gasket construction, in which diffracting and/or strongly absorbing matters were eliminated from the beam path. The in-situ measurements were complemented by laboratory XRD measurements and TEM investigations in order to get more comprehensive information on the microstructure of the h-BN precursors and the products of the HP/HT synthesis. A model will be discussed explaining which transition paths in the h-BN → w-BN | c-BN system are blocked or opened through the presence of individual microstructure defects in h-BN.

New Assemblies for High Pressure-High Temperature Multianvil Synchrotron Studies with low X-ray Absorption

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In a number of visits at the MAX200X multianvil X-ray diffractometer at Beamline W2 of the DORIS source at Hasylab, DESY, we have constructed and tested a number of different designs for the sample assembly, including heater design, X-ray transparent and high temperature resistant amorphous window materials and new types of gaskets with low absorption.

Subject of our research are nitride-based advanced and superhard materials, which – due to a higher degree of covalent bonding – often require more severe conditions than those sufficient to drive phase transformations in many mineralogical systems usually studied by in-situ multianvil methods. The combination of relatively high pressures and high temperatures limits the utilization of conventional X-ray transparent graphite heaters, because of their conversion to diamond.

Moreover, in the white beam mode, theoretically all diffraction lines from the sample environment (pressure medium, heater material, encapsulant) can be eliminated by adjusting a sufficiently small aperture width. However, under realistic measurement conditions with a limited number of shifts, these optimizations are often not possible.

In particular less symmetric phases have comparatively complex diffraction patterns, so that the number of interfering lines should be kept minimal.

In the case of conversion studies of hexagonal boron nitride to the superhard forms of BN, a graphite heater would suffice, but has to be kept out of the diffraction path in order not to obscure the changes in the h-BN, whilst diffraction lines of the standard pressure medium MgO within the pressure range of interest would interfere with the buildup of the c-BN reflections.

Third, compared to other multianvil synchrotron facilities that operate with very brilliant sources, the beam intensity at W2 DORIS is comparatively low, so that absorption effects in conventional gasket materials like pyrophyllite become eminent. This is especially the case if fast transient processes with desirably short collecting times are to be monitored in the higher pressure region, where the gaskets are considerably squeezed and the absorbing beam-path is long. We believe that our experiences and developments could be beneficial for future concepts at DESY as well as other synchrotron sources that apply the multianvil technique.

Offline in-situ pressure calibration for heated multianvil experiments and several other improvements to the multianvil-technique

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A method that is independent from synchrotron sources and provides continuous monitoring of the sample pressure during loading and unloading of individual heated multianvil experiments has been devised. Based on previous results [1], we managed to accommodate one or more fixed-point calibrants together with a piezoresistive manganine gauge within octahedral pressure cells of various sizes, without interfering with the furnace assembly. The method gives the final pressure within the octahedron before heating, and moreover provides important information about the unloading path of the experiment. It provides not only the possibility to combine room-temperature and high-temperature pressure calibration within a single experiment, but also proved to be a useful tool to speed up testing of new multianvil assemblies and configurations, e.g. in gasketing, without the need to go to a synchrotron facility. Three commonly used octahedron sizes, 10, 14 and 18 mm (edge length) have been tested. The accuracy of the pressure calibration was cross-checked by simultaneous synchroton measurements and showed good agreement.

Other improvements in the design and assembly of 'on-line' and 'off-line' multianvil experiments are presented. Utilization of the polymer-derived ceramic (PDC) technique in addition to the oxide ceramics traditionally used for multianvil experiments, such as MgO, ZrO_2 and Alumina, proved to be particularly useful. Polysilsesquioxane resin can be effectively used as an active binder to fabricate ZrO_2 insulator tubes and MgO-octahedra with firing temperatures as low as 900°C, without compromising mechanical properties. Pyrolysis of Polyborosilazane yields an amorphous Si/B/C/N-ceramic which shows excellent performance as a highly heat-resistant and thermally insulating material for X-ray beam windows. We also show first results on our attempts to fabricate high-ohmic precursor-based TiC-SiC heaters, that are directly painted on the ZrO_2 insulator tubes.

Finally, a new concept for quick, accurate, and 'fool-save' gasket assembly without the requirement to fix the gaskets on the tungsten carbide cubes, is presented.

[1] Schwarz, M. R. "Multianvil calibration and education: A four probe method to measure the entire force-versus-pressure curve in a single run - performed as an interdisciplinary lab-course for students." *Journal of Physics: Conference Series,* **2010***, 215*, 012193

HPCAT: an integrated high pressure synchrotron facility

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The high-pressure collaborative access team (HPCAT) was established to advance multidisciplinary high-pressure science and technology using synchrotron radiation at sector 16 of the Advanced Photon Source (APS). An array of x-ray diffraction, spectroscopy and inelastic scattering techniques has been integrated with high pressure and high/low temperature devices at four simultaneously operating beamlines. The HPCAT synchrotron probes characterize materials structure and dynamics over a wide range of time and length scale. The HPCAT program has been enabling myriad scientific breakthroughs in high-pressure physics, chemistry, materials science, and Earth and planetary sciences. This talk will highlight recent progresses in high-pressure synchrotron techniques at HPCAT.

Direct observation of a pressure-induced precursor in silicon

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Silicon displays a phase transition from the tetrahedrally coordinated cubic diamond structure (α-phase) to the octahedrally coordinated tetragonal β-Sn structure (β-phase) at around 13 GPa accompanied by a large volume collapse of about 22% and an abrupt electric resistivity jump of more than 5 orders of magnitude. The α−β phase transition in silicon has long been considered a prototypical strongly first-order transition with no indication of precursors. Here, we report the first experimental evidence of a precursor lattice in silicon over a wide pressure range, comprising two active groups of silicon atoms that collectively occupy the lattice sites of the β-phase and dynamically co-exist with the host lattice of the α-phase. We use the single crystal diffraction technique coupled with a diamond anvil cell for obtaining electron density distributions of silicon at high pressures, which provides the quantitative description of the embryonic lattice (precursor) in the host lattice. These results, supported by the first-principles calculations, provide insights for understanding the origin and mechanism of the α−β phase transition.

Dense sulfur rich Fe-S melt: The evolution and structure of the core with Fe-S composition

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At the early stage of the differentiated planetary bodies, a metallic component separated from a silicate component and formed a metallic core at the center of bodies. The early cores must be molten due to the several kinds of heat sources (e.g. impact, release of potential energy, and radioactive decay of short-lived isotopes such as ^{26}Al and ^{60}Fe). Then, cooling the core, it is divided into liquid and solid phases. Sulfur is considered to be the major light element in the cores because iron sulfide (FeS) has been found in many meteorites. If the core composition is Fe and S, the core consists of liquid Fe-S and solid Fe or FeS depending on the amount of sulfur in the core.

We compared the density of liquid Fe-S with that of coexisting solid Fe or FeS at high pressure using each equations of state. The results show that that of liquid Fe-S is predictably smaller than that of solid Fe at Fe-rich side whereas liquid Fe-S is denser than solid FeS at S-rich side except only much near FeS. This indicates that, if the core composition is located around the eutectic composition, the core may have multi layered structure because the eutectic composition shifts with increasing pressure.

Cation distribution in Fe^{3+} bearing (Mg,Fe)SiO_3 perovskite determined using single-crystal X-ray refinements and the synchrotron Mössbauer spectroscopy

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The Earth's lower mantle constitutes about half of the Earth by volume; hence it is important to understanding the Earth's bulk chemistry, structure and dynamics. Magnesium silicate perovskite is the dominant phase in the Earth's lower mantle, and it is well known that incorporation of iron has a strong effect on its crystal structure and physical properties. However, the crystal chemistry of iron-bearing silicate perovskite is so far not well constrained. We synthesized a single-crystal of $Mg_{0.95(2)}Fe_{0.06(1)}Si_{1.00(2)}O_3$ perovskite, which we used to investigate its crystal structure and the oxidation state and site occupancy of iron. We synthesized the material at 26 GPa and 2073 K from oxidized starting materials using a multi-anvil press. We determined the composition using the electron microprobe, we refined the crystal structure using single-crystal X-ray diffraction data, and we determined the valence state of iron using the synchrotron Mössbauer source (SMS) at the European Synchrotron Radiation Facility (ESRF). Single-crystal refinements indicate that iron occupies only the A-site, while Mössbauer spectroscopy measurements show the presence of Fe^{3+} ($Fe^{3+}/\Sigma Fe \sim 20\%$). These data enable an accurate determination of the cation distribution in the structure, including the mechanism of charge balance. SMS data collected during compression of the sample to ~ 70 GPa shows an increase in Fe^{2+} quadrupole splitting, consistent with a high-spin to intermediate-spin transition of Fe^{2+}, and no change in the Fe^{3+} hyperfine parameters, consistent with no spin transition of Fe^{3+}.

From a thermodynamic point of view to see the preservation mechanism of coesite during plate exhumation

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In light of the plate deep-subduction fast-exhumation hypothesis, the formation and preservation process itself of coesite UHP metamorphic product in the Earth’s surface should be a non-equilibrium thermodynamic process. However, up to date, the plate deep-subduction fast-exhumation hypothesis has been not self-consistently described this process by the equilibrium thermodynamic phase boundary, such as Mirwald and Massonne 1980 straight line equilibrium boundary. During the retrograde phase transformation from coesite → quartz (corresponding to the process of plate from the upper mantle back to the Earth’ s surface), below the equilibrium phase boundary line there exists only an alpha-SiO_2 stable region. Therefore, only the way of great increment of plate exhumation rate to achieve the quenching critical rate of phase transition could realize exhumation-save-coesite, but it is extremely difficult to achieve.

If by use of the non-equilibrium thermodynamic phase-transition to describe the retrograde transformation process of the coesite → quartz, there appears a metastable coesite region in the lower pressure and temperature zone resulting from the non-hydrostatic shearing role. In metastable coesite region during the retrograde phase-transition process, the resultant product with ultrahigh-pressure metamorphism can have alpha-SiO_2, amorphous SiO_2, coesite with a certain disorder, and crystalline coesite, but the formation of crystalline coesite is very unlikely. Therefore, whether the equilibrium thermodynamic or non-equilibrium thermodynamic mode, the possibility of exhumation save the crystalline coesite is very small.

Crystalline coesite found in the continental crust might be formed by non-subduction-exhumation mechanism. Small-scale local thermodynamic non-equilibrium and quasi-equilibrium region existing in the continental crust, under the roles of local impact, shearing, other dynamic factors, and the local high pressure and so on, without a plate deep-subduction, could occur the prograde phase-transition process from the quartz → crystalline coesite.

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Behaviors of Ammonia borane at High Pressure and Elevated Temperature before Decomposition

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Abstract:

Ammonia borane is determined to be a promising hydrogen storage material by its high (19.6 wt%) hydrogen density and mild hydrogen desorption condition. We conducted *in situ* Raman study of ammonia borane using Diamond Anvil Cell (DAC) and external heating at high pressure up to 15GPa and elevated temperature up to 373K. We found several pressure induced phase transitions shifted to higher pressures at elevated temperature compared to those at room temperature. Additional analysis on the temperature dependence of Raman peaks indicate a possible evidence that ammonia borane could start decomposing at around 330K. We have tested a few liquids as pressure medium for ammonia borane and no reaction between sample and pressure medium was observed during our experiment. No obvious difference was found from the Raman spectra with and without pressure medium at room temperature, which indicates the ammonia borane is soft enough to reach nearly hydrostatic condition without pressure medium at room temperature.

Stability and melting of Fe_3C at high pressure and temperature by in situ X-ray diffraction

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The Earth's core is regarded as an Fe-Ni alloy but the density of the core is lower than that of pure Fe at pressures and temperatures corresponding to the core conditions. Therefore, the Earth's core is supposed to contain light elements and carbon is one of the candidates of the light elements to explain the density deficit of the Earth's core. The recent studies on melting of Fe_3C were reported by Nakajima et al. (2009) and Lord et al. (2009). Nakajima et al. (2009) reported the melting temperature of Fe_3C up to around 30 GPa based on textual observations, the chemical analysis of the quenched run products and in situ X-ray diffraction experiments using a Kawai-type multi anvil apparatus. Lord et al. (2009) reported melting temperatures of Fe_3C up to 70 GPa, which was determined by the temperature plateau during increasing laser power using a laser-heated diamond anvil cell. They suggested $Fe+Fe_7C_3$ was stable as a subsolidus phase. There are obvious discrepancies between the melting curves of Fe_3C reported by Nakajima et al. (2009) and Lord et al. (2009). In this study, the melting temperatures of Fe_3C and a subsolidus phase relation were determined based on in situ X-ray diffraction experiments. This study aims to reveal the stability filed of Fe_3C and uncertainty of the melting temperature of Fe_3C and to discuss the behaviors of carbon in the Earth's core.

We have performed experiments using a diamond anvil cell combined in situ X-ray diffraction experiment at BL10XU beamline, SPring-8 synchrotron facility. An NaCl powder and a rhenium foil were used for the insulator and gasket, respectively. Melting of the sample was determined by disappearance of the X-ray diffraction peaks as described in previous works (e.g, Campbell et al., 2007; Morard et al., 2008; Kamada et al., 2010).

We determined the melting relation of Fe_3C up to 145 GPa by in situ X-ray diffraction experiments. The solidus temperature of Fe_3C is close to Nakajima et al. (2009) up to 30 GPa but becomes close to that reported by Lord et al. (2009) at higher pressure conditions. The present experiments revealed that Fe_3C was stable as a subsolidus phase at least up to 145 GPa and 3800 K. This indicates that Fe_3C is a potential candidate of the Earth's inner core although we need further studies at the inner core conditions.

Behavior of Niobium and Yttrium in Aqueous Fluid At High Pressures and Temperatures

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Constraining thermodynamically the mass transfer of the REE (rare earth-) and HFSE (high field strength) elements from subducted oceanic crust and metasediments to the mantle wedge is fundamental towards interpreting fluid release and partial melting in subduction zones and/or the mantle wedge and crust above subduction zones. HFSE-bearing Rutile has been proposed as an important host for HFSEs (Nb, Ta, Zr, Hf) in eclogites, where rutile is hypothesized to retain these HFSE in slab material upon partial melting and/or fluid release, thereby causing the observed characteristic depletion of HFSE in arc magmas. Several studies have evaluated the hypothesis of compatible host phase(s) in the subducted slab by performing experiments at subduction zone P-T conditions. Most experimental studies quantified the solubility of rutile in aqueous fluid, either by the mass-loss technique or visual estimation, and used Ti as a proxy for the HFSE, where the latter were not measured directly. Published experimental data have been consistent in demonstrating that rutile solubility in aqueous fluid varies with fluid composition, P and T; however, the data are inconsistent with respect to Ti solubility values, which vary by up to three orders of magnitude. Further, these recovery type experimental studies are often complicated by the difficulties/impossibility of retrieving the fluid.

Here we use an in situ x-ray fluorescence (XRF) technique that can measure directly the concentration of Nb and Y in aqueous fluid without having to recover the fluid or solid sample. The solid phase is rutile as the host of Nb. Our study focuses on the effects of P, T in combination with aqueous fluid salinity, on the partitioning of Nb between rutile and fluid. Synchrotron XRF at the HPCAT 16-IDD undulator beamline at the APS is used to measure in situ the concentration of Nb in the fluid. A hydrothermal diamond anvil cell is used to induce P and T in the range of 1 to 5 GPa and 300-500 C. Gold serves as an in situ pressure standard via XRD. We will present and discuss the new experimental partitioning results, which have important implications for the ability of aqueous fluid to scavenge and transport the HFSE and REE in arc environments.

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Experimental novaculite deformation: diffraction data, microstructures, EPSC models and IR- and Raman spectra

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We studied the deformation behavior of novaculite, (fine-grained polycrystalline quartz), experimentally using in-situ X-ray diffraction and electron-backscatter diffraction (EBSD) analyses and theoretically by elastic plastic self consistent modeling (EPSC). The combination of these techniques was chosen to obtain a better understanding of grain-to-grain interactions in deforming samples and to enhance the interpretation of experimental results.

Samples were deformed at 2.5 GPa and up to ~1000 °C in the D-DIA apparatus installed at the X17B2 beamline at the National Synchrotron Light Source (NSLS). In-situ synchrotron X-ray diffraction was used to monitor the sample stress during controlled deformation and to observe the strain behavior of the individual lattice reflections of quartz. Lattice reflections (101), (110) and (112) were fit with the software Plot85. The lattice strain was calculated as $\Delta d/d_0$ where d_0 is the intitial lattice spacing. The macroscopic sample strain was determined by sample radiographs. The strains provide information about how individual grains or grain populations react to stress depending on their orientation within the aggregate.

Independently, microstructures of deformed samples were studied using EBSD analyses. EPSC models were calculated to theoretically determine macroscopic sample stresses and to match measured rheological sample properties with simulations by activating basal, prism and/or pyramidal slip systems in the crystal structure. IR- and Raman spectroscopy were used for sample characterization and for water content analysis. The software BEARTEX was utilized to analyze preferred orientation in deformed polycrystalline quartz.

We discuss results, which are consistent across the series of experiments we have conducted so far. The elastic slope is reproducible and a temperature dependence of individual lattice strains and yield strength can be observed.

The COMPRES/GSECARS Gas Loading System at the APS

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Use of noble gas pressure media (Ne and He) in diamond anvil experiments greatly improves the hydrostatic sample environment and improves the data quality. Use of such gases requires a high-pressure gas loading system. The COMPES Infrastructure Development Committee funded the capital equipment costs (~$85,000) of a gas-loading system at the APS. GSECARS contributed the design and construction effort to build the system. The system began operation in February 2008 and has been running with minimal downtime since then. This system is available for use by any member of the COMPRES community, regardless of whether they are performing experiments at GSECARS, at another APS sector, at another synchrotron, or in their home laboratory.

GSECARS provides the support (training and supervision) for any users who come to the APS to use the system for experiments on the GSECARS sector. COMPRES is providing support for 50% of a post-doc, Dr. Sergey Tkachev, who resides at the APS. Sergey is responsible for providing a mail-in service, loading cells that are sent to us by users who will not be performing experiments at the APS. He is also responsible for hands-on assistance of users of other APS beamlines with loading their cells. Sergey began in this post-doc position in June, 2010.

We will present statistics on the operation of the system, including the mail-in service, for the past year.

Deformation experiments of (Mg,Fe)SiO$_3$-Perovskite at lower mantle conditions

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The Earth's lower mantle is thought to be consist-ing of ~77 vol% (Mg,Fe)SiO$_3$-perovskite (Pv), ~16 vol% ferropericlase and ~8 vol% CaSiO$_3$-perovskite in pyrolite model. In the uppermost and lowermost lower mantle, presence of seismic anisotropy has been reported. The anisotropy may be produced by lattice preferred orientation of the majority phase Pv. Thus, knowledge of slip system of Pv is important for understanding of rheology in the lower mantle.

Shear deformation experiments at the lower mantle conditions were conducted using Kawai-type apparatus triaxtial deformation (KATD) at Tokyo Institute of Technology. Dense aggregates of synthetic (Mg,Fe)SiO$_3$-orthopyroxene (Mg# = 0.97) was prepared as starting material using Kawai-type multi anvil apparatus at 2 GPa and 1273 K. The shear deformation assembly consists of a Cr$_2$O$_3$-doped MgO pressure medium and a cylindrical LaCrO$_3$ furnace. Temperature was estimated from power-temperature relationship in a similar cell assembly. Pt foil is placed at the back of Al$_2$O$_3$ piston to assist sideslip of the piston, and Ni foil placed at the center of sample is used as strain marker. Undeformed runs, which were not deliberately deformed by the differential rams, were quenched after phase transitions and relaxation of stress at 25 GPa and 1873 K. In deformation runs, samples were deformed by moving differential rams in the guide blocks each 75 μm (total 150 μm) during 1 h after the annealing process (for 30 min or 1 h). Lattice preferred orientation (LPO) of sample was determined using 2D-Xray diffraction patterns of sample. The 2D-Xray diffraction patterns were measured using Imaging-plate in the SPring-8 (BL04B1). Analysis of LPO was conducted by the software "ReciPro".

Strains of undeformed and deformed recovered samples measured from rotation of strain marker were ~0.4 ± 1 and ~1.3 ± 1, respectively. Then total stains of samples during deformation process in deformation runs are calculated to be ~0.9 ± 1. Average strain rates of sample are 3×10^{-4} s^{-1}. LPO of perovskite in undeformed experiments is similar to that of uniaxial deformation experiment. This LPO is thought to be formed by phase transition to perovskite with stress relaxation. LPO of perovskite in shear de-formation experiments developed greatly in comparison with that at undeformed experiment. The results suggest that main slip system of Pv at 25 GPa and 1873 K is [100](001).

Pearl @ ISIS

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Pearl is the high pressure powder diffractometer at the ISIS neutron spallation source in the UK. As such, it has been specifically designed for *in situ* studies of materials at high pressure, primarily using the Paris-Edinburgh press.

Until a decade ago, high-pressure neutron diffraction studies were confined to the rather limited pressure range 0-3 GPa. The development of the Paris-Edinburgh pressure cell for use at ISIS has now extended this range by almost a full order of magnitude up to ~25 GPa using sintered diamond anvils and experiments are now routinely carried out up to ~10 GPa using ISIS supplied tungsten carbide anvils.

Pearl is used for a wide range of science from investigations into hydrogen bonding of small molecular compounds to changes in electric and magnetic properties of ceramics, as well as geological relevant studies of earth forming minerals and the ices possibly found in the ice moons of the gas giants in our solar system.

There have been a number of recent developments and indeed the whole instrument has just undergone a major upgrade. In this presentation I will give a brief description of these developments together with a general overview of this extremely useful facility and its capabilities.

Recent advances in high pressure neutron scattering at the Spallation Neutron Source at Oak Ridge National Laboratory

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There have been significant improvements in the operation of the high pressure diffractometer, SNAP, at the Spallation Neutron Source over the past two years. This talk will highlight the current capacities which include low temperature systems, high temperature systems, and the introduction of new pressure cell technology that is based on supported diamond anvils and, with advances in software, is particularly suited for powder diffraction. Specific examples of our recent research will focus on high pressure transitions in hydrogen bonded systems such as methane and CO_2 hydrate. The high pressure hexagonal phase of methane hydrate is studied to determine the nature of the hydrate cage loading, this provides detailed experimental data that will lead to better intermolecular potentials for methane – methane interactions, particularly when methane molecules are in close contact and strongly repelling. The high pressure structural systematics of carbon dioxide hydrate is reported. While the structural transformation sequence of most hydrates progress from sI (or sII) to the hexagonal form then to a flied ice structure, CO_2 hydrate is an example of a system that skips the hexagonal phase and transforms directly into the filled ice structure. Finally examples of using SNAP to study disorder in amorphous systems will be given. Particularly amorphous vapor co-deposits of water, known as amorphous solid water, and clathrate forming molecules such as CO_2, and the structural response of these systems to increased pressure at low temperature.

Mechanical properties of a newly developed non-magnetic ultrafine-grained WC-Ni hardmetal.

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WC–Co based hardmetals, for example “TF05, TF06 and F08” produced by Fuji Die Co.,Ltd., have been used for the components of high pressure apparatus for long years, because of their high mechanical properties such as compressive strength, Young’s modulus and hardness. Recently, necessity of submicro(0.6 μm)-grained WC–10 mass% Ni hardmetal “MF10” where Ni is used instead of Co as the binder metal has increased for magnetic property measurements and neutron experiments under high pressure. Because, it is non-magnetic material at ambient condition and most radio active Ni created by neutron radiation have much shorter half-life compared with radio active Co. However, the mechanical properties of WC–Ni hardmetals “MF10” were considerably lower than those of WC-Co hardmetals.

Hitherto, the following have been reported; (1) hardness and compressive strength of hardmetals are both increased by decreasing WC grain size and/or by reducing binder content [1], (2) hardness and transverse-rupture strength of WC–10 mass% Ni hardmetals become close to those of WC–Co hardmetals by decreasing WC grain size [2], and (3) microstructural defects reducing mechanical properties tend to remain with lowering Ni contents.

Based on the above knowledge, the authors investigated uniaxial compressive strength of ultrafine(below 0.5μm)-grained WC–Ni hardmetals with low Ni contents less than 8 mass%, which were prepared by strictly controlling the preparation conditions so that no microstructural defects remain. Submicro-grained WC–Ni hardmetal “MF10” and three WC-Co hardmetals “TF05, TF06, and F08” were also used for comparison. Cylindrical specimens of φ8 × h16 mm with the parallelism of 2/1000 mm were uniaxially compressed to measure uniaxial compressive stress-strain curve and strength by “250 tonf DIA-type press” produced by Kobe Steel, Ltd. The axial and tangential strains were measured by four strain gages pasted on the side surface. The strain rate was controlled in the range of $1\times10^{-5} – 1\times10^{-6}$ s^{-1}. Young’s modulus, Vickers hardness and transverse-rupture strength were also measured.

It was found that the ultimate compressive strength of the ultrafine-grained WC–Ni hardmetals were over 7.5 GPa which were higher by about 1.5 GPa and 0.5 GPa respectively, compared with those of “MF10” and “F08”. The Young’s modulus, hardness and transverse-rupture strength were more than 605 GPa, 2150 HV and 3.2 GPa, respectively. These all values were high enough to use the hardmetal for components of high pressure apparatus. This newly developed hardmetal is now commercially available under the name of “TMS05”.

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Equation of state of silicate melts with densified intermediate-range structures at the pressure condition of the Earth's deep upper mantle

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The compression behavior of fully densified SiO_2 glass has been measured up to 9 GPa at room temperature by using a diamond-anvil cell with a mixture of methanol-ethanol as a pressure medium. Optical-microscope observations clarify that there is remarkable agreement between the volume data on compression and decompression and therefore the glass behaves in an elastic manner. The compression curve can be expressed accurately enough by a Birch-Murnaghan equation of state with the zero-pressure bulk modulus $K_0 = 60.2$ GPa and its pressure derivative $K_0' = 4$ (fixed). Extrapolating this equation of state to higher pressures, the density of the glass merges with that of ordinary glass (i.e., former ordinary glass compressed to high pressures) at about 13 GPa. X-ray diffraction and Raman scattering measurements show that the first sharp diffraction peak and the main Raman band of the glass also merge with those of ordinary glass at the similar pressure range. These results suggest that the compaction of interstitial voids dominates in compression mechanisms of densified SiO_2 glass, similar to the case for ordinary SiO_2 glass. Together with available information from the literature, it is presumed that the permanent densification of SiO_2 glass takes place between 9 and 13 GPa at room temperature while it takes place at lower pressures at high temperatures. The difference in the pressure range can be explained by the kinetic barrier, considering the permanent densification as phase transformation relevant to the change in intermediate-range structure caused by rebonding the network of SiO_4 tetrahedral units. The experimental results of densified SiO_2 glass suggest that, at the pressure range of the deep upper mantle (including the transition zone), SiO_2 melt behaves as a single phase having a densified network structure. In this study, a simple and easy-to-use equation of state of silicate melts in the deep upper mantle has been constructed based on the assumption that SiO_2 component in silicate melts is in its densified state (or phase) at the pressure range of the deep upper mantle. The equation of state proposed in this study is consistent with all the available density data of silicate melts with various chemical compositions at pressures between 8 and 22 GPa. There is a considerable discrepancy among the equations of state proposed so far. It is attributable to the problem that the compression behavior of multiple states (or phases) of silicate melts has been described with a single equation of state in previous studies.

Thermal equation of state of stishovite and $CaSiO_3$ perovskite up mid-lower mantle condition

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In situ *P-V-T* measurements of stishovite and $CaSiO_3$ perovskite were performed up to mid-lower mantle condition by using a sintered-diamond multi-anvil apparatus (SDMA) with synchrotron radiations at SPring-8. The lattice constants of these mineral phases and pressure marker are analyzed by using measured XRD profiles. And then thermal equations of state of these mineral phases were precisely determined on the basis of Mie-Grüneisen-Debye model with Morse-Rydberg (Vinet) and third-order Burch-Murnaghan equations for reference isotherms. Analyses using MGD model yielded parameters at ambient pressure, such as bulk modulus and its pressure derivative, Grüneisen parameter, Debye temperature. Using these parameters, the new equation of state of stishovite and $CaSiO_3$ give the thermoelastic properties such as density, adiabatic bulk modulus, thermal expansivity, and isobaric heat capacity with no extrapolation to mid-lower mantle conditions. Because of more precise SDMA experimental data comparing with previous measurements by DAC, current equation of state of stishovite and $CaSiO_3$ perovskite prides a tighter constraint on the elastic properties at high pressure and high temperature conditions. Along the geotherm, the present equation of state of stishovite yields lower density and larger adiabatic modulus compared to those obtained by extrapolating previous equation of state determined at upper mantle conditions, the density of the Ca-Pv agrees with that of the PREM within 2% through the lower mantle and the bulk modulus shows smaller pressure dependence along the geotherm. Thus determined equation of state and thermoelastic properties of stishovite and $CaSiO_3$ perovskite at lower mantle conditions are important to discuss the fate of subducted Si, Ca-rich rocks, such as MORB, pelagic sediments and continental crust, in the light of seismological observations.

Pressure-Induced Phase Transitions in $AlPO_4$ from First Principles

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Introduction: $AlPO_4$ and SiO_2 are isoelectronic to each other and mutually serving as models to understand the complex high-pressure polymorphic behaviors. To date, in-situ diamond anvil cell (DAC) experiments have found $AlPO_4$ have a pressure-induced phase transition sequence discovered as berlinte → $CrVO_4$ → m-$CaCl_2$ phase [1]. Recently, three more phases (moganite, $AlVO_4$ and the $P2_1$ /c phase) were synthesized by our quench experiments [2,3]. In parallel to SiO_2, there is a ferroelastic phase transition in $AlPO_4$ between stishovite-like and m-$CaCl_2$ phases once expected, but not confirmed [1]. In this study, in order to investigate the pressure-induced phase transitions in $AlPO_4$, we performed first-principles density functional theory (DFT) [4] for seven candidate structures, and furthermore, we extended to investigate the vibrational and thermodynamic properties of high-pressure phases.

Calculation: The first-principles calculations were conducted using DFT with plane-wave pseudopotential methods and generalized gradient approximation (GGA). Phonon frequencies were calculated with density functional perturbation theory (DFPT) [5]. Thermodynamic properties were analyzed with quasi-harmonic approximation (QHA) [6]. Quantum-ESPRESSO package was used for all these calculations.

Results and Discussion: Based on DFT calculations at 0 K, we revealed a new pressure induced phase transition sequence for $AlPO_4$ up to 100 GPa as follow: berlinite → moganite → $AlVO_4$ → $P2_1$ /c → $CrVO_4$ → stishovite-like → m-$CaCl_2$ phase, with the corresponding transition pressures 4.1, 5.0, 7.0, 7.3, 31.9 and 46.4 GPa respectively. The transition sequence is in good agreement with available experiments, with transition pressures slightly overestimated due to GGA approximation we used. There is a ferroelastic phase transition we confirmed between stishovite-like and m-$CaCl_2$ phases by witnessing a pressure-induced phenomena of symmetry-breaking spontaneous strain. The newly-established transition sequence would serve as an model case because it is the most detailed one among all the berlinite-type ABO_4 compounds to date. All the mixed-coordinated phases studied here exceptionally lack counterparts in SiO_2, phases of $CrVO_4$, $AlVO_4$ and the $P2_1$/c phase. With phonon frequencies calculated by DFPT, we explored lattice dynamic properties of these high-pressure phases. With QHA analysis, we provided a set of thermodynamic parameters and phase boundaries among those phases.

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Deformation of two-phase composites: implications for flow properties of the lower mantle

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We examine flow properties and deformation-induced fabric evolution in two-phase composites. Deformation experiments were performed on sintered polycrystalline rock samples synthesized from the disproportionation reaction of $CaMgGeO_4$ (olivine) → $CaGeO_3$ (perovskite) + MgO at 12 GPa, 1573 K for 4 h. The samples contain 28 vol% MgO, and are an excellent analog material for the lower mantle. In order to examine effects of the softer phase (i.e., MgO) on flow properties, a pure $CaGeO_3$ (GePv) sample was also deformed under identical conditions. Elastic constants of GePv were calculated using first-principles with the generalized gradient corrections (GGC) technique. Flow properties of MgO are available from previous studies [1]. Scanning electron microscopy showed that the average grain size was about 1 micron, with the perovskite phase (GePv) forming a load-bearing framework (LBF) texture. The samples were deformed in the D-DIA at pressures from 4 to 12 GPa, temperatures from 600 to 1200 K, and strain rates from 1 to $3x10^{-5}$ s^{-1}. Relative stress levels in GePv and MgO in the composite sample are in general agreement with numerical simulations up to 3% total bulk strain [2]. Differential stresses obtained from lattice distortion in GePv are 2-3 times greater than those in MgO and for bulk strains up to ~20%, GePv remains a LBF texture. Deformation experiments to higher bulk strains are planned. Another analog, a mixture of San Carlos olivine and Fe-S, was examined by high pressure X-ray tomography. The strength contrast of two phases is similar to that of perovskite and ferropericlase. The initial texture was also of the LBF type, with isolated “weak” Fe-S grains surrounded by “strong” a silicate framework. The development of deformation fabric was continuously monitored by tomographic imaging under high pressures up to 7 GPa and temperatures to 1000 K to a maximum shear strain of 1300%. Under these conditions, the flow stress of olivine is estimated to be 10 – 100 times that of Fe-S, which occupied about 10% of the total volume. A strong shape preferred orientation began to develop in the sample at shear strains above 300%, resulting in a highly anisotropic interconnected weak layer (IWL) texture. Applications of these results to dynamics of the lower mantle will be discussed.

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Large-volume high pressure facilities at GSECARS

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We report current status and new developments of the large-volume high-pressure facilities at GSECARS. The 2.5 MN press at the bending magnet beamline 13-BMD is now equipped with the following capabilities: (1) A DIA apparatus for melt density and ultrasonic velocity measurements up to 10 GPa and 2000 K. (2) A deformation DIA (D-DIA) for rheological property and brittle failure studies with maximum pressures and temperatures up to 18 GPa and 1800 K, respectively. Stress-strain relations are measured by lattice distortions and sample length change during deformation, using x-ray diffraction and imaging, respectively, and brittle failure is monitored by a state-of-the-art acoustic emission (AE) detection and analysis system. (3) A high-pressure tomography apparatus for 3-D imaging of non-crystalline materials and multi-phase composites with a spatial resolution of 2-3 microns. Ultrasonic velocity measurements can be conducted simultaneously with tomographic imaging. (4) Two T-10 (Kawai-type) multi-anvil modules for 20+ GPa and 2000+ K. Both white and monochromatic radiation can be used. A large Si (1 1 1) double-bounce monochromator allows switching between the two modes within a minute. An upgrade is underway to improve position accuracies for both the press and detector support. The 10 MN press at the insertion device beamline 13-IDD currently runs in two modes. (1) A T-25 (Kawai-type) module, capable of 27 GPa and 2700 K, is currently used for a range of experiments, from phase relations to falling sphere viscometry, to ultrasonic velocities of both solids and liquids. (2) A large D-DIA (DDIA-30) has been commissioned which allows deformation experiments on large samples or to higher pressures. DDIA-30 has also been tested for double-stage high pressure generation. Using sintered diamond as second stage anvils, pressures up to ~40 GPa have been generated at 2000 K. The insertion device beamline is currently undergoing upgrade with canted undulators. With the new monochromator, we will be able to conduct angle-dispersive diffraction up to 65 keV. We also expect a substantial increase in beamtime after the upgrade. In addition to these systems at the GSECARS sector, we have also set up a Paris-Edinburgh cell (PEC) at beamline 16-BM-B, in collaboration with scientists at HPCAT. The 2.3 MN PEC has been used extensively for liquid structure studies, with an emphasis on magmatic properties. Key scientific breakthroughs will be highlighted. We thank Guoyin Shen, Changyong Park, Yoshio Kono (HPCAT), Toru Inoue, Akihiro Yamada (GRC, Ehime University) for collaboration and support for the development and operation of PEC at 16-BM-B and NSF supports for the above technical and scientific developments. The PEC beamline and the DDIA-30 module development were partially supported by the COMPRES Infrastructural Development Projects.

ORIGIN OF THE EARTH'S LOW VELOCITY ZONE

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The association of low shear wave velocity and low viscosity with partial melting has been part of the lore since the early days of plate tectonic theory. The strong rigid lithosphere defined the plates that were lubricated from below by the plastically weak aesthenosphere. The seismic verification came from the ubiquitous low velocity zone (at least under oceanic plates). The implicit assertions of this model are 1) velocity and viscosity are both affected by small amounts of partial melting, 2) shear modulus is more affected than bulk modulus, and 3) the top of the low velocity zone marks the onset of melting and the bottom of the low velocity zone is thus defined by the disappearance of partial melting and the increase of both viscosity and velocity. However, the effect of a liquid phase dispersed in a solid matrix on elastic velocity should roughly depend on the volume per cent of the liquid.

However, the dynamic interaction of a solid and liquid that are in thermodynamic equilibrium can dramatically soften the sound velocities. The salient properties are the volume change on melting and the characteristic time of melting. In this context, the seismic wave perturbs the equilibrium between the solid and the liquid forcing the increase of one at the expense of the other.

Here we present data on KLB1 samples undergoing partial melting at LVZ conditions. Effective elastic moduli are derived from sinusoidal stress variations at seismic frequencies. The results support the dynamic melting model outline above.

Multi-anvil High Pressure Facilities at the National Synchrotron Light Source

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This has again been an exciting year at the multianvil facility at the NSLS. The 'B' portion of the superconducting wiggler beam (X17) supports three hutches, X17B1, X17B2, and X17B3. The X17B2 houses the multianvil facility and X17B3 houses a portion of the diamond anvil cell program. Together B2 and B3 are in use 2/3 of the time and can be run simultaneously. Thus the multianvil system is available 2/3 of the time that the NSLS is operating.

The B2 hutch has a white beam and a monochromatic beam that is generated by a single bounce monochromator. Thus it is possible to operate two high pressure stations on the floor, each receiving beam. The shielding of the mono station has been completed, so that these two stations can now operate simultaneously. We have successfully tested the T-cup and the DT-cup in the side station and all are functioning properly. The T-cup is a 6-8 styled module with 10mm second stage anvils. We have achieved 29GPa pressure in this system with 2 mm truncations in the past. The DT-cup is a modified T-cup with continuous deformation capabilities. These two guideblocks are compressed by a V-8 Paris-Edinburgh 500 ton press frame. The DT-cup has two additional 50 ton jacks to drive the differential rams. The main station currently operates with a 1000 tonne press, enabling multiple guide blocks.

Experimental programs include quantitative rheological studies, Q and modulus dependence on P, T and frequency, kinetic studies, thermal diffusivity, LPO with P and T, and many other properties. We can now routinely measure diffraction and images during a sinusoidal variation in stress, pressure, or temperature.

Hydroxyl content in omphacite at high pressure and implications for water transportation in subduction zones

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Unlike that of oceanic subduction zones where dense hydrous magnesium minerals (DHMMs) are the water-carrying phases, the presences of DHMMs are prohibited at high pressures and temperatures in magnesium- poor continental subduction zones. Therefore, it is of great interest that which mineral would be the major water carrier in subducting continental crust and how much water could be transported via continental subduction. The H_2O storage capacity of omphacitic clinopyroxene is thus an issue of significant importance for predicting and modeling Earth's deep water cycle through subduction. Toward a better understanding of hydrogen solubility in omphacite at high pressure and temperature conditions and their implications for water transportation in subduction zone, we performed multi-anvil experiments at pressure of 4-14 GPa and temperature of 750-1000 ^{o}C in a bulk composition of jadeite (Jd) +8 wt. % H_2O +5 wt. % SiO_2, diopside (Di) +8 wt. % H_2O+5 wt. % SiO_2, $Jd_{20}Di_{80}$+8 wt. % H_2O+5 wt. % SiO_2 and $Jd_{40}Di_{60}$+8 wt. % H_2O+5 wt. % SiO_2, respectively. The hydrogen solubility of run products was measured with SIMS. The water solubility of coexisted minerals is buffered by presence of three phases. Jadeite, coesite/stishovite and melt/fluid are found in the starting material of Jd + 8 wt. % H_2O; clinopyroxene, and olivine/garnet coexisted with melt/fluid in the starting material of Di + 8 wt. % H_2O+5 wt. % SiO_2 and $Jd_{20}Di_{80}$+8 wt. % H_2O+5 wt. % SiO_2; whereas clinopyroxene, garnet and melt/fluid occur in the $Jd_{40}Di_{60}$+8 wt. % H_2O+5 wt. % SiO_2 system. Water storage capacity of clinopyroxene is related to its complex chemistry. In pure jadeite and diopside, the storage capacity decreases with pressure, from 268±44 ppm for jadeite and 270±19 ppm for diopside at 4 GPa towards a minimum of 85±20 ppm for jadeite and 146±42 ppm for diopside at 10 GPa. The water solubility of coesite coexisted with jadeite shows almost constant value of 27-34 ppm from 4-7 GPa and increase drastically to 340 ppm at 10 GPa where coesite is replaced by stishovite. The storage capacity of olivine in the Di +8 wt. % H_2O+5 wt. % SiO_2 system prevails that of coexisted pure diopside by a factor of 1.5. Up to 2000 ppm water is incorporated into clinopyroxene with starting material of $Jd_{20}Di_{80}$+8 wt. % H_2O+5 wt. % SiO_2 and $Jd_{40}Di_{60}$+8 wt. % H_2O+5 wt. % SiO_2 at 4 GPa and decrease to c. 1200 ppm at 14 GPa, no measurement was attempted for the coexisting olivine/garnet because of their small grain size in those systems. Our results imply that clinopyroxene is an important water reservoir for the subducted slab at shallower conditions where it has the ompacitic composition. However, the water storage capacity of clinopyroxene decreases magnificently when its composition approaching that of pure jadeite with increasing pressure in continental subduction zones.

Dehydration Embrittlement in Serpentinized Peridotite is Composition Dependent

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Double seismic zones (DSZ) which have two parallel planes of seismicity separated by 15-40 km are a global feature of subduction zones in the 70-250 km depth range (Brudzinski et al., 2007). While the physical mechanism of lower plane seismicity is still controversial, the leading hypotheses currently are associated with dehydration of antigorite serpentine within the subducting mantle plate (Peacock, 2001; Jung et al., 2004). In this study, we are conducting high-pressure (1-3GPa), high-temperature (720-750˚C), deformation experiments on specimens of varying compositions of serpentine plus peridotite in our 4GPa Modified Griggs apparatus. Using samples composed of interlayered thin discs of antigorite and harzburgite, we find that dehydration embrittlement occurs down to less than ~30 vol % antigorite. Surprisingly, in many cases a macroscopic fault cuts through the olivine-rich layers but the fault is absent (or at least invisible) in the antigorite layers, even in the SEM. Interlayered mineralogy was impractical at lower antigorite fractions so we prepared homogeneous mixtures of powders of the two rock types (35-75 µm grain-size) and "warm" pressed them to a coherent solid with little porosity. Subsequent deformation of these specimens extended the faulting regime to as little as ~8 vol % antigorite. In summary, we find that faulting occurs during dehydration of a wide range of serpentinized peridotite compositions but not during dehydration of nearly pure serpentinite nor nearly pure peridotite. We suggest that the lack of faulting in nearly pure peridotite is a consequence of too little H_2O production and the lack of faulting in nearly pure serpentine is due to extensive crystal plasticity, as proposed previously by Chernak and Hirth (2007). We would expect that slower strain rates may change the compositions at which faulting stops but are unlikely to change the fact that there are cutoffs at both ends of the spectrum.

Likely consequences of this behavior in subduction zones are: (i) aseismic behavior of the plate interface that becomes serpentinized as fluid escapes from the slab due to dehydration below; (ii) serpentine diapirs frequently found in forearc basins; (iii) earthquake generation from moderately serpentinized lithosphere during dehydration.

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Microscopic Model of Bulk Modulus for Polar Covalent Crystals

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In the past several decades, numerous efforts have been made to establish direct links between the macroscopic mechanical properties and the microscopic parameters of solid. Some important progresses have been achieved. For example, microscopic hardness models have been constructed in terms of atomic-scale parameters and used extensively experimentally as well as in superhard materials design. However, microscopic models for other mechanical properties are still in the primary state. Here we report a microscopic model of bulk modulus based on atomic-scale parameters which include the bond length (d), a crystal-structure-dependent parameter (ρ) for bond density, and the effective bonded valence electron number (n_{ij}) we proposed previously. The estimated bulk moduli from this model are in good agreement with experimental values for various polar covalent crystals. Our current work sheds lights on the nature of bulk modulus, provides useful clues for design of low compressibility materials, and is applicable to complex solids such as minerals of geophysical importance.

Correlation between structure and electron spin state of earth's interiors under high pressure (degeneracy, hybridization, high-low spin transition, Jahn-Teller transition)

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To elucidate the correlation between structure transitions and spin state is one of the crucial problems for understanding the geophysical properties of earth interiors under high pressure. High-pressure studies of iron bearing spinels attract extensive attention in order to understand strong electronic correlation such as the charge transfer, electron hopping, electron high-low spin transition, Jahn-Teller distortion and charge disproponation in the lower mantle or subduction zone.

Jahn-Teller effect A cubic-to-tetragonal transition under pressure was confirmed by Jahn-Teller effect of $^{IV}Fe^{2+}$ $(3d^6)$ in the tetrahedral site of Fe_2TiO_4 and $FeCr_2O_4$. Tetragonal phase is formed by the degeneracy of e_g orbital of Fe^{2+} ion. Their c/a ratios are c/a<1 due to d_{x2-y2} orbital of the electronic tetrahedral configuration. However, Fe_3O_4 (I), Fe_2SiO_4 (N), do not have a tetragonal polymorph because of no $^{IV}Fe^{2+}$ ion.

Spin transition HS-to-LS transition starts from 15.6GPa in Fe_3O_4, 19.6 GPa in Fe_2TiO_4, 17GPa in Fe_2SiO_4. The transition is more capable due to $^{VI}Fe^{2+}$ in the octahedral site, than Fe ion in the 4-fold or 8-fold coordinated site. The extremely shortened octahedral bonds result in a distortion of 8-fold cation site. This structure change is accelerated at higher pressure due to HS-LS transition of Fe^{2+} in the 8-fold coordination site. This transition is induced by the 20% shrinkage of ionic radius of $^{VI}Fe^{2+}$at the low sin state.

Post spinel transition The transition to orthorhombic post-spinel structure with *Cmcm* has been confirmed in the whole compositional range of $Fe_{3-x}Ti_xO_4$ and $Fe_{3-x}Cr_xO_4$. There are two octahedral cation sites: M1 and M2 in the orthorhombic phase. Fe^{2+} and Ti^{4+} are disordered in the M2 site. At pressures above 53 GPa, the Fe_2TiO_4 structure transforms to *Pmma.* This structure change results from the order-disorder transition.

Rhombohedral Fe_2SiO_4 The spin transition exerts an influence to Fe_2SiO_4 spinel structure and triggers two distinct curves of the lattice constant in the spinel phase. The reversible structure transition from cubic to rhombohedral phase (R-Fe2SiO4) was observed at about 45 GPa. R- Fe_2SiO_4 structure has $R\bar{3}m$ space group symmetry with Z=6, D=5.867 g/cm3 about 5% larger than D=5.584 g/cm3 of spinel at 39 GPa. R- Fe_2SiO_4 structure has two alternated octahedral layers of Fe1 and Fe2 perpendicular to the *c* axis. Laser heating experiment at 1500 K has confirmed the decomposition from R- Fe_2SiO_4 to two oxides of FeO and SiO_2.

Model simulation for laser heating temperature measurement

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Abstract Code simulations of temperature from perfect laser heated hot spot without any optical aberration were carried out. The simulation variables includes hot spot (hot spot shape, spot size, and peak temperature), fitting techniques (Blackbody, and Greybody approximation), and fitting parameters (fitting wavelength range, and start fitting wavelength). Gaussian, Lorentzian, flat-top, and double-Gaussian hotspot were chosen as the hotspot shape. Simulation results indicate the normalized spatially scrambled average measured temperature is dependant on spot shape and peak temperature of hotspot, fitting technique, and fitting parameter, but not dependant on spot size given that all radiation is collected and no spatial effect. For the sharp hot spot, the fitting normalized temperature converges to both extreme wavelength ends, while, for flat-top hot spot, the normalized fitting temperature yields a closer temperature to the peak temperature. For an experimental laser heating data acquisition and analysis, the optical aberration is inevitable, and wide open slit with vertical summation were simulated, and suggested at beamline 12.2.2. Advanced Light Source, Lawrence Berkeley National Lab, which yield reasonable results.

In this presentation, 2D temperature map of laser heating spot by a single wavelength intensity conversion is discussed.

Phase relations and melt compositions in hydrous pyrolite system

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Many studies suggest that there could be significant amount of water in the deep Earth, especially in the mantle transition zone (Inoue et al., 1995; Kohlstedt et al., 1996). Water plays an important role in understanding the geodynamics process in the mantle, such as the melting behavior (Inoue, 1994; Kawamoto and Holloway, 1997), phase transformation (Ohtani and Litasov, 2006), and so on. To clarify the effect of water for the mantle peridotite, a series experiments were carried out in pressure range from 12 to 20 GPa and temperatures from 1400°C to 1600°C in pyrolite+water system, by using a Kawai-type multianvil apparatus (ORANGE 1000) with 3mm TEL second stage WC anvils in Ehime University. Some different compositions (Mg/ (Mg+Fe)) of olivine were used for pressure calibration under high temperature, and 2cpx-geothermometer was used to estimate the temperature gradient in the run charge. The starting samples were the mixtures of "pyrolite - MgO" glass, MgO and Mg (OH) 2, thus the water contents were adjusted by the Mg(OH)2/MgO ratio. The samples were sealed by AuPd capsule to prevent the loss of water. The recovered samples were polished, and then the phases were identified by micro-Raman spectroscopy, the textures were observed by BEI, and the chemical compositions were measured by SEM-EDS system. Under hydrous condition, the phase boundary of α/β moved to lower pressure, while the appearance of γ moved to higher pressure, and both phase boundaries became much sharper, compared with dry condition. Garnet was the liquidus phase in the whole P-T range.

Compositions of partial melts formed by low degree of melting (<20%) at 12-20 GPa had high CaO/Al2O3 ratio (8-15), and magnesium-rich with (Mg+Fe)/Si ratio larger than 2, which is beyond komatiite composition and much different from that from dry system. The water contents of hydrous melt were determined by mass balance calculations. It shows that the water content of melt is more than 10wt% at 410km depth, even along the hot geotherm, which is much larger than the critical value reported by Matsukage et al. (2005) and Sakamaki et al. (2006). That means the hydrous melt may not be stable atop the 410km depth, and should migrate upwards into the mantle. Thus the low velocity zone atop the 410 km depth reported by Revenaugh and Sipkin (1994) may not come from the melting of the mantle minerals.

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Diamondoids under high pressure

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Diamondoid molecules are ultra-stable, saturated hydrocarbons consisting of fused carbon cages superimposing on the diamond lattice, originally found in petroleum. These hydrocarbons, especially the larger higher diamondoids, have been of great interest in recent years due to their potential role in nanotechnology, electronics, and medical technologies. However, the large number of possible intermediates, reaction pathways, and complex reaction kinetics make the synthesis of higher diamondoids extremely difficult. Here we report our efforts in investigating the [121] tetramantane at high pressure by combining x-ray diffraction (XRD) and Raman spectroscopy up to 20 GPa. XRD shows that the [121] tetramantane in a monoclinic structure starts to transform into a high pressure phase at approximately 6 GPa and the transition was almost complete at around 20 GPa. In addition, the high pressure phase displays a large metastability field upon decompression. Raman studies also confirmed this phase transition and the metastability of the high pressure phase based on the peak splitting and pressure shifts, as well as changes in the relative intensity of the most intense peaks. Our study may have implications for developing alternative approaches to synthesize higher diamondoids.

Spin transition of iron in deep-mantle ferromagnesite

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Ferromagnesite (Mg,Fe)CO_3 has been proposed to be a potential candidate mineral for deep-carbon storage in the mantle (e.g., Isshiki et al. 2004). Recent experimental and theoretical studies have indicated that Fe-containing magnesite undergoes a spin transition at approximately 40-45 GPa. Since ferromagnesite is the only common deep-mantle carbonate mineral known to undergo the spin-pairing transition (e.g., Mattila et al. 2007; Lavina et al. 2009), there is of great interest to understand the effects of the spin transition on the behavior of this deep mantle carbonate, including its thermodynamic, elastic, and vibrational properties. According to previous studies, the observed effects of the transition on the properties of ferropericlase (e.g., Lin et al. 2005, 2007), including elasticity, electrical and thermal conductivity, and deformation, have dramatically affected our understanding of the seismic, geochemical, and geomagnetic observations of the lower mantle. Experimental studies on the effects of the spin transition on the properties of ferromagnesite can thus provide new insight into understanding the properties of the lower-mantle iron-containing carbonates. Thus, we have studied vibrational, elastic, and thermodynamic properties of ferromagnesite across the spin transition using Raman and X-ray diffraction techniques in a diamond anvil cell up to 80 GPa. Our combined results show that the spin transition dramatically affects the physical properties of the ferromagnesite. With recent studies and thermodynamic modeling constraint, we aim to address how spin transitions of iron in lower-mantle minerals affect our understanding on the physics and chemistry of the deep Earth.

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Introduction of SSRF and the development of high pressure techniques at SSRF

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Shanghai Synchrotron Radiation Facility (SSRF) is the first 3rd generation synchrotron facility constructed in China. SSRF was open for users since May 2009. In this talk, a brief introduction of SSRF and user work will be given firstly. In china, high pressure related user community is growing fast. But up to now, there is no dedicated high pressure beamline at 3rd generation synchrotron radiation facility in China. We have developed some instruments at micro-beam beamline BL15U1 of SSRF for high pressure μ-XRD experiment and off-line ruby system for the pressure measurement. A dedicated high pressure beamline is in plan at 2nd phase of SSRF. I will also introduce the development of HP related techniques at SSRF and conception design of HP-dedicated beamline.

Compressibility and thermal expansion studies of hydrous Fo100 wadsleyite

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Compressibility and thermal expansion studies of hydrous Fo_{100} wadsleyite were conducted at Sector 13 of the Advanced Photon Source, Argonne National Laboratory (2010), and Mineral Physics and Crystallography Lab, University of Colorado at Boulder (2011), respectively. High-pressure single-crystal synchrotron diffraction was carried out in a diamond anvil cell with neon as pressure medium up to 60 GPa. A third-order Birch-Murnaghan equation of state (EoS) fitting gives the results of V_0 = 542.7(8) Å^3, K_{T0} = 137(5) GPa, K' = 4.6(3), while 2nd-order B-M EoS fitting indicate: V_0 = 542.7(8) Å^3, K_T = 147(2) GPa. High-temperature single-crystal X-ray diffraction measurements from 300 K to 767 indicate thermal expansion coefficient of 38.4(3) × 10^{-6} K^{-1} for the hydrous sample and 28.1(8) x 10^{-6} $K^{-1}$for the dehydrated one. XRD intensity measurements were also conducted to determine internal structure at high temperatures and after dehydration. The most significant changes during dehydration are systematic decreases in M2 – O1 and M3 – O1 bond lengths, which decrease by 3 % and 2.5 %, respectively after dehydration. While the length changes of the other M – O bonds are no more than 1 %, consistent with the hydration mechanism being protonation of O1. Both results support the conclusion that hydration would significantly increase both compressibility and thermal expansion of silicates in transition zone. In addition, *c* axis has larger thermal expansion and compressibility than *a* and *b* axes, indicating that wadsleyite is most 'flexible' in *c* direction.

Elasticity of sodium disilicate glass at high pressures and high temperatures

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Studying the structures and physical properties of silicate melts under extreme conditions is essential for understanding the dynamics of the earth's interior. Sodium disilicate ($Na_2Si_2O_5$) glass is a good model material for studying basalt magma since they have similar ratios of non-bridging oxygen to tetrahedrally coordinated cations (NBO/T). Synthesized sodium disilicate glass was loaded into a Paris-Edinburgh (PE) cell and pressed up to 4.5GPa and heated up to 1000°C at the HPCAT beamline 16-BM-B of the Advanced Photon Source. Ultrasonic elastic wave travel times of the amorphous sample were collected using the pulse reflection method with a 10° Y-cut $LiNbO_3$ transducer. X-ray radiographs were used to determine the elastic wave travel distance. Experimental results show similar pressure dependences of elastic wave velocities at room temperature for sodium disilicate comparing with pure SiO_2 from previous studies. Under high pressure (3GPa), both the longitudinal wave velocity (Vp) and the transverse wave velocity (Vs) for sodium disilicate glass remain nearly constant with increasing temperature before the glass transition temperature (~700°C at ambient pressure). An 8.7% drop in Vp and a 11.9% drop in Vs were observed above the glass transition temperature. Structure information of the glass sample has recently been collected and the results will be presented. We acknowledge the supports of COMPRES on facility development, of GSECARS for the Paris-Edinburgh press system, and of HPCAT for elastic wave velocity measurement developments.

Elasticity, local site-symmetry, and atomic dynamics of iron-bearing enstatite at high pressure

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Abstract

Enstatite is an abundant, yet complex, mineral in Earth's upper mantle. The high pressure behavior of synthetic powdered ^{57}Fe-enriched enstatite $(Mg_{0.980}Fe_{0.020(5)})(Mg_{0.760}Fe_{0.240})Si_2O_6$ has been explored by X-ray diffraction (XRD) and nuclear resonance inelastic X-ray scattering (NRIXS) at room temperature using neon as a pressure medium. The NRIXS measurements with in-situ XRD were performed at sector 3-IDB of the Advanced Photon Source. From the raw NRIXS data, quantities such as the mean-square displacement of the iron atoms and average force constant of the iron sub-lattice in enstatite are directly determined as a function of density and pressure.

In a separate investigation, we compressed the *Pbca*-structured enstatite sample in a neon pressure medium from ambient pressure to 20 GPa in fine pressure increments at the Advanced Light Source. One structural transition between 10.1 and 12.1 GPa has been identified from the XRD data, in agreement with recent synchrotron Mössbauer spectroscopy results (Zhang et al., Am. Min. 2010) from the same sample as that investigated here. A third-order Birch-Murnaghan equation of state fitting gives a K_{T0}=109±5 GPa and K_{T0}'=10±2 for the *Pbca* phase. We use the equation of state determined from XRD and Debye sound velocity determined from NRIXS to compute the compressional (V_P) and shear (V_S) wave velocities at different pressures. In the pressure range of the structural transition (~10 GPa to 12 GPa), we find that V_S decreases by ~6%. The decrease of V_S can be explained by a soft-mode observed in the DOS. We discuss the implications of these results to the "X-discontinuity" and the low velocity zone in Earth's upper mantle.

The Elastic Properties of Fine-Grained Polycrystalline/Cryptocrystalline and Amorphous Samples by Brillouin Scattering

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5. COMPRES

Near-perfect single crystals are often used for elasticity measurements when available, though in nature, fine-grained polycrystalline and/or cryptocrystalline and even amorphous minerals compose a large part of it. Therefore, the relationship between the properties of single-crystal and polycrystalline/cryptocrystalline and even amorphous samples is of fundamental importance in applying lab measurement results to real Earth. In this study we have investigated the sound velocities and elastic properties of fine-grained polycrystalline/cryptocrystalline and even amorphous materials using Brillouin scattering.

Our initial experiments were performed on silica minerals, among the most abundant minerals in the crust, and MgO, one of the major components of Earth's lower mantle. Three silica minerals (wood opal, hyalite and agate) and three MgO samples have been measured. The grain size of the silica minerals is under one micron in average, whereas that of the MgO is around 5 to 10 microns. Samples were ground and polished into plate-like shapes (~30 microns thick) for 90° geometry symmetric Brillouin scattering at room conditions. Our initial results indicate that the velocities of agate, hyalite and wood opal are nearly 9%, 10% and 20% lower than the velocities obtained from single-crystal quartz measurements using Voigt-Reuss-Hill averaging respectively. Several features, such as the grain size, the inclusions and the internal structures, may contribute to the velocities drops. However, Brillouin spectra from MgO samples show broad peaks that are roughly symmetric in shape. Peak shifts and velocities of the fine-grained MgO are close to the velocities obtained from single-crystal MgO measurements. The sharpness of the peaks increases with the decreasing grain size. Some of our observations may be a result of grain boundary effects.

Compositional effect on the pressure-induced phase transition of natural orthoenstatite

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High-pressure Raman spectroscopy has been employed to investigate possible compositional effects on the newly-discovered high-pressure phase transition by Mg-rich orthoenstatite. Three natural orthoenstatite samples were used in this study, with chemical compositions determined by EMPA: pure Mg orthoenstatite from Zabargad Island, Egypt, Al-free Fe-bearing orthoenstatite from Tanzania, and Al-rich Fe-bearing orthoenstatite from Kilbourne Hole. The transition is characterized by the split of 660-680 cm^{-1} doublet in the Raman spectrum into a triplet, and also by the corresponding change of peak intensities. This is caused by the lowered symmetry of Si-O tetrahedral chains in the high-pressure phase, as indicated by structural refinement from single-crystal X-ray diffraction results. Preliminary results show that the presence of several percent Al and Fe could increase and decrease the phase transition pressure by over 3GPa.

Petrofabric and strength of SiO_2 near the quartz-coesite phase boundary

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Quartz and coesite constitute an important portion of deep subducted continental upper crust. However, the rheology and petrofabric of quartz and coesite are not well constrained under HP/UHP metamorphic conditions both in laboratory and in nature. We report here first deformation microstructure and lattice preferred orientations of garnet, omphacite, interstitial coesite, quartz and rutile in the UHP eclogites from the Yangkou Bay, Sulu UHP terrane, as well as fabric development of quartz and coesite in shear experiments at P/T conditions near the quartz-coesite phase boundary. Our results show: 1) Garnet and coesite develop weak to random fabric while omphacite, quartz and rutile develop pronounced fabrics in deformed natural eclogites; 2) Quartz develops fabrics in responding to a dominant **c**-slip with increasing shear strain under HP/UHP conditions; 3) Coesite develops fabrics in responding to a dominant [100](010) slip with increasing shear strain under UHP conditions; 4) The relative strength of major constituent minerals in continental deep subduction zones are quartz < omphacite < coesite. These results are consistent with the rheology determined in the laboratory for quartz, jadeite/omphacite and coesite. We propose that jadeite/omphacite dominates the rheology and seismic anisotropy of deeply subducted continental crust.

Phase transformation of harzburtite at mantle transition zone conditions and its geophysical implications

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Subducted materials play an important role in affecting chemical composition and structure of the mantle transition zone (e.g. [1]). Harzburgite is generally accepted as an important part of subducting slab, overlaid by a layer of basalt. Seismic tomography studies have detected widespread fast anomalies in the mantle transition zone (MTZ) and lower mantle around the circum-Pacific and the Mediterranean region (e.g. Kárason and van der Hilst, 2000; Huang and Zhao, 2006; Fukao et al., 2009; Li and van der Hilst, 2010). These anomalies have been interpreted as stagnant oceanic lithosphere based on the assumption that high velocities correspond to low temperatures. However, experimental studies on the phase transformation of harzburgite have been very lmited. There is also very little information on density and velocity of harzburgite as a function of depth. In this study, we conducted high-temperature and high-pressure experiments, conducted using a 1000 ton Kawai type multianvil apparatus at GPMR, on a natural harzburgite at 14.1-24.2 GPa and 1200-1400 ^{o}C. At 1400 ^{o}C, harzburgite transforms to wadsleyite + garnet by 19 GPa and further disproportionate to an assembly of ringwoodite + garnet + stishovite above 20 GPa. Some amounts of akimotoite were produced at still higher pressures (22-23 GPa), and finally perovskite and magnesiowustite were found to coexist with garnet at 24.2 GPa. Density and velocity profiles of subducted harzburgite are calculated as a function of depth along a subducting slab geotherm [1], which is 1000 ^{o}C at 410 km and 1200 ^{o}C at 660 km, based on the mineral proportions and compositions determined in this study. The mantle is assumed to be pyrolitic whose density profile along normal mantle geotherm (about 1400 ^{o}C at 410 km and 1600 ^{o}C at 660 km) was adopted for comparison [2]. Harzburgite is about 0.2 g/cm^3 and 0.1 g/cm^3 denser than pyrolite at 380-410 km and 520-660 km depths respectively, about 0.05-0.1 g/cm^3 less dense than the surrounding mantle in the upper part of MTZ, and ca. 0.2 g/cm^3 less dense at about 660-680 Km. Seismic velocities of harzburgite are about 3-7 % and 4-10 % higher than that of PREM and AK135 for Vp and Vs, respectively. The discrepancies in sound velocities between seismic observations and experimental results of pyrolite [3] in the lower part of mantle transition zone can be explained by the stagnation of ca.30 vol% harzburgite materials.

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Recent activities of high pressure and high temperature studies at 3ID using nuclear resonant x-ray scattering

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Nuclear resonant scattering (NRS) using synchrotron radiation, including synchrotron Mossbauer spectroscopy and nuclear resonant inelastic x-ray scattering techniques, has been successfully applied for high pressure research field over the last 10 years. Hyperfine interactions, magnetic ordering information, as well as thermal dynamic properties of materials can be uniquely revealed by this technique. Recent activities of NRS under high pressure and high temperature at 3ID on Fe-contained and Eu-contained systems will be shown. Further development of the beamline capabilities including fast chopper enabled synchrotron Mossbauer spectroscopy and micron-resolution Mossbauer microscopy will be presented.

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An improved Griggs-type apparatus for mechanical testing at high pressure and temperature

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A new deformation apparatus has been developed to study the mechanical properties of materials at high pressure and temperature. It is based on the modified Griggs design introduced by H.W. Green II and detailed stress analyses of the load frame and the pressure vessel. The improvements include a servo-controlled 30000-Psi large-oil-reservoir pressure intensifier designed to generate up to 5 GPa pressure on sample assembly, a servo-controlled 10-ton integrated high-resolution ball screw drive to deform materials under true constant strain or constant stress modes, and new and optimized electronics and software for improved pressure, stress and temperature stability, apparatus safety, and user-friendly operation. We have calibrated the sample assembly pressure (calculated based on oil pressure) at room temperature against the phase transition of Bi metal from phase I to phase II (2.55 GPa), and at high temperatures against the phase transitions of quartz to coesite (1200℃ and 3.21GPa), albite to jadeite + quartz (800℃ and 2.15GPa), and calcite to aragonite (800℃, 2.07GPa and 600℃, 1.42GPa). The uncertainty of pressure was estimated to be 35MPa at a 3 GPa pressure and decreases with pressure. The sample temperature variation was estimated to be within 25℃ by the two-pyroxene thermometer through the solid-reaction between orthopyroxene and clinopyroxene at 1200 ℃ and 2.5 GPa. The temperature gradient is smaller along the sample longitudinal direction compared to along the radial direction. About 20 successful hydrostatic and deformation experiments have been performed at confining pressures up to 3.5 GPa and temperatures up to 1200 ℃.

Equation of state and ultrasonic measurement of $MgSiO_3$ akimotoite at high pressures

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Akimotoite, the ilmenite form of $MgSiO_3$ magnesium silicate, is stable at pressures between ~18 and 25 GPa and at relative low temperatures in the $MgSiO_3$ system. Experimental measurements on the thermo-elastic properties of akimotoite at high pressures and high temperatures in the mantle are so far limited except for recent theoretical simulations. Particularly the shear elastic constant and its pressure and temperature dependences have not yet been directly measured by high pressure experiments. Here we carried out ultrasonic measurement on $MgSiO_3$ akimotoite polycrystalline with in situ X-ray diffraction at high pressures up to 25 GPa and temperatures to 700 K. Bulk and shear modulus and their pressure and temperature dependences at high pressures are determined as a result: $\boldsymbol{K}_{S0}$=217(3) GPa, $\boldsymbol{K'}_{S0}$=4.4(2), $\partial K_S/\partial T$= -0.017(6) GPa/K, $\boldsymbol{G}_{S0}$=132.4(5), $\boldsymbol{G'}_{S0}$=1.56(3), $\partial G_S/\partial T$= -0.017(6) GPa/K. The bulk and shear modulus are similar with previous Brillouin measurement (*Weidner and Ito, 1985*). The seismic velocities of harzburgite with presence of akimotoite in the transition zone are evaluated based on present data and phase relations.