

are similar to those of pure Fe melt [1]. These results imply that the effect of S content in the Earth's outer core may not be so strong.

[1] Alfe D., Kresse G., Gillan M. J., *Phys. Rev. B*, 2000, **61**, 132.

Keywords: viscosity, high pressure, Fe-FeS melt

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Accommodation Mechanism of Kr Trapped in Terrestrial and Planetary Materials

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The trapping or adsorption of noble gases in minerals has great interest in solving the "missing Xe" problem. We focus on Kr slightly trapped in terrestrial and planetary materials in a *ppb* level. In this study we have examined the solubility and local structures of Kr trapped in (1) minerals such as quartz, olivine, coesite, stishovite, olivine and wadsleyite and (2) synthetic model-samples of carbon fine powder, silica gel and zeolite.

Samples were synthesized in Kr-atmosphere at high pressure and high temperature, by using the Kawai-type high-pressure apparatus. Kr-doped silica gels, and partly MgO, were used as starting materials and sealed in Pt-capsule to prevent Kr-escape under high pressure. Degassing of Kr for all samples were measured as a function of temperature up to 1850°C by the mass spectrometer. The results showed that the degassing of silica gel causes at temperatures between 500°C and 800°C. On the other hand, Kr-doped natural olivine has the degassing, giving two peaks observed at 800°C and 1800°C. It is notable that small amount of Kr still remains in olivine even at 1800°C. XAFS measurements in the fluorescence mode were made to determine the atomic distances between Kr and the neighboring atoms and the local structures around Kr atoms. There are structural differences in the Kr coordination between terrestrial materials and model samples.

Keywords: gas-solid interaction, high pressure, meteorite

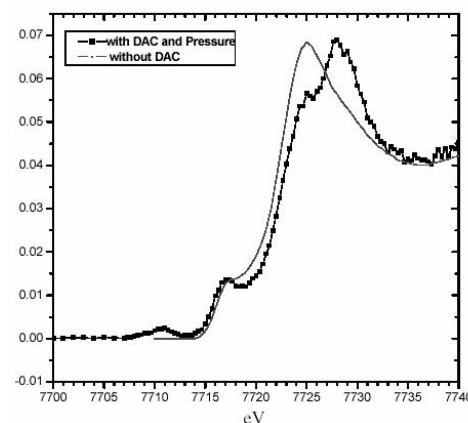
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Pressure-induced Electron Transfer in Cobalt-iron Prussian Blue Complex Studied by RIXS

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Resonant Inelastic X-ray Scattering (RIXS) recently has become one of the most advanced techniques that probe electronic excitations in solids, combining both advantages of a high resolution and bulk sensitivity. In our measurement, we attempted to study the charge transfer in $K_0.2Co_{1.4}[Fe(CN)_6] \cdot xH_2O^{[1]}$ as a function of pressure by RIXS. The photoinduced magnetization at low temperature in Co-Fe Prussian blue analogues was explained by the presence of diamagnetic Co(III)-Fe(II) low spin pairs, this step can be pushed by low temperature or high pressure^[2]. Then the photoinduced electron transfer from Fe(II) to Co(III) can be happened. We had performed a preliminary Resonant inelastic x-ray scattering (RIXS) studies on the $K_0.2Co_{1.4}[Fe(CN)_6] \cdot xH_2O$ at 0.33GPa under Diamond Anvil Cell(DAC) to study and confirmed the charge transfer behavior successfully during this time. From the comparison of the title compound at 0.33GPa pressure and ambient pressure, we can see the Co(III) ratio increase very clearly, that mean the charge transfer Fe(III)-Co(II)→Fe(II)-Co(III) happened. This confirms the outstanding resolving power of RIXS and fruitful quantitative determinate the ligand field strength and also the Co(II)/[Co(II)+Co(III)] ratio can be determined from this kind of measurement. In here, we will present the measurement results on Iron



K-edge and Cobalt K-edge partial fluorescence yield mode (PFY) by RIXS experiment to get the ligand field strength and charge transfer information related with pressure.

Figure 1. Cobalt K-edge X-ray absorption spectra with 0.33GPa and without pressure by partial fluorescence yield mode (PFY)

[1] Verdager M., *Science*, **272**, 698 [2] Ksenofontov V., Levchenko G., Reiman S., Gutlich P., *Phys. Rev. B*, 2003, **68**, 24415.

Keywords: inelastic X-ray scattering, cobalt-iron prussian blue, pressure

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ALS Beamline 12.2.2, A High-pressure X-ray User Facility at the US-West-Coast

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Beamline 12.2.2 is a hard-X-ray beamline making use of the radiation spectrum ($\sim 5 \text{ keV} < E < 40 \text{ keV}$) emitted from a superconducting bending magnet. The radiation is conditioned using a plane parabola collimating mirror (M1), a Kohzu monochromator assembly with a Si(111) crystal ($E/\Delta E \sim 7000$) or W/B₄C multilayer ($E/\Delta E \sim 100$), and a toroidal focusing mirror (M2) with variable focusing distance, before it is directed into the experimental hutch

In the hutch, two experimental stations facilitate a variety of high-pressure experiments, focusing on *in-situ* high-pressure - high-temperature powder diffraction, EXAFS and X-ray imaging. End-station 1 is presently optimized for combining externally heated diamond anvil cells (DACs) with powder X-ray diffraction and can be used for high-pressure EXAFS experiments as well. End-station 2 is designed for *in situ* laser heating of DACs using a set of Kirkpatrick-Baez mirrors for secondary focusing (spot size at sample = $0.01 \times 0.01 \text{ mm}^2$) as well as a double sided YLF laser heating system

Samples are placed on a kinematic mount equipped with 2 rotation stages as well as 4 linear stages in order to center the DAC reproducibly at the same reference position. The overall accuracy of the distance calibration is $\sim 0.01 \text{ mm}$ corresponding to a theoretical $\Delta d/d$ of 10^{-4} at a sample to detector distance of 100 mm. Further benchmarks as well as examples of current research will be presented.

Keywords: synchrotron X-ray instrumentation, high pressure crystallography, mineralogy geophysics high pressure

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High Pressure Single-Crystal Neutron Diffraction of DKDP

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The initial results of a new initiative of the Paris-Edinburgh (PE) collaboration to develop single-crystal technology for high-pressure neutron diffraction are presented. Single-crystal neutron diffraction data have been collected from D₂KPO₄ at pressures up to 7.5 GPa. At 4.2 GPa it has been suggested by Endo [1] that the hydrogen bond lengths elongate and the proton centres in a single minimum between the two oxygen atoms. However, these results were obtained using x-

rays which are insensitive to the scattering of light atoms and hence neutron diffraction data on a single crystal sample are essential in determining precisely the atomic co-ordinates during this possible transition. Previously only data up to 2.0 GPa have been collected using single crystal neutron diffraction. We present structures determined from a single crystal of 4 mm³ using time-of-flight Laue diffraction on the SXD instrument at the ISIS Facility at Rutherford Appleton Laboratory in the UK.

[1] Endo S. et al, *Nature*, **340**, 452, 1989.

Keywords: high pressure, single crystal diffraction, neutron diffraction

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Compression of Oxygen Vacancy Type Al-bearing MgSiO₃ Perovskite

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MgSiO₃ perovskite is believed to be a dominant constituent of the Earth's lower mantle. Experimental results on the effect of Al on the compressibility of MgSiO₃ perovskite have been pretty controversial. Two kinds of the Al substitution mechanisms are expected: 2Al³⁺ = Mg²⁺ + Si⁴⁺ and 2Al³⁺ = 2Si⁴⁺ + (as a vacancy site) O²⁻ [1]. Theory predicts that the latter mechanism significantly increases the compressibility [2].

Recently, Kojitani et al (2005) demonstrated the structural differences between these two types of Al-bearing MgSiO₃ perovskite on the basis of Rietveld analyses. In this work, the volume compression measurements were performed on the oxygen vacancy type Al-MgSiO₃ perovskite by using synchrotron radiation x-rays. Data were collected under hydrostatic conditions using helium pressure transmitting medium. Preliminary results show that the isothermal bulk modulus is reduced due to the incorporation of Al₂O₃ in perovskite with oxygen vacancy.

[1] Navrotsky A., Shoenitz M., Kojitani H., Xu H., Zhang J., Weidner D. J., Jeanloz R., *J. Geophys. Res.*, 2003, **108-B7**, ECV2-1. [2] Yamamoto T., Yuen D.A., Ebisuzaki T., *Earth Planet. Sci. Lett.*, 2003, **206**, 617.

Keywords: perovskite, high pressure mineralogy, synchrotron X-ray diffraction

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New High-pressure Phase of Calcite at Room Temperature

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A new high-pressure phase of calcite (CaCO₃) has been found by single-crystal X-ray analyses. Recently we determined the crystal data of calcite III (monoclinic C-centered lattice, Z=8) using single-crystal X-ray diffraction methods at 300K and pressures between 2.3 and 4.1 GPa [1]. The present studies have been carried out to determine the crystal data of calcite at room temperature and higher pressures.

The samples are single crystals of natural calcite from Creel, Chihuahua, Mexico. For high-pressure experiments, the Merrill-Bassett type diamond-anvil-cells were used. Pressures were estimated based on the ruby-fluorescence method. X-ray diffraction measurements were performed using an Enraf-Nonius CAD-4 diffractometer with MoK α radiation monochromatized by graphite. A film cassette for a flat imaging plate was attached on the diffractometer.

On the oscillation photographs taken at 2.5GPa, all the reflections could be assigned as calcite III being composed two twinned individuals. By increasing the sample pressure to 4.5GPa, several reflections disappeared. Then remained reflections were re-indexed and the cell parameters were determined with the angular data of 25 reflections. The triclinic unit-cell are $a=3.855(3)$, $b=4.815(2)$, $c=6.165(5)\text{\AA}$, $\alpha=84.10(4)$, $\beta=72.15(5)$, $\gamma=88.73(5)$, $Z=2$ at 300K and

4.5GPa. The cell parameters were determined at pressures between 4.3 and 5.6GPa. Thus the phase transition from calcite III to the new phase may occur at 4.1-4.3GPa.

[1] Hagiya K., Matsui M., Kimura Y., Akahama Y., *J. Mineralogical and Petrological Sciences*, 2005, **100**, 31.

Keywords: calcium compounds, carbonates, high-pressure phase

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High-pressure Single-crystal Study of Chlorite

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Chlorite is a major constituent of hydrated oceanic crust, and may represent an important water carrier in subducting slabs. In this context, its stability under high pressure has relevant implications on rheology and melting behaviour of mantle rocks.

The structural effects of pressure on a natural chlorite from Val Malenco (Italy) [clinoclhor, polytype IIb-4, S.G. C-1, pseudomonoclinic metric, composition (Mg_{7.82}Al_{3.36}Fe²⁺_{0.52}Fe³⁺_{0.30})(Si_{7.70}Al_{0.30})O₂₀(OH)₁₆] have been studied by X-ray diffraction on single-crystal mounted in a DAC. Pressure was calibrated through the EoS of α -quartz.

Structural refinements were performed at 0, 0.8, 1.8, 2.7, 3.5, 4.4, 5.1 GPa with intensity data collected on a CCD Xcalibur diffractometer (Oxford Instr.) equipped with monochromatized MoK α . Lattice parameters were measured with the point-detector mounted on the same instrument.

The compressibility data of chlorite (bulk modulus K= 83(1) GPa, K'= 4) are in fair agreement with data based on powder neutron [1] and synchrotron diffraction methods [2,3]. Axial moduli are 102(2), 97(3) and 63(1) GPa respectively for a , b and c axes. The main structural deformations affect the interlayer region where the hydrogen bonds are relevant to the structural properties of the phase. The OH-O distances decrease of about 4% in the 0-5 GPa range. Work is in progress and further details will be presented at the conference site.

[1] Welch M.D., Marshall W.G., *Am. Mineral.*, 2001, 1380. [2] Pawley A.R., Clark S.M., Chinnery N.J., *Am. Mineral.*, 2002, 1172. [3] Welch M.D., Crichton W.A., *Eur. J. Min.*, 2002, 561.

Keywords: high-pressure diffraction, chlorite, compressibility

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Research of Nanostructured X-ray Amorphous Minerals by AFM

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Among products of geological processes the noncrystal substances are distributed. Before among geomaterials the basic attention was given to research of crystal mineral substance and microdisperse mineral phases. Progress of methods of research has allowed to find out and considerably to expand last decade ideas about nanodisperse mineral phases. Their study causes interest due to active influence of nanoconstitution on properties, to detection of some new unique forms in mineral substances.

In our work an attempt of search characteristics of nanoconstitution of various noncrystal mineral substances will be undertaken. Among them simple substances (ex., fullerene shungite Karelia), oxides (ex., natural photonic crystals – opals), complex substances (ex., solid hydrocarbons). Nanoconstitution visualized on a fractures surface off by atomic force and electronic microscopies. The quantitative superstructural data were received by a combination of microscopic researches with diffractational and statistical-geometrical analysis of morphological features.

As a result of the carried out researches local and global characteristics of supermolecular structures, in particular, a degree of orderliness of a relative positioning of elements nanostructures have been established. The multilevel fullerene-like structure of shungite