Inelastic X-ray Spectrometer at the Advanced Photon Source for High Pressure Experiments*

A. Alatas, E. E. Alp, B. Leu, H. Yavas, and J. Zhao

Advanced Photon Source, Argonne National laboratory, Argonne, IL 60439

Measurement of physical properties of solids or liquids under pressure and/or at high temperatures is important in understanding the behavior of materials. It is clear that any new methodology development will contribute greatly in solving some of the puzzles related to the structure of earth and other terrestrial planets, whether this interest originates from a condensed matter physics, mineral physics, or geology point of view. One of the methods for achieving high pressure in a very small area is the use of a diamond anvill cell (DAC). Combining a DAC with an inelastic x-ray scattering (IXS) spectrometer that has small beam size and improved flux would allow measurement of phonon dispersions and consequently determine the sound velocity and elastic properties of the given system [1,2].

In order to achieve very small beam size in the order of microns, we have used both toroidal and KB mirrors in tandem. This combination has an advantage of focusing the entire undulator beam more efficiently [3]. While toroidal mirror can be used to focus the beam in the order of 100 μm, it allows KB mirror to accept full beam that is available for the photon hungry IXS experiments. This combination can provide beam sizes below 30 μm. We have used both toroidal and KB mirrors in tandem at sector 3 for the IXS spectrometer. 16.5(V) x 19(H) μm^2 FWHM is achieved for spot size with 75% throughput from the KB mirrors.

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The equation of state of FeO + SiO$_2$ in the Earth's lower mantle
Matt Armentrout and Abby Kavner

The lower mantle is comprised mostly of perovskite and magnesiowustite. While both of these phases are rich in magnesium, about ~10% iron is likely present in both phases. Possible reactions between the core and the mantle may generate even more iron-rich material at the base of the lower mantle. At approximately 20% stoichiometric iron content silica becomes stable. Constraining the amount of iron in the lower mantle and its distribution between phases requires measurements of iron's effects on the phase stability, density, and thermal equation of state of lower mantle minerals at the relevant pressure and temperature conditions. We are interested in establishing a consistent equation of state for an assemblage of iron oxide and silicon oxide at the conditions of the lower mantle. An FeO + SiO2 assemblage was synthesized *in situ* from an Fe2SiO4 starting material in the laser heated diamond anvil cell. Density of both phases were measured as a function of pressure and temperature using synchrotron X-ray diffraction in conjunction with laser heating in the diamond anvil cell at pressures and temperatures up to 60 GPa and 2700 K. We use these measurements to calculate equations of state for each of these phases with regard to a published EoS. Our measured equation of state of stishovite relative to that of wustite disagrees with previous equations of state by a significant amount. From this we propose a new EoS of wustite anchored to the EoS of stishovite. We use this well constrained EoS of stishovite and wustite in good thermal contact to predict bulk sound speed and perturbations thereof with respect to temperature and iron content. The temperature dependence of the bulk sound speed for wustite and stishovite have opposite signs. Together, it suggests that the temperature dependence of the bulk sound speed might be characteristic of compositional behavior, with a silica-rich (and therefore stishovite-rich) composition displaying very different wavespeed behavior in a thermal anomaly than Mg/Fe-rich (and therefore oxide-rich) compositions.

Constraints on the Fe-S melt connectivity in mantle silicates

Bagdassarov N.[1] , Schmidt M.W.[2] ,Golabek G. J.[1,3], Solferino G.[2]

[1]*Institute for Geosciences, Goethe University, Frankfurt, nickbagd@geophysik.uni-frankfurt.de*
[2]*Institute for Mineralogy and Petrology, ETH Zürich, max.schmidt@erdw.ethz.ch*
[3] *Institute for Geophysics, ETH Zürich, gregor.golabek@erdw.ethz.ch*

The mechanism which segregates molten Fe-S into metallic cores of planetary bodies is still not fully understood. Due to the high interfacial energy and wetting angle between Fe-S melts and silicate mantle minerals, the continuous percolative flow of such melts cannot be efficient for the core segregation in planetary bodies. A series of percolation experiments has been realised on a partially molten fertile garnet peridotite, employing a centrifuging piston-cylinder. A high temperature garnet-peridotite with Mg# $\sim$ 0.90 composed of 60 vol% olivine, 15 vol% orthopyroxene, 6 vol% clinopyroxene and 19 vol% garnet has been used as a silicate matrix. Peridotite powders with the 100-200 or 20-30 μm grain size were mixed with 5-30 vol% Fe-S of eutectic composition $Fe_{70}S_{30}$. The aggregates were centrifuged at 500-700 g temperatures below and above the melting point of the peridotite. The centrifuge experiments revealed a negligible percolation of Fe-S melts through the unmolten peridotite matrix. Only at T>1260 °C, *i.e.* above the solidus of the peridotite, and starting with 5 vol% of $Fe_{70}S_{30}$ the vertical melt gradient achieved 1-2 vol%/mm. In samples with 15 vol% $Fe_{70}S_{30}$ the vertical separation achieved 2-2.5 vol%/mm after 10 h of centrifuging at 500 *g*. An increase in the degree of partial silicate melting in the peridotite leads to an increase of the Fe-S separation rate from the peridotite matrix. Fe-S contents > 10 vol% cause an increase of the Fe-S melt droplet size and of the effective percolation velocity of Fe-S melt. A threshold dividing fast (>10 cm per year) and slow percolations (<1 mm per year) of Fe-S melt is found around 14-15 vol% of $Fe_{70}S_{30}$. The experimentally determined permeabilities of Fe-S melt in the unmolten peridotite with 7-10 vol% of $Fe_{70}S_{30}$ melt are 10^{-18}-10^{-19} m^2, which is 2-3 orders of magnitude lower than the values calculated previously from static experiments. The presence of the silicate melt increases the segregation velocity of Fe-S melt in a partially molten peridotite by more than one order of magnitude with respect to the unmolten peridotite matrix. This could provide an effective segregation of Fe-S melt in a planetary mantle down to 2.5 vol% of residual Fe-S melt. The extremely slow percolation of Fe-S melt in the absence of the partial silicate melting precludes a scenario of metallic core formation *via* percolation before temperatures allow a substantial partial melting of mantle silicates in planetary bodies.

High pressure behavior of $[HN(CH_2CH_2)_3NH][V_2^{V}V_4^{IV}O_{14}] \cdot H_2O$

Lauren A Borkowski[1], Lars Ehm[2], Karah E. Knope[3], Christopher L. Cahill[3], John B. Parise[1,4]

[1]Department of Chemistry, Stony Brook University, Stony Brook, NY 11778
[2]National Synchrotron Light Source, Brookhaven National Laboratory, Brookhaven, NY 11973
[3]Department of Chemistry, The George Washington University, Washington, DC 20052
[4]Department of Geosciences, Stony Brook University, Stony Brook, NY 11778

Abstract

$[HN(CH_2CH_2)_3NH][V_2^{V}V_4^{IV}O_{14}] \cdot H_2O$ is a layered $V_6O_{14}^{2-}$ material templated by 1,4-diazabicyclo[2.2.2]octane (DABCO) and has been shown to have reversible Li intercalation[1,2] as well as non Curie-Weiss type magnetism[3,4]. The structure consists of a V_6O_{14} layer containing edge-shared V^{IV} square pyramids, which form zig-zag chains that are connected by corner-shared V^V tetrahedra. The protonated DABCO and water molecules reside in the inter-layer space and interact with the layers via H-bonds.

We decided to investigate the effect of hydrostatic pressure on this layered material since preliminary single-crystal studies conducted at GSECARS (Sector 13, APS) indicated the possibility of a volume increase with increasing pressure. Powder diffraction studies were conducted at the National Synchrotron Light Source using the X17c beamline to investigate the effects of pressure on this material. Additionally, single crystal high pressure studies were conducted at ChemMatCars (Sector 15, APS) to investigate the effect of pressure on the DABCO molecules.

The Fe-rich liquidus in the Fe-FeS system from 1 Bar to 14 GPa

Antonio S. Buono, David Walker

Lamont Doherty Earth Observatory, Department of Earth and Environmental Sciences,

Columbia University, Palisades, NY 10964, USA

ABSTRACT:

The composition and evolution of a planetary metallic core can be approximately understood in terms of the behavior with pressure of the eutectic and the liquidus on the Fe-rich side of the Fe-FeS eutectic. New experiments at 6 GPa presented here, along with literature data at 1 bar, 3 GPa, 10 GPa and 14 GPa inform a thermodynamic model for this liquidus from 1 bar to at least 10 GPa. Fe-FeS has a eutectic that becomes more Fe-rich but remains constant in T up to 6 GPa. The 1 bar, 3 GPa, and 6 GPa liquidi all cross each other at a pivot point at 1640±5K and $FeS_{37±0.5}$. This liquid/crystalline metal equilibrium is T-X independent through 6 GPa. Our models of the 1 bar through 10 GPa experimental liquidi show that with increasing P there is an increase in the T separation between the liquidus and the crest of the metastable two-liquid solvus. The solvus crest decreases in T with increasing P and becomes more asymmetric towards the Fe-rich end-member. The model accurately reproduces the liquidus from 1 bar to 10 GPa, as well as reproducing the pivot point and the eutectic values. The 14 GPa experimental liquidus deviates sharply from the lower pressure trends indicating that the model no longer applies at 14 GPa or above.

First results on the deformation of polyphase aggregates at pressures and temperatures of the upper mantle

Bystricky M., Béjina F., Ingrin J.

LMTG, UPS, CNRS, Observatoire Midi-Pyrénées, Université de Toulouse, France

Modelling the solid-state flow of the upper mantle requires a thorough understanding of the rheology of upper mantle rocks 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, we have performed deformation experiments on two-phase aggregates of forsterite and MgO at pressure and temperature conditions of the upper mantle. 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 [1]. Deformation experiments were performed at 3-8 GPa and 1000-1300°C in a D-DIA apparatus coupled with synchrotron X-ray radiation allowing *in situ* stress and strain rate measurements. 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%. The technique permits the measurement of macroscopic strain rates, and of the stress levels sustained by different subpopulations of grains of each phase [2]. Mean stresses experienced by forsterite and by MgO in each aggregate are obtained by elastic-plastic self-consistent (EPSC) modelling of the experimental data. Results on stress partitioning in these polyphase aggregates and on the effect of the presence of a second phase on the rheology of forsterite-rich aggregates will be discussed.

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Developments in GHz-ultrasonics for elasticity studies of geophysical and applied materials: application to diamond

Yun-Yuan Chang[1], Steven D. Jacobsen[1], Chih-Shiue Yan[2], Joseph Lai[2], Russell J. Hemley[2], and Craig R. Bina[1]

Northwestern University, Department of Earth and Planetary Sciences
Geophysical Laboratory, Carnegie Institution of Washington

Science and society alike have long been fascinated with diamond as the hardest known substance. Various forms of diamond are used in applications ranging from machine tooling to synchrotron-light optics, and it remains a critical material in emerging high-pressure science and technology. The quest for larger single crystals and tougher bulk polycrystalline diamond has motivated recent developments in diamond synthesis. Can a better, ultimate diamond material be created? And how can improvements in diamond material properties be established considering the relatively large uncertainties in traditional testing methods on this challenging material? The C_{11} elastic constant of diamond is over 1000 GPa, with typical uncertainties in the range of 10 GPa and higher. Elastic properties of materials are governed by interatomic bonding forces, and diamond exhibits the highest shear modulus of any known material. It follows that atomic-scale defects may influence diamond elasticity. Does the wide variation in reported diamond elastic constants reflect material differences or technique uncertainties? These and other questions are being addressed by developments in GHz-ultrasonic interferometry, a high-precision acoustic technique. Using pulse-modulated ultrasound with carrier frequencies in the 1-2 GHz range, in conjunction with a newly developed sample length-measuring instrument, we baseline future studies of superhard materials by measuring the elastic constants of diamond with accuracies on the order of 0.1-0.3%, sufficient to scrutinize the observed variation in reported values of various forms of diamond. Results on natural type-IA and IIA diamond, along with single-crystal CVD diamond synthesized under varying conditions will be reported. Knowledge of elastic properties of diamond and other superhard materials will illuminate why certain substances are hard, and improve our ability to identify and design them in the future.

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Title
Rapid terrestrial core formation from high-pressure X-ray microtomography

Authors and Affiliations
<u>Bin Chen</u>[1], Dongzhou Zhang[1]; Tony Yu[2]; Yanbin Wang[2]; Jennifer M. Jackson[1]

1. Seismological Laboratory, California Institute of Technology, Pasadena, CA, USA
   (binchen@gps.caltech.edu)
2. Center for Advanced Radiation Sources, The University of Chicago, Chicago, IL, USA

Abstract

The time scales of the terrestrial core formation constrained from the hafnium-tungsten chronometer are within 30 Myr after the Solar System formation. The mechanism for the rapid core formation of proto-planets or planetesimals during their early history remains poorly understood. Several metal-silicate separation scenarios have been proposed to account for the core formation, including percolation of molten iron through solid silicates (e.g. Shannon and Agee, 1998) and sinking of iron-rich diapirs through a viscous silicate mantle by Rayleigh-Taylor instability (e.g. Stevenson, 1981; Samuel and Tackley, 2008). Penetration of dense metallic diapirs prevails upon the percolation of molten iron-rich alloys in quickly and efficiently separating core materials from silicates (Stevenson, 1981; Rubie et al., 2007; Golabek et al., 2008). In this study, we performed high-pressure X-ray microtomography experiments to measure the sinking velocity of iron-sulfur liquid through solid silicates and to observe possible drainage processes under simultaneously high pressure and high temperature in the large volume press at Sector 13 (GSECARS) at the Advanced Photon Source (Argonne National Laboratory). Preliminary results show that at 1 GPa and 1.5 GPa the iron-sulfur liquid sank through the underlying olivine layer at a speed consistent with the core formation time scales. The measured sinking velocities of iron-sulfur liquid through the olivine layer and observed drainage processes support rapid core formation for terrestrial planets or planetesimals.

Exploring Data Collection Strategies Using the Brillouin Scattering Experimental Method – An Error Analysis and Data Accuracy

Jia Chen, Zhen (James) Xu, Jay Bass

Department of Geology, University of Illinois at Urbana-Champaign

Brillouin spectroscopy enables accurate elasticity measurements on small single crystals samples for determination of the single-crystal elastic moduli (Cij's). The strategy for collecting data as a function of crystallographic direction not, however, strictly standardized. Different strategies have been employed depending on the difficulty of collecting data, desire for efficiency, availability of samples, and other factors. In principle, data from any general crystallographic plane of a single crystal is sufficient to characterize the true Cij of an orthorhombic (and perhaps lower symmetry) mineral. However, the results of different previous measurements on single crystals sometimes are spread over a wide range. Some of these discrepancies may be due to the data collection strategies used in different studies. The questions we wished to address are the following: 1) How do the results compare in studies where data were collected in a general plane, versus data collection in several orthogonal principal planes? What compromises (if any) are made by efficient collection of data within a single general plane; 2) How many data points should be collected to secure the accuracy of the results. We collected Brillouin spectra for five orientations of high-quality San Carlos Olivine single crystals. One set of Cij's is calculated based on velocities from propagation directions in the (100), (010), (001) planes. The other two sets of Cij's are calculated from crystallographic (111) direction, and the (111) in an ortho-normal coordinate system, respectively. The CIJs are compared to previous results obtained by both Brillouin scaterring and other acoustic methods. Our goal is to assess the compromises and advantages to each data collection strategy, and to assess the formal errors, accuracy, and uniqueness of each set of Cij's. We also examined two datasets - one with twice as much data than the other – to address the question: How much data is enough?

Geometric and magnetic properties of MPb_{10} and $[MPb_{10}]_2$ clusters (M = Fe, Co, Ni)

Xuan Chen*, Jiuhua Chen
Center for the Study of Matter at Extreme Condition(CeSMEC)
Department of Mechanical and Materials Engineering
Florida International University(FIU)
Miami, FL33199

Abstract:

The geometric properties of the MPb_{10} clusters and their dimmers (M = Fe, Co, Ni) are studied using the density-functional method. The calculated results show that the structure of MPb_{10} with an encapsulated square antiprism geometry is energetically favorable. These clusters could form a stable dimer cluster and retain their structural identity. The most stable structure of the $[MPb_{10}]_2$ dimer is the two MPb_{10} monomers to be bonded at the triangular faces which are facing upside down with respect to each other. The magnetism calculations reveal that the total magnetic moments of the $FePb_{10}$ and $CoPb_{10}$ clusters are $2\mu_B$ and $1\mu_B$, respectively, while the magnetic moment of $NiPb_{10}$ is quenched. Moreover, the dimerization does not lead to the substantial changes of the local magnetic moments of Fe atoms and the magnetic order between Fe and Pb atoms, but can affect the local magnetic moments of Co (or Ni) atoms and the magnetic order between Co (or Ni) and Pb atoms.

* Presenter, also at Nanjing University of Science and Technology

Compressibility of Vitreous Silica by High Pressure X-ray Microtomography

A. N. Clark[a] C.E. Lesher[a] , S.J. Gaudio[a], , Y. Wang[b]

a Department of Geology, University of California, Davis, CA 95616, USA

bGeoSCoilEnvironCARS, Center for Advanced Radiation Sources, Univ. Chicago, Chicago, Il 60637, USA

The high-pressure X-ray tomographic microscope (HPXTM) on the 13-BM-D beamline at the Advanced Photon Source at Argonne National Laboratory is used to investigate *in situ* compressibility of simple system glasses (i.e. Mg_2SiO_4, $MgSiO_3$, SiO_2) as part of a larger study to characterize the volumetric properties (compressibility and thermal expansion) of earth materials (Lesher et al., 2009). To this end, we have measured the (volumetric) compressibility of vitreous SiO_2 up to 8 GPa. Previous experimental studies using Brillouin scattering, ultrasonic, and strain gauge techniques indicate anomalous compressibility between 2-5 GPa (Schroeder et al., 1990; Suito, 1992; El'kin et al., 2002; Tsiok et al, 1998). Molecular dynamic simulations also show anomalous behavior in this pressure range attributed to phase transitions (Trachenko and Dove, 2003) and/or changes in network flexibility (Walker et al., 2007). Our initial results are in good agreement with previous experimental measurements reflecting changes in bulk modulus (K) of ~40 to 25 GPa between 0 to 3 GPa and an increase in K at higher pressures. Given the consistency of results obtained by very different experimental methods, anelastic effects appear to be small. We are exploring the time-dependence of irreversible densification further by *in situ* HPXTM methods and *ex situ* density measurements of vitreous silica recovered from high pressure (e.g., Gaudio et al., 2008).

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Nano-hardness and Young's modulus of nano-polycrystalline diamond

Hélène Couvy[1], Jiuhua Chen[1], Debrupa Lahiri[2], Arvin Agarwal[2], Gautam Sen[3]

[1]CeSMEC, Department of Mechanical and Materials Engineering
[2] Department of Mechanical and Materials Engineering
[3]Department of Earth & Environment
Florida International University, Miami

The sample of nano-polycrystalline diamond (NPD) has been synthesized at the Center for the Study of Matter at Extreme Conditions using a 500t multi-anvil press equipped with a Walker module. A rod of polycrystalline graphite (99.9995%; Alfa Aesar) was used as starting material. The sample was directly inserted into the Re furnace of a 8mm octahedral edge length cell assembly. Tungsten carbide cubes with 3mm truncation edge length were used as anvils. The graphite sample converted directly into diamond at 19 GPa and 2400ºC. The temperature was maintained for 30 seconds. The transparency of the sample demonstrates the formation of a good polycrystalline material. Raman spectroscopy and X-ray diffraction confirm the total conversion into cubic diamond. Scanning probe microscope image shows a maximum grain size of 100nm. The sample has been mounted in epoxy and was finely polished for nano-indentation hardness test using metal-bonding diamond abrasive disks.

In this study we do not measure the hardness of the material using the traditional method (impression hardness) which measures the dimension of the indent impression after indentation. We use depth-sensing nanoindentation. Our indenter used is equipped with piezo controller which monitor dynamically the depth of the indent during the hardness test. This method is considered to be more accurate and more adapted to elastic material. The hardness test has been performed using a Hysitron TriboIndenter. The probe is a diamond Berkovich tip (three sided pyramid) with 100 nm tip radius. NPD hardness and Young's modulus has been compared with commercially available polycrystalline superhard material used in high-pressure devices as anvils: a cubic boron nitrite anvil (cBN) from Linatec (Ukraine) and a sintered polycrystalline diamond anvil (PCD) from Ringwood Superabrasives Pty Ltd (USA). The results show the superior hardness and elastic properties of NPD on cBN and PCD.

Length scales of chemical transport in the lower mantle: Mn, Fe, Co, and Ni diffusion in periclase

Katherine L. Crispin[1] and James A. Van Orman[2]

[1]Department of Geological Sciences, Case Western Reserve University, Cleveland, OH 44106; klc24@case.edu

[2]Department of Geological Sciences, Case Western Reserve University, Cleveland, OH 44106; jav12@case.edu

Periclase is the second most abundant mineral in Earth's lower mantle and is key to understanding chemical exchange mechanisms and diffusive length scales in the lower mantle. There have been several previous studies on transition metal diffusion in periclase but this study is the first to examine multiple transition metals simultaneously in a single crystal with a fixed concentration of cation vacancies. Experiments were performed to determine diffusion rates of Mn, Fe, Co, and Ni in periclase at temperatures between 1473K and 2073K in a piston cylinder apparatus. Both graphite and nickel capsules were used to control the f_{O_2}. The cation vacancy concentration was controlled by doping the periclase crystal with approximately 5000 ppm Al^{3+}. This approach eliminates the influence of additional vacancies being introduced during the experiment due to oxidation of the diffusing transition metal cations. This may have been an issue in early experiments [1] that examined Fe, Co, and Ni in separate experiments with variable atmospheres.

Wuensch and Vasilos [1] found that diffusivity in periclase increases in the order Ni<Co<Fe, contrary to expectations when considering their ionic radii. The same trend is found here, but with significantly smaller differences among the elements. The activation energies are not correlated with ionic radius but are correlated with crystal field stabilization energy, similar to results reported for Cr^{3+} and Ga^{3+} [2]. If Fe, Co and Mn undergo spin transitions within Earth's lower mantle, their crystal field stabilization energies in periclase would increase. This might cause these elements to diffuse more slowly in the deep mantle, with a reversal in their relative diffusivities.

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Event:

2010 COMPRES Annual Meeting

Title:

A High Pressure Study of Fluorapatite up to 60 GPa

Authors:

Joseph Crow[1], P. R. Scott[1], G. Knotts[1], and M. B. Kruger[1]

[1] *Department of Physics, University of Missouri-Kansas City, MO 64110, USA*

Abstract:

Angle dispersive x-ray diffraction through a diamond anvil cell were used to determine phase stability and compressibility of fluorapatite ($Ca_5(PO_4)_3F_2$) up to pressures of approximately 60 GPa. Using beamline X17C at NSLS, it was observed that fluorapatite is structurally stable and does not undergo any phase changes under pressure at room temperature. A Birch-Murnaghan fit to the equation of state data yields a bulk modulus of ~ 98 GPa ± 3 GPa with a pressure derivative of the bulk modulus as 3.5 ± .25. Raman data collected up to 15 GPa supports the x-ray diffraction data, showing no soft modes or phase changes.

Metadata Management for Distributed First Principles Calculations in VLab - A Collaborative Cyber infrastructure for Materials Computation

Pedro R. C. da Silveira, Renata M. Wentzcovitch

Abstract

This paper describes the metadata and metadata management algorithms necessary to handle the concurrent execution of multiple tasks from a single workflow, in a collaborative service oriented architecture environment. Metadata requirements are imposed by the distributed workflow that calculates thermo elastic properties of materials at high pressures and temperatures. The scientific relevance of this workflow is also discussed. We explain the basic metaphor, the receipt, underlying the metadata management. We show the actual java representation of the receipt, and explain how it is converted to XML in order to be transferred between servers, and stored in a database. We also discuss how the collaborative aspect of user activity on running workflows could potentially lead to race conditions, how this affects requirements on metadata, and how these race conditions are precluded. Finally We describe an additional metadata structure, complementary to the receipts, that contains general information about the workflow.

Abstract:

Near "real-time" stress measurements from synchrotron x-ray diffraction in the D-DIA

Nathaniel A. Dixon[1], William B. Durham[1], Kenneth J. Baldwin[2], Michael T. Vaughan[2], Ayako Suzuki[3], Shenghua Mei[3].

[1] Department of Earth, Atmospheric and Planetary Sciences, Massachusetts Institute of Technology
[2] Mineral Physics Institute, Stonybrook University
[3] Department of Geology and Geophysics, University of Minnesota, Twin Cities

Over the past several years, improvements in D-DIA materials and techniques have given a robust high-pressure deformation system with low cell failure rates and excellent measurement resolution. However, one significant impediment to realizing the full potential of the D-DIA has been managing the large amounts of diffraction data produced during a run. These data sets ordinarily require a trained eye and many hours of calculation to turn diffraction patterns into values of pressure and differential stress in deformation experiments. With the introduction of an improved conical slit system at X17B2 a year ago, sharper and significantly less ambiguous diffraction patterns opened the possibility of computer recognition of peak shifts (e.g., with changing deviatoric stress) during an experiment. The new system also tripled the number of active detectors (and thus diffraction patterns) so enormously increased the data volume. Here we present code and procedures that have recently been developed to automatically follow diffraction peaks for many detectors during an ongoing experiment. In operation, the complete data reduction process—from raw diffraction patterns to pressure and differential stress—is reduced to less than 10 minutes, allowing for real-time pressure correction and constant-stress experiments. We finally have the possibility of conducting intelligently guided dynamic experiments wherein the operator has knowledge of the current state of the sample, and can make the best decisions accordingly.

Equation of state and stability of $(Mg_{0.25}Fe_{0.70}Ca_{0.05})SiO_3$ perovskite and post-perovskite to 122 GPa

Susannah Dorfman, Jue Wang, Gregory Finkelstein, Vitali Prakapenka, Thomas Duffy

Extreme Fe-enrichment at the base of the lower mantle has been proposed as an explanation for seismic anomalies such as large low shear velocity provinces (LLSVPs) and ultralow velocity zones (ULVZs). In order to resolve the effect of Fe on the stability and equation of state of the lower mantle's dominant constituent, $(Mg,Fe)SiO_3$ perovskite, we have studied a sample of extremely Fe-rich natural orthopyroxene, $(Mg_{0.25}Fe_{0.70}Ca_{0.05})SiO_3$ (composition determined by microprobe analysis), at lower mantle P-T conditions. The pyroxene starting material was mixed with Au (pressure calibrant and laser absorber) and loaded with NaCl (pressure medium and thermal insulator) in a symmetric diamond anvil cell. X-ray diffraction experiments at pressures up to 122 GPa with *in-situ* laser heating were performed at the GSECARS (13-ID-D) sector of the Advanced Photon Source. Heating to 2000 K at 80 GPa produced a single-phase orthorhombic $GdFeO_3$-type perovskite. Upon decompression, the single-phase perovskite remained stable down to 72 GPa, but heating to 2000 K at 67 GPa produced a mixture of perovskite and oxide phases. This 67-72 GPa transition pressure for the $(Mg_{0.25}Fe_{0.70}Ca_{0.05})SiO_3$ composition adds a new constraint on the dependence of $(Mg,Fe)SiO_3$ perovskite stability on pressure and composition. Upon compression, we observed a two-phase mixture of perovskite and post-perovskite at 93 GPa and higher pressures. This is consistent with previous findings that Fe substitution destabilizes $(Mg,Fe)SiO_3$ perovskite relative to $(Mg,Fe)SiO_3$ post-perovskite (Mao *et al*. 2004, Caracas and Cohen 2005). The bulk modulus of $(Mg_{0.25}Fe_{0.70}Ca_{0.05})SiO_3$ perovskite at 80 GPa (K_{80}) is 500-570 GPa, comparable to values measured for $MgSiO_3$ perovskite (~550 GPa, Lundin *et al*. 2008). However, the volume of the Fe-rich perovskite is 3% greater at 80 GPa than V_{80} for the Mg end-member, corresponding to a 20% density difference. This volume difference is expressed only in the *a* (2% larger than Fe-free perovskite) and *c* (1% larger) lattice parameters. Volumes under compression show no evidence of a discontinuity in the range measured; any magnetic spin transition in the Fe is either gradual or has too weak an effect on volume to be observed.

Thermodynamic Stability of the Pyrope-grossular Garnet Solvus

Wei Du, Dave Walker
Columbia University in the city of New York
Lamont-Doherty Earth Observatory
P.O. Box 1000 61 Route 9W Palisades, NY 10964-8000 USA
weidu@ldeo.columbia.edu

Multi-anvil (MA) techniques were used to study the pressure and temperature stability conditions for pyrope-grossular garnet solid solution and to assess the possible emergence of a stable solvus with increasing pressure. The large positive excess volumes of garnets measured on this join suggest that the solvus crest increases in temperature quickly with pressure. Perhaps high pressure experiment may confirm the existence of the garnet solvus at experimentally accessible temperatures for demonstrating compositional convergence and divergence of 2-phase garnet pairs on the solvus limbs.

At 6 GPa, garnets with two different compositions Py90Gr10 and Py40Gr60 converged completely into one solid solution with a composition Gr35Py65 at 1200C and then 1150C. This implies that the critical temperature for pyrope-grossular garnet at 6 GPa is lower than 1150 °C, which is much lower than the modeling result calculated by Dachs and Geiger (2006).

At 8 GPa, after heating for more than 20 days at 1100 °C and 1200 °C, two phases of garnet were observed to coexist in both convergence and divergence experiments. So the critical temperature for pyrope-grossular garnet at 8 GPa is higher than 1200 °C. XRD results showed that the final equilibrated compositions for the two coexisted garnet phases have not yet been found but that compositional approach to equilibrium compositions is slowly in progress.

2010 COMPRES Annual Meeting Abstract:

Hydrogen self diffusion coefficients for olivine: Implications for point defect chemistry and electrical conductivity

Wyatt L. Du Frane (presenting), James A. Tyburczy, Thomas G. Sharp

Hydrogen diffusivity in mantle minerals is important for understanding point defects and defect dominated processes such as electrical conductivity. In this study deuterium was interdiffused into hydrated San Carlos olivine and enstatite as a traceable species of hydrogen to simulate the process of hydrogen self diffusion. We used the Cameca 6f SIMS at ASU to measure deuterium and hydrogen across samples from each of these experiments, and fit interdiffusion coefficients to the resulting deuterium profiles. For olivine we measure $D_{H, [100]} = 1.35 \times 10^{-4} * e^{(-163 kJ/mol)/(RT)}$ at 2GPa. This value is ~1 order of magnitude lower than the chemical diffusion coefficient for exchange reactions in San Carlos olivine (Kohlstedt and Mackwell, 1998). Activation energies for the chemical diffusion coefficient (145 +/- 15 kJ/mol) and our self diffusion coefficient (163 +/- 20 kJ/mol) are within error of each other. From comparison of these coefficients we calculate small polaron diffusion coefficients, and shed new light on diffusion of metal vacancies. We present an electrical conductivity model based on olivine D-H interdiffusion coefficients for comparison to conflicting measurements of electrical conductivity in hydrous olivine. The magnitude of electrical conductivity predicted by our model is in range of direct measurements; however the activation energy is significantly higher from diffusion coefficient measurements. Additionally, we measure D-H interdiffusion coefficient of non-oriented enstatite as $\sim 3.88 \times 10^{-14}$ m^2/s at 800 °C. This is ~1 order of magnitude lower than D-H interdiffusion coefficients for pure, synthetic enstatite at 800 °C and ~1 order of magnitude higher than chromium-doped synthetic enstatite (Stalder and Behrens, 2006).

Equation of State and B1/B8 Phase Transition in FeO

R. A. Fischer[1,*], A. J. Campbell[1], G. A. Shofner[1], O. Lord[2], P. Dera[3], and V. Prakapenka[3]

[1]Department of Geology, University of Maryland, College Park
[2]Department of Earth Sciences, Bristol University
[3]GSECARS, University of Chicago
(*raf815@umd.edu)

Wüstite, $Fe_{1-x}O$, is an important endmember of (Mg,Fe)O in the Earth's lower mantle, and possibly also a significant component of the core. Therefore the high pressure, high temperature behavior of FeO, including its phase diagram and equation of state, is essential knowledge for understanding the properties and evolution of Earth's deep interior. We performed X-ray diffraction measurements on wüstite+iron mixtures at beamline 13-ID-D of the GSECARS sector of the Advanced Photon Source, using a laser-heated diamond anvil cell to achieve simultaneous high pressures and temperatures (90 to 155 GPa and up to 3100 K). The wüstite in our experiments was mixed with metallic iron to produce stoichiometric FeO at high pressures and temperatures [1].

We have determined the thermal equation of state of FeO to 155 GPa, combining our new P-V-T data with earlier data [1,2]. We find a bulk modulus K_0 = 147 GPa, and whereas prior studies assumed a value of four for the pressure derivative of the bulk modulus, K_0' [1], our higher pressure data require a lower value, K_0' = 3.72. The location of the subsolidus phase boundary between the B1 and B8 structures in FeO has been controversial, but our results are broadly consistent with a recent study showing a positive slope for the transition [2]. Our results for the equation of state for FeO can be compared to the equation of state of hcp-Fe [3] to match the seismologically determined density of the outer core. Assuming a core-mantle boundary temperature of 4000 ± 500 K and a 1-2% density decrease upon melting, the amount of oxygen in the outer core required to match PREM at the core-mantle boundary is 8.0 ± 1.1 weight percent, under the simplifying assumption of a purely Fe-O outer core.

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Abstract

Title

Equation of State and Magnetic Properties of Fe₃C to Multi-Megabar Pressure with Implications for the Earth's Inner Core

Authors and Affiliations

Lili Gao[1, 2]; Bin Chen[1,3]; Przemyslaw K. Dera[4]; Barbara Lavina[4,5]; Vitali B. Prakapenka[4]; Esen E. Alp[2]; Wolfgang Sturhahn[2]; Jiyong Zhao[2]; Yuming Xiao[6]; Jie Li[1,7]

1.Department of Geology, University of Illinois, Urbana, IL, USA
2.Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60561, USA
3.now at Seismological Lab, California Institute of Technology, Pasadena, CA, USA
4.Center for Advanced Radiation Sources, University of Chicago, Chicago, IL, USA
5.now at Department of Geoscience, University of Nevada, Las Vegas, NV 89154, USA
6.HPCAT and Geophysical Laboratory, Carnegie Institution of Washington, Argonne, IL 60439 USA
7.now at Department of Geological Sciences, University of Michigan, Ann Arbor, MI 48109, USA

It has been widely accepted that Earth's inner core is composed of mainly iron-nickel alloy and a small fraction of lighter elements. The nature and composition of the light element however still remain mysterious, yet it is a fundamental issue in understanding the evolution of Earth and the solar system. Carbon is one of the possible light elements in the Earth's core. Wood (1993) proposed Fe₃C as the dominant inner core component rather than iron-nickel alloy based on thermodynamic calculations. However, equations of state of Fe₃C derived from experimental data up to 73 GPa and the EoS from a first-principle calculation indicate that Fe₃C alone is too light to match the density of the inner core. In this study, we carried out single crystal X-ray diffraction measurements on Fe₃C in quasi-hydrostatic neon pressure medium up to 200 GPa, well into the pressures of Earth's core, as well as synchrotron Mössbauer spectra up to 90 GPa. Both data sets suggest a discontinuity at ~ 6 GPa and indicate another discontinuity at ~ 70 GPa. The compression data above 70 GPa suggest a good match between the density of the inner core and that of Fe₃C at corresponding conditions.

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Structural characterization of borohydrides of Na, Mg and Ca at high pressures

Lyci George, Vadym Drozd and Surendra K. Saxena

Department of Mechanical and Materials Engineering, Center for the Study of Matter at Extreme Conditions, Florida International University, Miami, FL 33199, United States

The depletion of fossil fuel and increase in environmental pollution require us to discover new energy sources. Hydrogen stored in the form of hydrides is safe and has high hydrogen density. The transport sector requires not only cheap, safe and reliable way of storing hydrogen but also high storage capacity, fast kinetics and favorable thermodynamics. A usable hydrogen storage candidate, which meets all the mentioned criteria, has yet to be discovered. Light metal hydrides have high storage capacity, but high operation temperature hinders their application. To achieve potentially improved thermodynamics is to form complex hydrides with addition of Al, B etc., to alkali or alkaline earth metal hydrides. The application of pressure on materials can have influence on their properties favoring hydrogen storage. Hydrogen desorption in many complex hydrides occurs above the transition temperature. Therefore, it is important to study the structural properties of the hydride compounds at ambient and high pressure conditions, which can assist in the design of suitable storage materials with desired thermodynamic properties. The high pressure-temperature phase diagram, thermal expansion and compressibility have only been evaluated for a limited number of hydrides so far. This situation serves as a main motivation for studying such properties of a number of technologically important hydrides. This presentation focus on X-ray diffraction and Raman spectroscopy studies of $Ca(BH_4)_2$, $Mg(BH_4)_2$ and $NaBH_4$ at high pressures. Results of these investigations are potential source of information regarding inter-atomic forces and also act as a basis for developing theoretical models. It is found that the highly compressible B-H bonds and the associated bond disordering under pressure is responsible for phase transitions in these brorohydrides. Complex hydrides show very high compressibility suggesting possibility to destabilize them with pressure. With high capacity and favorable thermodynamics these borohydrides are suitable for reversible storage.

Effect of water on the slip systems activity in the deformation of olivine single crystals

Girard J[1], Chen J[1], Raterron P[2], Holyoke C[3]

[1], CeSMEC, Florida International University, University Park Campus, Blg. VH140, Miami, Florida 33199, USA
[2],LSPES, CNRS, Bat C6, Universite des Sciences et Technologies de Lille, Villeneuve d'Ascq F-59655, France
[3] Texas A&M University, Dept. of Geology and Geophysics, College Station, TX 77843- 3115, USA

Seismic velocity anisotropies observed in the upper mantle are interpreted from lattice-preferred orientations (LPO) produced experimentally in olivine, which depends on the dominant dislocation slip systems. At low pressure P<3GPa, mantle temperature (T) and in dry conditions, olivine [100] dislocation slip dominates the less active [001] slip. This tends to align crystal fast velocity [100] axis with the principal shear direction. Yet recent high-pressure deformation experiments, show that [001](010) slip system dominates [100](010) system in the (P,T) range of the deep upper mantle (Couvy et al (2004), Raterron et al (2007), Raterron (2008)). This may promote a shear-parallel slow-velocity [001] axis and may explain the seismic-velocity attenuation observed at depth >200 km (Mainprice et al. (2005)). According to those studies, the slip systems activities depend on pressure. However previous studies revealed that at high temperature and pressure the water could affect the olivine plastic deformation and induce the transition between the [100] slip to the [001] slip (Jung et al. (2001), Jung at al. (2006)).

In order to further constrain the effect of water on olivine slip system activities at high pressure and high temperature, deformation experiments were carried out in wet condition, at pressure between 4-7GPa and temperature around 1473K on olivine single crystals, using the Deformation-DIA apparatus at the X17B2 beamline of the NSLS (Upton, NY). Crystals were oriented in order to active either [100] slip alone or [001] slip alone in (010) plane. A talc sleeve surrounding the single crystal was used as source of water during the deformation. Constant applied stress σ and specimen strain rates were monitored in situ using time-resolved x-ray diffraction and radiography respectively. The obtained rheological data were compared with data previously obtained in comparable single crystal deformation experiment carried in drier condition.

The results revealed that the transition between the [100] slip and [001] slip in wet condition occur around 5-6GPa, which is a much lower pressure than the pressure predicted by the deformation results obtain in dry condition which was around 8GPa Raterron et al. (2008). Thus, our results indicate that presence of water in the single crystals decreases the transition pressure between the a-slip and the c-slip notably.

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Trace element partitioning in the Fe-S-C±P system and the chemical differentiation of planetary cores

L.A. Hayden[1]*, J.A. Van Orman[1], W.F. McDonough[2], R. D. Ash[2]

[1]Dept of Geological Sciences, Case Western Reserve University, Cleveland OH 44106(*correspondence: lah91@case.edu; jav12@case.edu); [2]Dept of Geology, University of Maryland, College Park MD 20742 (mcdonoug@umd.edu, rdash@umd.edu)

Element partitioning in metal-light element systems is important to our understanding of planetary differentiation processes. There have been numerous studies of partitioning in solid-metal/liquid-metal systems, but none have explored the simultaneous influence of S and C. Here we study the influence of both elements on trace element partitioning in the graphite-saturated Fe-S-C±P system.

Fe-S±P powders were doped with a powder mix of Ag, As, Au, Co, Cr, Cu, Ge, Ir, Ni, Os, Pd, Pt, Mo, Mn, Re, Ru, Se and W such that the concentration of each element was several hundred ppm. The Fe-S±P powders were packed into a graphite capsule, placed in a silica glass tube, and sealed under vacuum to buffer the fO_2 at C-CO. The capsules were held in a 1-atm furnace at temperatures between 975–1350°C for a period of 4–600 hrs and then quenched in cold water. Although core formation processes are inherently high-pressure processes (compared to this 1-atm study), the composition of coexisting liquids exerts a much greater influence on partitioning than does pressure.

Solid-metal/liquid-metal partition coefficients (1025-1125°C in Fe-S-C) show only small changes with temperature that correspond to a small change in S in the liquid. They are generally consistent with solid/liquid partition coefficients predicted by the pure Fe-S system [1], which is likely due to the nearly equal distribution of C between the solid and liquid.

Above 1125°C the solid Fe metal is replaced by a C-rich, S-bearing liquid. The presence of C keeps the S content of the sulfide liquid high, and thus partition coefficients for the highly siderophile elements (HSE) remain high up to 1350°C, 10-100x higher than in pure Fe-S [1]. The immiscible liquids in the Fe-S-C system are therefore capable of producing strong HSE fractionations at much higher temperatures than pure Fe-S.

In the Fe-S-C-P system, liquid immiscibility exists over a wider range, at least 975-1350C°. If the metallic cores of planetary bodies generated immiscible liquids at certain stages of their evolution [2], such coexisting liquids could have very different trace element compositions The immiscible phases could separate due to a density contrast (Fe-S-C) or remain well-mixed (Fe-S-C-P), thereby influencing both the chemical and physical properties of planetary cores.

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Investigation of the effect of stress on melting point determination in a laser heated diamond anvil cell and application to Iron

Rostislav HRUBIAK, Surendra K. SAXENA, Andriy DURYGIN

CeSMEC, Florida International University, University Park Campus, VH140
Miami, Florida 33199, USA

Melting temperature of transition metals at high pressures have been measured by various groups using a laser heated diamond anvil cell technique (LH-DAC) [1,3] Also, melting temperatures have been extrapolated using shockwave Hugoniot curves. [2] It is well known that the LH-DAC and shockwave techniques have not been reported to be in good agreement with each other over the past decades. Several investigated compounds like NaCl, KBr, have been seen to show accord between LH-DAC and shockwave measurements by following certain superheating systematics as calculated using molecular dynamics. However, iron and other transition metals still show large differences. [2] Melting temperatures at high pressures reported from LH-DAC techniques are still considerably lower than the melting temperatures extrapolated from shockwave data. The understanding of accuracy of temperature measurement, melting determination criteria and other factors which play a role in the accuracy of the LH-DAC technique is continuously improving [3] but is still a matter for discussion.

Using a LH-DAC with a radiometric temperature measurement system in CeSMEC (Miami, FL), we investigate whether axial stress in the DAC has an effect on the melting temperature determination. In order to measure what effect stress has on the melting temperature measurement we use a combination of laser heating with external heating. A focused YAG laser is used for the melting of the sample and external heating is provided in order to reduce the stresses present in the sample and the pressure medium. Melting curves with and without external heating will be presented.

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Spin states and hyperfine interactions of iron in (Mg,Fe)SiO$_3$ perovskite under pressure

Han Hsu

Department of Chemical Engineering and Material Science
University of Minnesota, Minneapolis, Minnesota, USA

With the guidance of first-principles phonon calculations, we have searched and found several metastable equilibrium sites for substitutional ferrous iron in MgSiO$_3$ perovskite. In the relevant energy range, there are two distinct sites for high-spin, one for low-spin, and one for intermediate-spin iron. Because of variable d-orbital occupancy across these sites, the two competing high-spin sites have different iron quadrupole splittings (QS). At low pressure, the high-spin iron with QS of 2.3 - 2.5 mm/s is more stable, while the high-spin iron with QS of 3.3 - 3.6 mm/s is more favorable at higher pressure. The crossover occurs between 4 and 24 GPa, depending on the choice of exchange-correlation functional and the inclusion of on-site Coulomb interaction (Hubbard U). Our calculation supports the notion that the transition observed in recent Mössbauer spectra corresponds to an atomic-site change rather than a spin-state crossover. Our result also helps to explain the lack of anomaly in the compression curve of iron bearing silicate perovskite in the presence of a large change of quadrupole splitting, and provides important guidance for future studies of thermodynamic properties of this phase.

Relative strength of the pyrope-majorite solid solution and the flow-law of majorite containing garnets.

Simon Hunt[1,2], David Dobson[1], Li Li[2], Don Weidner[2], and John Brodholt[1]

(1) Department of Earth Sciences, University College London, Gower Street, London, WC1E 6BT
(s.hunt@ucl.ac.uk),
(2) SUNY Stony Brook, Institute of Mineral Physics, Department of Geosciences, Stony Brook, NY 11790

Even though the garnet phase is the second most abundant phase in the upper-mantle and transition-zone, no previous studies have directly measured the effect of majorite content on the strength of garnet under mantle conditions. Here we report the results of constant strain-rate and stress-relaxation experiments on garnets in the pyrope–majorite solid solution which constrain the strength of majoritic containing garnets relative to pyrope as a function of majorite content and temperature. We find that at temperatures below 650°C both pure pyrope and majoritic garnets have the same strength. Conversely, above 650°C we find that majoritic garnets are initially stronger than pure pyrope but weaken with increasing temperature and majorite content and with significant majorite contents are weaker than pyrope above approximately 800°C. We develop a flow law for the entire pyrope–majorite solid solution as a function of temperature and majorite content. From our experimental results it is necessary that majorite is the weak phase in the transition-zone.

Furthermore, the relative-weakness of majorite garnet, which dominates the mineralogy of the subducted MORB layer at transitionzone depths, further promotes thermal run-away processes as the causal mechanism for deep-focus Earthquakes.

A new X-radiography based method for measuring thermal diffusivity at high pressures

Simon Hunt[1,2], Don Weidner[2], Li Li[2], Richard McCormack[1], and David Dobson[1]

(1) Department of Earth Sciences, University College London, Gower Street, London. WC1E 6BT
(s.hunt@ucl.ac.uk),
(2) SUNY Stony Brook, Institute of Mineral Physics, Department of Geosciences, Stony Brook, NY 11790

We have developed a new variation of the Ångstrom method for measuring thermal diffusivity at high pressures by using X-radiography. We have measured the thermal diffusivity of a number of upper-mantle and transition-zone phases and our data are in agreement with previous measurements.

The Ångstrom method for measuring thermal diffusivity at high pressure uses a stationary thermal wave which is induced in the sample by varying the power sinusoidally in the surrounding cylindrical furnace. The thermal diffusivity (κ) is determined from the phase lag, $\Phi_0 - \Phi_R$, and amplitude difference, θ_0/θ_R, of the thermal wave between points at the axis of the sample and radius, R (e.g. Khedari et al., 1995).

Our method differs from previous multi-anvil implementations of the Ångstrom method in that instead of using thermocouples to monitor the temperature variation we use thin strips of metal foil, which are placed at discrete intervals along the sample length and imaged X-radiographically. The metal strips monitor the thermal expansion of a slice across the sample in response to the sinusoidal temperature profile. This represents an improvement over previous methods since (i) the change in temperature is averaged along the sample length, (ii) we measure the phase of the thermal wave at all radii and (iii) since the expansion of the sample is observed as a proxy for the change in temperature there are no problems associated with contact thermal resistance at the thermocouples. Furthermore, this development does away with the need to prepare long cylinders of weakly metastable phases with a thermocouple inserted precisely down the middle; a process which is technically extremely difficult.

To date we have measured the thermal diffusivity of NaCl, olivine, majorite and a number of other upper-mantle and transition-zone phases. The measurements we have made are all in agreement with previously published data. The simplifications to the technique inherent in this X-radiographic technique will allow us to measure the thermal diffusivity of lower-mantle phases.

Bulk Attenuation of the Earth's Mantle Due to Phase Transitions

Li Li

Mineral Physics Institute

Stony Brook University, Stony Brook, NY, 11794-2100, USA

Bulk attenuation occurs in the Earth's interior as manifest by the damping of the radial modes of free oscillation [1]. Among various physical mechanisms contributing to the bulk dissipation, phase transitions are likely to be important [2-4]. Here we contrast two distinctive models: a kinetic mode [3] and a diffusion model [2] to define the relaxation times and the magnitude of the bulk attenuation using the available experimental data [5]. These results are compared with the inverted results from radial modes of free oscillation data [1]. We find that the phase transitions in the mantle can contribute to the bulk attenuation significantly; and that the relaxation times of phase transitions are likely less than 1 second.

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Modeling Effects of Hydration on Mantle Elasticity: A Comparison with Seismology

Xiaoting Lou, Steven Jacobsen, Suzan van der Lee, Craig Bina and Yun-Yuan Chang

Northwestern University, Department of Earth and Planetary Sciences

Seismological observations can provide constraints on velocity and density structures in the Earth's interior and yield various global-average models (such as PREM, IASP91 and AK135) and regional models (such as TNA, SNA and NA04). Velocity anomalies in regional models with respect to reference global model can be caused by variations in temperature, composition, and mineralogy. Particularly, water content is an important factor because it can be incorporated (up to several weight percent) in the structures of major mantle minerals olivine, wadsleyite and ringwoodite, in the form of hydroxyl $(OH)^-$. Hydration influences seismic velocities of the $(Mg,Fe)_2SiO_4$ polymorphs by reducing the elastic moduli and changing the pressure and temperature derivatives of the moduli. To study the effects of hydration on seismic velocities in detail, we perform finite-strain forward modeling of velocities using a compilation of recent mineral physics laboratory data. P and S wave velocity profiles are calculated from density, elastic moduli, pressure and temperature derivatives of moduli, and thermal expansion coefficients of mantle rock-forming minerals. In the calculations, water content is a variable in the depth profile which can be derived from studies of water solubility and water partitioning coefficients for olivine, wadsleyite and ringwoodite. Therefore, velocity models from both mineral physics and seismology can be compared and help to interpret the velocity variations in seismic models.

High pressure studies on extended phases of CO_2

Recent findings have shown that the chemistry of CO_2 at high pressure and temperature is richer than previously thought and that the activation of the C=O bond can give origin to different forms of solid non-molecular CO_2. Such findings may have important implications for the understanding of the Earth's deep carbon cycle and CO_2 sequestration technologies. First principles simulations of CO_2's electronic properties under different pressure and temperature conditions can constrain the thermodynamic phase diagram of CO_2 and explore P-T conditions necessary for the C=O bond activation. Our calculations show that at about 50 GPa molecular CO_2 can transform to a metastable amorphous form characterized by an almost equal proportion of three- and four-fold coordinated carbon atoms [1], while higher carbon coordination does not take place up to at least 900 GPa [2-3]. Our results suggest that pressure can radically alter the oxidation chemistry of carbon. Finally, we analyze the possible implications of these findings on CO_2's liquid phase at high pressure [4].

[1] J. A. Montoya *et al.*, PRL 100, 163002 (2008)
[2] J. Sun et al., PNAS 106, 6077 (2009)
[3] M-S. Lee, J. A. Montoya and S. Scandolo, PRB 79, 144102 (2009)
[4] J. A. Montoya, M. Fontana, M-S. Lee, and S. Scandolo, unpublished.

Abstract

First principles study of the effect of lattice anharmonicity on the thermodynamic properties of minerals at high temperatures

Moses Ntam and Jianjun Dong

Physics Department, Auburn University, Auburn, Alabama 36849-5311

We develop a first principle method which predicts high temperature thermodynamic properties of minerals beyond the conventional quasi-harmonic approximation (QHA). Our method is based on a perturbative approach to evaluate the canonical partition function of an *interacting* many-phonon system. The phonon-phonon interaction is directly calculated up to 3rd and 4th order of lattice anharmonicity using our new algorithm that combines the first principles density functional theory (DFT) and group theoretical analysis. We have adopted this technique to study the anharmonic contribution to the free energy of MgO and Al_2O_3. We find a significant improvement of the predicted thermal equation of state at ambient pressure and higher temperatures compared to the previous results based on QHA. Our technique should be applicable to a wide range of minerals, allowing increased accuracy in the prediction of thermodynamic properties at high temperatures.

Fundamentals of Elasticity of $(\mathrm{Mg}_{1-x},\mathrm{Fe}_x)_2\mathrm{SiO}_4$ Olivine

Maribel Núñez,[*] K. Umemoto,[†] and R. M. Wentzcovitch[‡]

May 13, 2010

Abstract

We study the influence of iron on the elasticity of $(\mathrm{Mg}_{1-x},\mathrm{Fe}_x)_2\mathrm{SiO}_4$ olivine, a major constituent of the Earth's upper mantle. We calculate its static elastic properties by first principles for a set of representative upper mantle pressures for $x = 0$ and $x = 0.125$. We investigate the effect of atomic arrangement and composition on all single crystal and poly-crystalline elastic moduli. We also calculate wave propagation velocities and anisotropies in single crystals and in aggregates with transverse anisotropy. Thus a thorough comparison between the elasticity of olivine $(x = 0.125)$ and forsterite $(x = 0)$ is made.

[*]School of Physics and Astronomy, University of Minnesota, Minneapolis, Minnesota, USA. (valdez@physics.umn.edu)

[†]Department of Geology and Geophysics, University of Minnesota, Minneapolis, Minnesota, USA. (umemoto@cems.umn.edu)

[‡]Department of Chemical Engineering and Materials Science, Minnesota Supercomputing Institute for Digital Technology and Advanced Computation, University of Minnesota, Minneapolis, Minnesota, USA.(wentzcov@cems.umn.edu)

Influence of iron oxidation state on Mg-Fe interdiffuion in anhydrous and hydrous (Mg,Fe)O

Kazuhiko Otsuka[1]*, Micaela Longo[2], Catherine A. McCammon[2], Shun-ichiro Karato[1]

[1]Yale University, Department of Geology and Geophysics, 210 Whitney Avenue, New Haven, CT, 06511, USA

[2]Bayerisches Geoinstitut, Universität Bayreuth, D-95440, Bayreuth, Germany

Abstract:

The knowledge of transport properties such as atomic diffusion, viscosity and electrical conductivity in the ferropericlase–magnesiowüstite solid solution, (Mg,Fe)O, is critical for understanding the dynamics of the lower mantle and interpreting its geophysical observations. Some previous studies based on point defects concentrations suggest that the behavior of transport properties in (Mg,Fe)O may change with increasing pressure from mechanisms sensitive to Fe^{3+} content (or oxygen fugacity) to those sensitive to H^+ content (or water fugacity). It is thus important to explore if such a transition is likely to be present in the lower mantle. However, the critical conditions above which water fugacity plays an important role are not well explored at present.

We have conducted high pressure experiments on interdiffusion of Mg and Fe in (Mg,Fe)O in order to quantify the relative contributions of oxygen fugacity and water fugacity. The diffusion couples consisting of single crystals of MgO and (Mg,Fe)O with Mg# around 70 were annealed at temperatures from 1673 to 2073 K and pressures from 5 to 15 GPa under hydrous and anhydrous conditions with several oxygen fugacity buffers (Mo-MoO$_2$, Ni-NiO, Re-ReO$_2$). We measured the iron oxidation state of (Mg,Fe)O

diffusion pairs by the flank method. Preliminary results suggest that water fugacity does not significantly increase Mg and Fe interdiffusion at 5 to 8 GPa and 1873 K with Mo-MoO$_2$ oxygen fugacity buffer. This is consistent with a diffusion model of defect mechanisms since cation vacancies charge balanced by Fe^{3+} are significantly more abundant than those charge balanced by H$^+$ under these conditions.

Viscosity of water at high pressures and high temperatures

Pigott, J.S.

Reaman, D.M.

Panero, W.R.

The viscosity of subduction-zone fluids is a first-order constraint on the transport of material from a subducting plate to the mantle, varying up to 10 orders of magnitude between the limits of pure liquid water and silicate melts. Accurate determination of fluid viscosity at high pressures (>4 GPa) and temperatures (~800 °C) is hindered by the geometry and sample size of high-pressure devices. We report the viscosity of water at pressures representative of the deep crust and upper mantle using a new experimental method. By tracking the Brownian motion of 3 micron polystyrene spheres suspended in fluid contained in a hydrothermal diamond anvil cell (HDAC), the *in situ* viscosity of the fluid at high pressures and temperatures can be determined by Einstein's relation. Initial experiments to 4 GPa and 250 °C show that particle tracking of 5-6 particles for 6-30 seconds at 30-60 x magnification results in viscosities accurate to within 0.35 log units of published values using complementary methods. Development of a method that uses Brownian motion observation inside of a HDAC will validate laws regarding the temperature dependence of viscosity for water at supercritical conditions and also allow for systematic viscosity experiments on realistic subduction zone fluids.

Developments on resistive heating in diamond anvil cell at beamline 12.2.2

Selva Vennila Raju[1], J.Knight[1], B.Chen[1], J.Yan[1], S.M. Clark[1],
B.K. Godwal[2], G. Zack [2], R. Benneditti[2], R. Jeanloz[2]
L. Miagi[3], K.Jane[2], P.M. Kaercher[2], R.Wenk[2]

[1]Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, CA 94720
[2] Department of Earth and Planetary Sciences, University of Califomia, Berkeley, CA 94720
[3]Department of Geology and Geophysics, Kline Geology Laboratory, New Haven, CT 06511

Resistive heating in diamond anvil cell offers larger samples volume under study and absence of localized heating in contrary to laser heated samples[1]. We have developed resistive heating set–up for use in axial diffraction geometry up to 873K and Radial diffraction geometry up to a maximum temperature of 1073K combined with laser heating temperatures up to 2500K was recorded. Recently, we performed high temperature studies on Gallium gold using resistive heating technique in diamond anvil cell and recorded evolution of melting. A.R.Storm et. al. (1966)[2] studied the melting temperature in belt apparutus using Differential Thermal Conductivity Analysis (DTCA) technique by ex-situ measurements. Our melting point measurement by in-situ X-ray diffraction technique is found to be in close agreement with the literature[2]. Description of the experimental set-up and pressure dependence of melting temperature of $AuGa_2$ will be presented.

References:

1. Dubrovinsky et. *al.,* High Pressure-High Ternpemtzim, 31(1999).
2. A.R.Storm et. al. J. Phys. Chem. Solids (1966).

Equation of state of $Fe_{64}Ni_{36}$ alloy by synchrotron radiation and applications to Earth's inner core

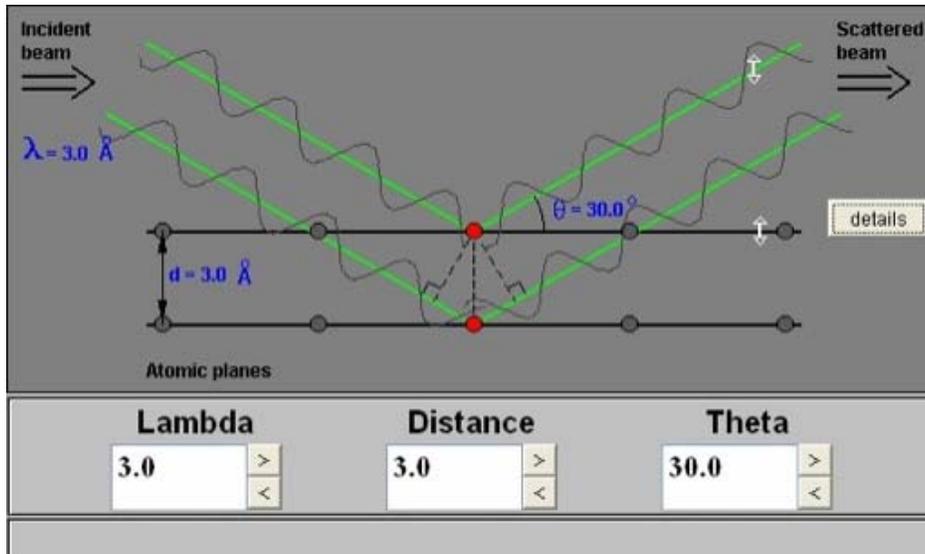Reaman, D.M.

Pigott, J. S.

Meng, Y.

Panero, W.R.

Geophysical data and cosmochemical models suggest that the Earth's core is composed of iron alloyed with 5-35% nickel. Therefore, the high-pressure, high-temperature behavior of iron-nickel alloys is necessary to interpret geophysical data and to place constraints on models of inner-core structure and deformation. Although nickel has been shown to stabilize the *fcc* phase of iron-nickel alloys, the phase relations and crystal structure at temperatures and pressures of Earth's inner core are still somewhat controversial. Equation of state parameters have been determined for different iron-nickel alloy compositions at high pressures and temperatures [e.g. *Lin et al*, 2002; *Huang et al*, 1992], and phase relations of iron and iron-nickel alloys were recently reported by *Kuwayama et al* [2008] up to 300 GPa. Here, we report results on the high-temperature equation of state, phase stability of *fcc*-structured $Fe_{64}Ni_{36}$ alloy to 95 GPa and 3100 K. Experiments were performed at 16 ID-B (HPCAT) at APS. Ar, NaCl and MgO served both as pressure media and internal pressure standards and the pressure values from these standards were compared with ruby fluorescence at room temperature. The room-temperature bulk modulus is 164(2) GPa (K' fixed at 4), with a corresponding V_0 of 6.92(2) cc mol^{-1}. High temperature measurements are consistent with $dK/dT = -1.2(1) \times 10^{-2}$ and a decreasing average thermal expansion coefficient, from $16.6(1) \times 10^{-6}$ K^{-1} at 38 GPa to $8.6(1) \times 10^{-6}$ K^{-1} at 71 GPa, with a $d\alpha/dP$ of $-2.4(1) \times 10^{-7}$ K^{-1} GPa^{-1}. Data are also compared with previous studies to determine the coefficient of thermal expansion as a function of nickel content in iron-nickel alloys at high pressures and temperatures, and implications for Earth's inner core are explored.

A Virtual Workshop for Educators on Understanding the Deep Earth

Glenn A. Richard – Stony Brook University

In February, 2010, the National Association of Geoscience Teachers (NAGT) hosted a virtual workshop entitled "Understanding the Deep Earth: Slabs, Drips, Plumes and More", as part of its "On the Cutting Edge" series. The workshop was conducted online through the Science Education Resource Center at Carleton College. Information about the event is available at:

http://serc.carleton.edu/NAGTWorkshops/deepearth10/index.html

Bragg's Law Java Applet

Goals of the Workshop

- Help participants stay current about data, tools, services, and research related to the deep earth.
- Address the "big science questions" related to deep earth (e.g. plumes, slabs, drips, post-perovskite, etc.) and explore exciting new scientific approaches (e.g. noise processing to derive images of the deep crust).
- To consider ways to effectively teach about "what can't be seen", at least not directly.
- Develop and review classroom teaching activities for undergraduate education using these data, tools, services, and research results to facilitate teaching about the deep earth across the geoscience curriculum.

In order to achieve its goals, the workshop offered a set of formal audiovisual presentations as well as working sessions that facilitated the development, review, and refinement of classroom teaching activities for undergraduate education.

Several members of the Consortium for Materials Properties Research in Earth Sciences (COMPRES) community participated and contributed activities to the workshop. Development and refinement of the projects is ongoing. As of May 19, 2010, the following activities, alphabetized by author, had been developed and contributed by people from COMPRES and other research communities:

- **What does the core/mantle boundary look like?**
  - Suzanne Baldwin
  - This activity explores how Earth scientists infer what materials are present at the core mantle boundary and what this boundary might look like.
- **How do Faults Slip: Earthquakes versus Episodic Tremor and Slip**
  - Mike Brudzinski
  - A comparison of earthquakes and episodic tremor and slip using GPS and seismic data to illustrate how faults slip.
- **Multi-Anvil Tutorial**
  - Pamela Burnley
  - Students in mineralogy, igneous petrology or metamorphic petrology classes learn about multi-anvil devices.
- **What Does Seismic Anisotropy Reveal About the Deep Earth?**
  - Eric Christiansen
  - The objective of this assignment is to help students describe the cause of shear wave splitting and explain what it reveals about deep Earth structure and dynamics by examining the recent literature on the topic.
- **Adopt a Blob**
  - Catherine Cooper
  - Blobs are everywhere in the deep Earth! Students adopt a "blob" and develop the various associated observable anomalies for it (tomography, gravity, etc).
- **Determining Mantle Temperature and Composition from Discontinuities**
  - Anna Courtier
  - This pair of activities uses observations of mantle discontinuity structure to examine mantle heterogeneity and differences in the depth extent of melt sources for ridges and "hotspots." The first activity focuses on interpretations linked with temperature anomalies, and the second activity emphasizes that contributions from variable composition (iron and water) are important to consider as well.

- **How do modeled P-wave travel times compare with travel times for an actual earthquake?**
  - Vince Cronin
  - Students use published travel times for a standard radial-earth model, acquire similar data for a significant earthquake from a web-accessible seismic-data center (or from their teacher), and compare the observed with the model data.
- **Finding the Moho under Milwaukee**
  - Vince Cronin and Keith Sverdrup
  - Students study a seismogram that was recorded at the University of Wisconsin-Milwaukee during the explosive demolition of a section of the nearby Hoan Bridge on December 28, 2000.
- **Comparing Solar System and Chondrite Elemental Compositions**
  - Ben Edwards
  - This assignment gives students a broad picture of the distribution of elements in the solar system and in C1 chondritic meteorites.
- **Waves Go Deep**
  - Ed Garnero
  - This classroom experiment demonstrates bending wave fronts in a medium as velocity increases with depth. Each row in the classroom models an earth layer and students in that row have a "property" (how long they count before passing a wave along).
- **Tomography? How do we know what is below our feet?**
  - Barbara Graham
  - A short Powerpoint presentation that introduces seismic topography to begin understanding how scientists infer the interior of the earth. Followed with a crossword puzzle to encourage vocabulary.
- **How do we know?**
  - Vicki Hansen
  - Students in groups of 4-5 are charged with making posters that explain various first order concepts/facts about the Earth's interior, including articulating the 'embedded' assumptions.
- **Scientific debate: Mantle plumes**
  - Brennan Jordan
  - Students engage in a structured debate on the mantle plume hypothesis and alternative hypotheses with supporting materials.
- **One-Di Earth Model Toy Model**
  - Abby Kavner
  - This is an Excel PREM toy model combining mineral physics and seismology for the deep Earth.
- **Structural transitions in olivine**
  - Wendy Mao
  - Students use a crystal structure visualization program (e.g. VESTA) to study how the major mineral of the upper mantle, olivine, changes with depth.

- **Interrogating the EarthChem Deep Lithosphere Petrological Dataset**
  - Dave Mogk
  - The EarthChem Deep Lithosphere Petrological Dataset provides global geochemical data for xenoliths and exposed sections of Deep Earth.
- **Light Diffraction**
  - Wendy Panero
  - This activity demonstrates the concept of diffraction through a variety of lattice types using a laser pointer and printed gratings.
- **Bragg's Law**
  - Glenn A. Richard
  - Students use an interactive Java applet to explore Bragg's law.
- **Wave Interference**
  - Glenn Richard
  - This activity engages students in the exploration of interference between sinusoidal wave forms. They use an interactive applet to manipulate the phase, wavelength, amplitude, and phase velocity of two waveforms which produce a sum waveform.
- **Imaging the Earth through tomography**
  - John Taber
  - Students learn the basics of how a tomographic model is created.
- **Modeling Mantle Minerals**
  - Laura Reiser Wetzel
  - Students enhance their understanding of crystal chemistry by building minerals with easy to use modeling kits.

Although the workshop is past, NAGT continues to host workshops that are of interest for educators. In addition, researchers and educators can contribute their own material to the collections by visiting:

http://serc.carleton.edu/NAGTWorkshops/deepearth10/upload.html

For information about COMPRES, see:

http://compres.us

Event:

2010 COMPRES Annual Meeting

Title:

Compressibility and Phase Transitions in β-Tricalcium Phosphate and Magnesium Substituted β-Tricalcium Phosphate to 50 GPa

Authors:

Paul R. Scott[1], J. Crow[1], G. Knotts[1], R. LeGeros[2] and M. B. Kruger[1]

[1] *Department of Physics, University of Missouri-Kansas City, MO 64110, USA*
[2] *New York University, College of Dentistry, New York, NY, USA*

Abstract:

Synchrotron based x-ray diffraction was used to determine the equations of state and phase transitions in β-tricalcium phosphate (β-TCP) and magnesium substituted β-tricalcium phosphate (β-TCMP). β-TCP was prepared by sintering commercially purchased TCP powder. β-TCMP was prepared by sintering β-TCMP precipitate obtained by a reaction between a solution containing Mg and Ca ions with a solution containing phosphate ions. It was observed that β-TCP undergoes two phase transformations, to an undetermined crystalline phase at ~ 10 GPa and an amorphous structure at 20 GPa. The high pressure glass was quenchable to 0 GPa. However, for β-TCMP there was no intermediate crystalline phase, the low pressure rhombohedral phase converted directly to a glass at 21 GPa. This was also quenchable to 0 GPa. Rietveld refinement was used to calculate unit cell parameters and the Burch-Murnaghan equation of state was used to calculate the bulk moduli.

Experimental Investigation of the Fe-Si-O System at High Pressures

Seagle, C.T.[1*,2], Cottrell, E.[1], & Fei, Y.[2]

[1]Dept. of Mineral Sciences, National Museum of Natural History, Smithsonian Institution, Washington, DC
[2]Geophysical Laboratory, Carnegie Institution of Washington, Washington, DC ([*]seaglec@si.edu)

Our only direct information regarding Earth's core results from seismological observations. Acoustic waves propagate through Earth at velocities dependent on the density and elastic properties of the medium. Interpretations of these measurements are dependent on our understanding of the material properties at the environmental conditions that exist within Earth's deep interior. Earth's outer core is composed of an iron rich alloy plus ~10 wt. % light elements while the inner core consists of ~3-6 wt. % light elements; possible light elements are O, S, Si, and others [1].

We have investigated the iron rich portion of the Fe-Si-O system at pressures between 40 and 90 GPa using the laser heated diamond anvil cell in conjunction with synchrotron x-ray diffraction (XRD) at the Advanced Photon Source, Argonne National Laboratory. The XRD data indicates that at sub-solidus temperatures, solid iron coexists with wüstite and stishovite, in general agreement with previous data obtained at lower pressures [2]. However, as temperature is further increased (or decreased in reversal experiments) Bragg reflections associated with stishovite disappear (appear) around 1800 K at 40 GPa. This temperature is too low to be associated with melting and implies two phase coexistence at high temperatures (Fe-Si alloy and wüstite).
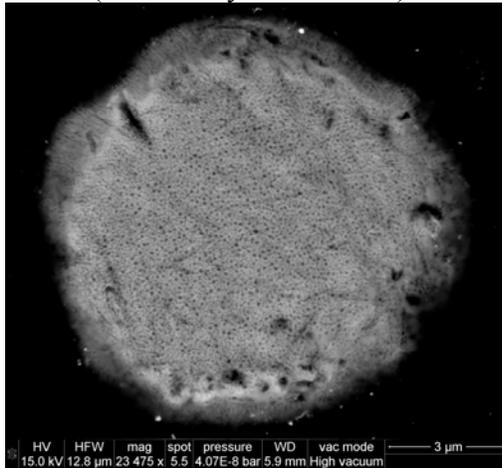

Fig. 1: Backscattered electron image of a laser heated spot in an Fe-Si-O sample recovered from 60 GPa and 4900 K. X-ray diffraction data indicates that at these conditions the sample was completely molten.

The samples were recovered after the XRD experiments and polished for analysis using a SEM and electron microprobe. Very small (~50 nm) circular features in the heated areas quenched from liquid are thought to indicate the exsolution of wüstite during rapid quenching. The bulk compositions of the quenched liquids were determined at several pressures and temperatures, an increasing oxygen content of the liquid with increasing temperature was observed. The in-situ XRD measurements and ex-situ characterization will help us to better understand the melting relations in the system at high pressure.

[1] Poirier, J.P. (1994), "Light elements in the Earth's outer core: A critical review" Phys. Earth Planet. Inter. 85, 319-337. [2] Ohtani, E. (1979), "Melting relation of Fe_2SiO_4 up to about 200 kbar", J. Phys. Earth, 27, 189-208.

High Pressure Study of Rehydrogenation of Ammonia Borane

in SBA-15 as Hydrogen Storage Material

Yongzhou Sun*, Jiuhua Chen, Vadym Drozd
Center for the Study of Matter at Extreme Condition(CeSMEC)
Department of Mechanical and Materials Engineering
Florida International University(FIU)
Miami, FL33199
*Applicant and Presenter

Abstract:

Ammonia borane has high potentials to be applied as on-board hydrogen storage material due to its abundant (19.6 wt%) hydrogen content. However, rehydrogenation of the spent fuel (decomposed ammonia borane) has been very challenging. We have conducted *in situ* Raman spectroscopy study to investigate possibilities of the rehydrogenation at high pressures using a diamond anvil cell (DAC). The result indicates that pressure may induce rehydrogenation of ammonia borane at about 9 GPa). Furthermore, mixing ammonia borane with meso-porous material SBA-15 may reduce the pressure required for the rehydrogenation down to 6 GPa. This result is consistent with the previous observation of the reduction of decomposition enthalpy. The pressure-induced rehydrogenation of ammonia borane will give guidance to further investigations on hydrogen storage.

The Elastic Properties of Diopside, $CaMgSi_2O_6$

Carine B. Vanpeteghem, Liqin Sang, Stanislas V. Sinogeikin and Jay D. Bass

Department of Geology, 1301 W Green St, University of Illinois at Urbana-Champaign, Urbana, IL61801, USA

Abstract

The thirteen single-crystal elastic moduli of two natural diopside($CaMgSi_2O_6$) samples from Wakefield Ontario (#1) and DeKalb NY (#2) have been measured at ambient pressure and temperature by Brillouin spectroscopy. We obtain C_{11} = 229.0(4), C_{22} = 179.0(4), C_{33} = 242.5(4), C_{44} = 78.9(3), C_{55} = 68.1(2), C_{66} = 78.2(3), C_{12} = 78.0(7), C_{13} = 69.8(6), C_{23} = 58.0(7), C_{15} = 9.9(3) C_{25} = 6.1(5), C_{35} = 40.9(3) for diopside 1, and C_{11} = 226.1(9), C_{22} = 179.5(8), C_{33} = 239.2(9), C_{44} = 78.1(6), C_{55} = 69.2(4), C_{66} = 76.4(8), C_{12} = 77(1), C_{13} = 70(1.3), C_{23} = 56.7(1.6), C_{15} = 9.9(7) C_{25} = 5.9(1.2), C_{35} = 41.0(7), C_{46} = 6.8(4) for diopside 2. The Cij show excellent agreement despite the differences in composition: namely, that diopside 2 has slightly highter Fe and Al content. Our C_{ij} values also compare well with the respective values obtained for chrome-diopside using the rectangular parallelepiped resonance (RPR) form of resonant ultrasound spectroscopy (RUS) by Isaak and Ohno (2003). From the measured C_{ij}'s, the isotropic aggregate bulk and shear moduli K_s and G were calculated from the elastic constants C_{ij}. We find K_s = 114.6(6) GPa, G = 72.7(1) GPa for diposide 1 and K_s = 114.0(1.2) GPa, G = 72.1(3) GPa for diopside 2, which are in good agreement with those found by Isaak and Ohno (2003). We confirm the systematic correlations between the M2 site composition and most of the C_{ij} values. We do not observe any unusual compositional dependence of the elastic moduli near end-member diopside .

Energy-Dispersive X-ray Diffraction Investigation of Amorphous Lithium Borate Structure: A Demonstration of the Paris-Edinburgh Cell Setup at 16BM-B at the APS

Tony Yu[1](tyu@cars.uchicago.edu), Yanbin Wang[1], Changyong Park[2], Jonathan Stebbins[3], Guoyin Shen[2]

1: GeoSoilEnviroCARS, Center for Advanced Radiation Sources, The University of Chicago, 5640 S. Ellis Avenue, Chicago, Illinois 60637, USA

2: High Pressure Collaborative Access Team, Geophysical Laboratory, Carnegie Institution of Washington, Argonne, Illinois 60439, USA

3: Department of Geological and Environmental Sciences, Stanford University, Stanford, CA 94305-2115, USA

Abstract

Knowledge of the structure and physical properties of silicate melts at a fundamental level is essential to help us understand the dynamics of mineral crystallization and fractionation, magma activities, thermal transport, etc. inside the Earth. It has been reported that fractions of liquid alkali borate in the silicate magma could possibly play an effective role in the dynamics of the Earth's interior [Lee et al., 2007]. Results from previous NMR, Raman, and neutron diffraction studies on borate glasses show that boron has mixed coordination numbers that vary strongly with composition and temperature [Cormier et al., 2006]. Inelastic x-ray scattering of borate glass structure under pressure has been studied by Lee et al. [2007]. However, studies of the pressure effect on liquid alkali borate structure are limited. In situ high pressure and high temperature x-ray total scattering of liquid state alkali borate has not even been reported. A 230-ton Paris-Edinburgh (PE) Press has been installed at Beamline 16BM-B at the Advanced Photon Source, Argonne National Laboratory since last summer. The PE Cell is capable of generating high pressure up to ~10GPa and can heat the sample cell up to 2000K in temperature. The bending magnet white beam spanning a large energy range up to 120 KeV provides sufficient energy to reach our experimental needs. The germanium solid state detector is mounted on a Huber rotation stage, which allows us to collect diffraction signal at two theta angles as high as 39.5 degrees. This, combined with the broad white beam spectrum, will provide a large Q range up to about 40 inverse Angstroms, making this setup an ideal beamline for studying non-crystalline materials. We have successfully collected high pressure (up to 7GPa) energy-dispersive x-ray diffraction data for lithium borate glass at room temperature. The preliminary result is promising and it demonstrates that the PE setup at Beamline 16-BM-B is capable of producing good quality structure data for amorphous materials and melts. We observed systematic shifts in peak positions with change of pressure in the reconstructed structure factors. However, further work needs to be done in order to completely understand our data and the pressure-induced structure change of alkali borates. Structure modeling of the detected atomic coordination number change between three-coordinated boron ($^{[3]}$B) and four-coordinated boron ($^{[4]}$B) may also be compared with previous experimental results from other methods to justify the validity of the x-ray total scattering method on studying borate melts. Equally important, this project will help us continue to develop the PE system, so that it will be ready for general users.

Changes in the local environments of iron in orthopyroxene at high-pressures

Dongzhou Zhang[1], Jennifer M. Jackson[1], Wolfgang Sturhahn[2], and Yuming Xiao[3]

[1] Seismological Laboratory, Division of Geological and Planetary Sciences, California Institute of Technology, 1200 E. California Blvd, Pasadena, CA 91125, U.S.A.
[2] Advanced Photon Source, Argonne National Laboratory, 9700 S. Cass Ave., Argonne, IL 60439, U.S.A; current address: Jet Propulsion Laboratory, Pasadena, CA, U.S.A.
[3] HPCAT, Geophysical Laboratory, Carnegie Institution of Washington, Bldg. 434E, 9700 S. Cass Ave., Argonne, IL 60439, U.S.A.

Abstract:

The site-specific behavior of iron in a ^{57}Fe-enriched orthopyroxene-structured $(Mg_{0.99}Fe_{0.01})(Mg_{0.86}Fe_{0.12(2)})Si_2O_6$ powdered sample was explored using Synchrotron Mössbauer spectroscopy and diamond anvil cells in two independent experiments. Experiments were performed at sectors 3 and 16 of the Advanced Photon Source at Argonne National Laboratory. In one experiment, NaCl was used as the pressure-transmitting medium, and in the other experiment, Ne surrounded the sample. The data were collected at ambient temperature, from 0 to 36 GPa for the cell loaded with NaCl as pressure medium, and from 4.1 to 26.8 GPa for the cell loaded with Ne as the pressure medium. The hyperfine parameters of the M1 and M2 sites at room pressure are in excellent agreement with previous literature values obtained using conventional Mössbauer spectroscopy. Analyses of both data sets reveal a change in the trend of the hyperfine parameters around 10 GPa, indicative of a transformation in orthopyroxene. However, the detailed behaviors of the iron sites with pressure depend on the local stress conditions provided by the different pressure media. We will discuss our results and potential implications for pyroxene transformations in Earth's upper mantle.