

rigid octahedral rotation around the tetragonal *c* axis as proposed by some first-principles calculations. This discovery may have important implications for the elastic moduli and anisotropy, and element partitioning in CaSiO<sub>3</sub> perovskite at lower-mantle pressures.

#### MR62B-1084 1330h POSTER

##### High Pressure Structural Properties of FeAlO<sub>3</sub>

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Recent diamond anvil cell experiments carried out at the GSECARS sector of the Advanced Photon Source indicate that FeAlO<sub>3</sub> transforms between 5 and 7 GPa to a tetragonal garnet structure (*a* = 11.529 Å, *c* = 11.046 Å). At 20–25 GPa, the garnet phase becomes unstable with respect to an orthorhombic perovskite phase (space group *Pbnm*, *a* = 4.584 Å, *b* = 4.883 Å, *c* = 6.669 Å). Atomistic simulations of these structures made using an empirical potential model show that at low pressure the fully-ordered garnet phase is metastable with respect to the ambient pressure phase, and a disordered phase can become stable as a result of its higher entropy. Theoretical results also show that the perovskite phase becomes stable at 25 GPa, in agreement with experiment. Similarities in compression behavior between MgSiO<sub>3</sub> suggest that the MgSiO<sub>3</sub>-FeAlO<sub>3</sub> join provides a suitable model system for the study of Fe<sup>3+</sup> in aluminous perovskite at lower mantle conditions.

#### MR62B-1085 1330h POSTER

##### Kinetics of the Pyroxene-Garnet Transformation: Preliminary Results

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Previous experimental studies have shown that in a "pyrolyte" mantle, Ca-poor pyroxene is completely dissolved into garnet at about 10 GPa, and Ca-rich pyroxene at about 16 GPa. Current mineralogical view of the Earth's upper mantle and the transition zone is mainly based on these results with the assumption that the pyroxene-garnet transformation proceeds with relative ease under the mantle conditions. In order to assess the validity of this critical assumption, knowledge about kinetics of the transformation is required. Here we report the preliminary results from an experimental study designed to investigate kinetics of the pyroxene to garnet transformation.

A 2000-ton uniaxial-split-sphere high-pressure apparatus was used to achieve mantle conditions of 6 to 14.5 GPa and 1200–1400 °C. Powdered KLB-1, whose bulk composition is close to "pyrolyte", was used as starting material. The powder sample was annealed at 6 GPa and 1300 °C for at least 4 hours before the target pressure and temperature were applied. Run duration ranged from 4 to 21 hours. Run products were analyzed using a micro-fractometer and an electron microprobe. It was found that substantial amount of Ca-poor pyroxene was still present in all samples, including the sample in which olivine has completely transformed to wadsleyite. Concentrations of oxide indicators for phase transition in garnet, such as SiO<sub>2</sub>, CaO, and Al<sub>2</sub>O<sub>3</sub>, were fairly unchanged from the pre-annealed material, suggesting that the transformation is extremely slow in occurring. We also observed exolutions of Ca-rich pyroxene or garnet in Ca-poor pyroxene, but they were too small to be analyzed. These results indicate that the pyroxene-garnet phase transformation can take complicated paths, and metastable pyroxene may exist in the Earth's lower upper mantle and the transition zone.

#### MR62B-1086 1330h POSTER

##### First principles prediction of the akimotoite-to-perovskite transition in MgSiO<sub>3</sub>

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The akimotoite-to-perovskite phase transition in MgSiO<sub>3</sub> is expected to contribute to the structure of the "660" km discontinuity in colder regions of the mantle. The transformation has a negative Clapeyron slope, behavior that has important implications for mantle dynamics. We have used first principles calculations to illuminate the origin of the negative slope and to test the ability of theoretical methods to predict the temperature dependence of phase transformation boundaries. We used density functional perturbation theory to investigate the structure, vibrational modes, and thermodynamic properties of akimotoite and perovskite.

We find that the negative Clapeyron slope can be attributed to the larger density of states of low frequency vibrations in the perovskite phase. Such vibrations consist of 1) Mg displacements: increased magnesium coordination and larger Mg-O bond lengths in perovskite lower vibrational frequencies of modes involving these displacements; 2) octahedral rotations: a lower degree of polyhedral connectivity in perovskite introduces low frequency nearly rigid unit modes. The larger density of state of low frequency modes in perovskite accounts also for the increase in other thermodynamic properties across the phase transition. This detailed calculation of a solid-solid phase boundary provides new insights into our ability to predict high pressure-temperature transformations by first principles.

#### MR62B-1087 1330h POSTER

##### Stability of Magnesite under the Lower Mantle Conditions

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MgCO<sub>3</sub> magnesite is an important mineral of some sedimentary rocks, and is probably the only stable carbonate in the deep mantle [1]. It was suggested that magnesite disproportionates to form an assemblage of MgO + C (diamond) + O<sub>2</sub> at pressures above 40–50 GPa [2]. We have examined this hypothesis and investigated stability of magnesite under the lower mantle conditions using a laser-heated diamond anvil cell with a synchrotron radiation [3]. The sample was a mixture of natural magnesite and a small amount of the platinum black powders. The latter powder was used as a heat absorber in laser heating as well as a pressure marker at high temperature. An Al<sub>2</sub>O<sub>3</sub> powder was used as a thermal insulator between the sample and the diamond anvil. The sample was enclosed in a hole of a Re gasket. All experiments were carried out under the condition of 15–110 GPa and 1800–3000 K at the Spring-8 high pressure beamline BL10XU. The x-ray beam was monochromatized to the wavelength of 0.3571–0.4130 angstrom and was collimated to 20 micrometer in diameter. In spite of the proposed stability limit of magnesite, we did not see any evidence for the dissociation at the investigated pressures and temperatures. On the other hand, although magnesite with a trigonal structure was stable at pressures up to 110 GPa, it transformed to an unknown phase with an orthorhombic structure at 110 GPa and 2200 K. The present results show that magnesite is stable down to depths of 2500 km and remains the major host phase of

carbon throughout the most part of the lower mantle. The newly found phase could be stable at deeper parts, and may play an important role in the slab-mantle-core interactions. [1] T. Katsura et al., Proc. Japan Acad. B67, 57 (1991) [2] L. Liu, Contrib. Mineral. Petrol. 134, 170 (1999) [3] T. Watanuki et al., Rev. Sci. Instrum. 72, 1289 (2001)

#### MR71A MCC: 122 Sunday 0830h

##### Applications of Neutron Scattering in Earth Sciences I (joint with V, DI)

Presiding: N L Ross, Virginia

Polytechnic Institute and State

University; J Parise, State University of New York, Stony Brook

#### MR71A-01 0835h INVITED

##### Insights into Analogue Perovskite Solid Solutions from High-Resolution Neutron Powder Diffraction

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Neutron powder diffraction provides a powerful tool for the study of phase transitions as a function of pressure, temperature, or chemical composition. The structural information obtainable from powders using diffractometers such as HRPD (ISIS, UK) or D2B (ILL, France) rivals, and in some respects exceeds what may be possible using conventional crystallographic techniques reliant on single crystals.

We have used both instruments to explore the subtle phase transitions observed in the (Ca<sub>x</sub>Sr<sub>1-x</sub>)TiO<sub>3</sub> and (Sr<sub>x</sub>Ba<sub>1-x</sub>)SnO<sub>3</sub> solid solutions. We have also used low resolution high flux instruments to explore the thermal dependence of the superlattice behavior below the cubic to tetragonal phase transition in (Ca<sub>x</sub>Sr<sub>1-x</sub>)TiO<sub>3</sub>. In each case a sequence of phase transitions from Pm-3m through I4/mcm to Pbnm is seen as a function of composition, and is driven by the change in average radius of the B-cation. This sequence of transitions is the same as expected for a magnesium silicate perovskite structure on increasing temperature. It is now recognised that lower mantle perovskite is likely aluminous, with solid solution towards either a stoichiometric or oxygen-defect end-member. The analogue systems we have characterised have been doped with trivalent cations on the B site to explore the effect of such substitution on the sequence of phase transitions. High-temperature neutron diffraction shows that oxygen defects stabilize the higher symmetry structures, lowering T<sub>C</sub> for the transition to cubic.

New developments in high-T high-P neutron diffraction techniques will allow the extension of these ambient pressure studies to the investigation of the influence of pressure as a variable, and hence the extension of such analogue studies to the whole range of variables experienced in the lower mantle. These will be briefly outlined.

#### MR71A-02 0850h

##### Neutron Diffraction Study of Silicate Perovskites

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The oxygen deficiency and cation-site distribution of silicate perovskite control its physical and chemical properties, including density, bulk modulus, defect mobility, ionic transport, flow behavior, oxidation states, hydration, and minor-element solubility. These properties of perovskite, in turn control the geophysical and

geochemical processes of the Earth. The possibility of oxygen deficiency was first recognized in perovskite with minor amounts of Al replacing Mg and Si [1, 2], and its significance is compared to the analogous defect perovskite in ceramics [3]. Basic crystallographic characteristics of the silicate perovskite, including the lattice parameters of the orthorhombic unit cell, the Pbm space group, and atomic positions, were previously determined by x-ray diffraction [4]. The defect crystallography of silicate perovskite, however, cannot be measured by x-rays because the relevant ions (Mg<sup>2+</sup>, Al<sup>3+</sup>, Si<sup>4+</sup> and O<sup>2-</sup>) are isoelectronic. These ions have very different neutron cross-section and can be readily resolved by neutron diffraction. Using multi-anvil apparatus, we synthesized perovskite samples at 1700°C and 25-28 GPa. We perform multiple runs to accumulate 3 mm<sup>3</sup> sample each for the MgSiO<sub>3</sub> end member and MgSiO<sub>3</sub> plus 5 weight % Al<sub>2</sub>O<sub>3</sub> in perovskite structure. Excellent powder diffraction data were collected at the POLARIS Beamline of ISIS, Rutherford Appleton Lab, and were subjected to Rietveld analysis. Neutron derived information sheds light on the unusual effects found for Al<sup>3+</sup> substitution on the compressibility of the silicate perovskite [1]. 1. J. Zhang and D. J. Weidner, *Science* 284, 782 (1999). 2. J. P. Brodholt, *Nature* 407, 620 (2000). 3. A. Navrotsky, *Science* 284, 1788 (1999). 4. N. L. Ross and R. M. Hazen, *Phys. Chem. Minerals* 17, 228 (1990).

#### MR71A-03 0905h INVITED

##### In Situ Observation of Texture Changes During Phase Transformations

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In one of the first user experiments with the new general purpose TOF neutron diffractometer HIPPO at Los Alamos, phase transformations during heating of bulk samples of quartzite and zirconium were investigated with a vacuum furnace. Both have geological relevance, for the crust and the inner core, respectively. A cylindrical sample (9mm diameter, 12mm high) of mylonitic quartzite was measured at room temperature, then at 500C, 625C, then cooled to 500C and heated again to 625C and finally cooled to 500C again to investigate memory effects during thermal cycling through the alpha-beta transition. A cubic (5mm) sample of zirconium was measured at 750C, 850C, 950C, 750C, 950C, 750C to investigate the hcp - bcc phase transformation. 30 detector banks at 150, 90 and 40 degree diffraction angles record differently oriented crystals. 180 spectra (in 6 sample orientations at each temperature) were then refined simultaneously with the Rietveld method MAUD to determine texture and crystal structure. The results indicate that in both materials there is a selection of orientation variants when samples are cooled from the disordered high temperature structure. It is speculated that a memory is imposed by the microstructure and topology of neighboring grains.

URL: <http://www.seismo.berkeley.edu/~wenk/hippo.htm>

#### MR71A-04 0920h INVITED

##### Dynamic Neutron Imaging of Silicate Melts

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We show that dynamic neutron imaging, i. e. the position- and time-resolved measurement of the attenuation of a neutron beam after transmission through a sample, can be used to study *in situ* processes in and physical properties of silicate melts.

Specifically, we have used this method to investigate viscosities and densities of melts, and visualized and quantified the growth of bubbles in a basaltic melt. A comparison to earlier experiments shows in all these cases that *in situ* measurements in large sample vessels are a prerequisite for reliable measurements. For example, the first experimental verification of the theoretically derived and often applied 'Faxen correction factor' for the falling sphere method shows that the application of this term leads to viscosities which

are too high by about 10%.

Current developments include the use of a large volume autoclave, with which the degassing of melts can be observed *in situ* for temperatures up to 1000 C and pressures of 1 kbar.

#### MR71A-05 0935h INVITED

##### Recent advances in the study of H environments and behavior in minerals using neutron powder diffraction

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It is now possible to probe the structural environments and behavior of H atoms directly in complex minerals such as amphiboles, micas, chlorites and humites using neutron powder diffraction, in some cases as a function of pressure and/or temperature. A combination of high neutron flux and increased detector sensitivity and size offers the chance to see details of H behaviour. In the last year or so the advent of new gasket designs for the Paris-Edinburgh pressure cell allow the use of ethanol/methanol (EtOD/MeOD) as a pressure medium, removing peak broadening arising from deviatoric stress that occurs above 3 GPa for the standard fluorinert pressure medium. Essentially hydrostatic conditions obtain with EtOD/MeOD to 8 GPa at 298 K. A further recent development has been the design of a high P-T module for use with the Paris-Edinburgh cell. These technological improvements in pressure-cell design now allow us to make meaningful correlations between OH vibrational spectra collected at high P and/or T and detailed structural information on H behaviour obtained from neutron diffraction under similar conditions.

In this talk I shall discuss recent neutron diffraction experiments on the effect of pressure upon hydrogen bonding in deuterated chlorite to 5 GPa (298 K), and a high P-T study of hydrogen bonding in deuterated brucite to 7 GPa, 1100 K. These two studies illustrate how far high-pressure neutron diffraction has come in the last 5 years.

Finally, I shall describe a neutron powder diffraction study (ambient conditions) of leucophoenicite, Mn<sub>7</sub>Si<sub>3</sub>O<sub>12</sub>(OH)<sub>2</sub>, a close structural analogue of Phase-B and Superhydrous-B: the structure of leucophoenicite is topologically identical to the hydrous sheet of Phase-B and similar to that of Superhydrous-B. For various reasons it was not possible to deuterate the sample. Nonetheless, the two distinct H atoms were approximately located in difference-Fourier maps and then refined isotropically. The H positions in Phase-B were only very approximately known from X-ray diffraction. The H...H separation determined by neutron diffraction is 2.16(1), and is much larger than that determined for Phase-B by X-ray diffraction, 1.93(6).

The examples discussed in this talk will serve to illustrate the potential and current limitations of the study of H in complex minerals.

#### MR71A-06 0950h

##### New results on H<sub>2</sub>O Ice VI. Phase equilibria, elasticity, molecular geometry and proton disorder.

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H<sub>2</sub>O is one of the most abundant substances on Earth. Despite the simplicity of the molecule, H<sub>2</sub>O has one of the richest phase diagrams amongst all the substances. Thus far, about 14 different phases of H<sub>2</sub>O have been identified, with varying pressure and temperature regions of stability. Ice VI is the central phase in the H<sub>2</sub>O phase diagram. It has boundaries with the liquid phase, as well as with solid phases V, VI, VII, VIII, and II. Detailed knowledge of the structure and properties of ice VI is crucial for understanding the phase behavior and mechanisms of phase transformations. Ice VI and VII are the highest-pressure forms of ice expected to be present in planetary interiors. They have been found trapped in natural diamonds, in the form of solid inclusions. According to estimates of PT conditions, ice VI can also be stable in the cold subduction zones. Pressures and temperatures at the core/mantle boundary of Ganymede are also estimated to be ideal for the this phase to exist ( 1.2-1.3 GPa). We report results of single-crystal XRD experiments with an ice VI crystal grown from a water-formic acid

solution. The presence of the formic acid liquid phase surrounding solid ice VI provided hydrostatic conditions for ice compression, and enabled precise crystal structure refinements at 6 pressures between 1.2 and 2.1 GPa. A special XRD data processing methodology was developed and applied to reliably trace small changes in molecular geometry and disorder of hydrogen atoms at elevated pressure. Our results show presence of an anomaly in pressure dependence of the C12 elastic constant. This anomaly was observed in earlier Brillouin experiments, but has never been interpreted. Our results show pressure-induced changes in the probability distribution of water molecules assuming different orientations, which is contrary to the assumption of the full disorder allowed by ice rules. The new data processing approach enabled us also to determine trends in pressure dependencies of bond lengths and angles. We found that all the O-H bonds extend with pressure as a consequence of increasing strength of hydrogen bonds. We also found that only two out of four symmetry independent O-H distances reflect the above mentioned anomaly in compression behavior. Our results shed new light on the understanding of the nature and role of proton disorder in ice VI as well as present new insight into interpretation of Brillouin experiments. Apart from structural data we present results regarding phase equilibria in the water-formic acid system that allow us to better understand nature and strength of hydrogen bonding interactions in solution.

#### MR71B MCC: 122 Sunday 1020h

##### Elasticity and Constitution of the Earth's Interior II (joint with S, T, V, DI)

Presiding: T Duffy, Princeton University

#### MR71B-01 1020h INVITED

##### Inner Core 50 Years Later

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In 1952, Francis Birch predicted that the inner core should be solid with shear velocity of 3.4 km/s and the Poisson ratio of 0.45. Almost 20 years later, observations of normal modes with sensitivity to inner-core properties confirmed the solidity of the inner core (Dziewoński and Gilbert, 1971). Although Birch's prediction was based upon an incorrect assumption that the bulk modulus and the density do not change between the outer and inner core, the best estimates of the shear velocity (3.53 km/s) and Poisson ratio (0.44) are extremely close to Birch's values.

Inner-core anisotropy in the form of transverse isotropy introduced a new level of complexity; it is required to explain both travel-time (Morelli et al., 1986) and normal-mode (Woodhouse et al., 1986) data. Such complexity imposes heavier demand on the spatial distribution of raypaths, and these are difficult to meet because of the non-uniform distribution of sources and receivers. The additional assumptions, to make up for the missing data, have led to highly divergent estimates of the strength and radial dependence of anisotropy. We now believe that most of the travel-time and normal-mode observations can be explained by a relatively simple model of anisotropy (Ishii et al., 2002a), a model that does not vary with radius and is characterized by 2ξ term (where ξ is the angle between the raypath and the axis of symmetry of transverse isotropy) with the slow direction in the equatorial plane. Inferences of complexities such as an isotropic layer near the inner-outer core boundary (Song and Helmberger, 1998) or a longitudinal variations at hemispheric scales (Tanaka and Hamaguchi, 1997) are either not necessary or not robust (Ishii et al., 2002b). However, travel-time data from nearly antipodal distances (173°-180°) indicate that the anisotropy changes substantially at about 300 km radius: it is dominated by the 4ξ term with the slowest direction ~ 45° from the equatorial plane (Ishii and Dziewoński, 2002; also this meeting). We call this distinct new region of the Earth the "inner-most inner core".

#### MR71B-02 1035h INVITED

##### The State of the Earth's Core

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