[7] let us envisage that the chemistry of Xe with oxygen at extreme conditions could be flourishing.

We have thus explored the reactivity of Xe with water up to 80 GPa by using a laser-heated diamond-anvil cell combined with in situ x-ray diffraction measurements. Formation of a compound is indeed observed at conditions that could occur in the interiors of ice-rich giant planets Uranus and Neptune. To complement the x-ray diffraction data, theoretical calculations have been carried out to determine the molecular structure of the newly found compound. These results hence add another example of noble gas sequestration in giant planets, as recently proposed for Ne [8].

[1] W. Grochala, *Chem. Soc. Rev.* **2007**, *1632*, 36. [2] R.B. Gerber, *Annu. Rev. Phys. Chem.* **2004,** *55*, 55. [3] A.I. Klatz, V.A. Apkarian, *J. Phys. Chem.* **1990**, *94*, 6671-6678. [4] C. Sanloup et al., *PNAS* **2002**, *99*, 25. [5] M. Pettersson et al., *Eur. J. Inorg. Chem.* **1990**, *505*, 729. [6] L. Khriachtchev et al., *JACS* **2008**, *130*, 6114. [7] C. Sanloup et al., *Science* **2005**, *310*, 1174. [8] H.F. Wilson, B. Militzer, *Phys. Rev. Lett.* **2010**, *104*, 121101.

**Keywords: noble gases, high pressure, water**

## MS.19.5

*Acta Cryst.* (2011) A**67,** C58

**Structural, magnetic and electrical properties of iron-hydride**

Naohisa Hirao,[a] Yasuo Ohishi,[a] Takahiro Matsuoka,[a] Takaya Mitsui,[b] Ken-ichi Takemura,[c] Katsuya Shimizu,[d] Akihiko Machida,[b] Katsutoshi Aoki,[b] [a]*Japan Synchrotron Radiation Research Institute (JASRI/SPring-8), Sayo, Hyogo, (Japan).* [b]*Japan Atomic Energy Agency (JAEA), Sayo, Hyogo, (Japan).* [c]*National Institute for Materials Science (NIMS), Tsukuba, Ibaraki (Japan).* [d]*Research Center for Quantum Science and Technology under Extreme Conditions, KYOKUGEN, Osaka-university, Toyonaka, Osaka (Japan).* E-mail: ohishi@spring8.or.jp

Iron-hydride ($FeH_x$) is synthesized at 3.5 GPa in dense hydrogen at room temperature, and is unique in its melting curve, its phase relation, and its magnetism [1]. The hydrogenation of iron leads to dramatic changes in the volume and electronic structure of host metal Fe, resulting in the ferromagnetic double hexagonal-close-packing (dhcp)-$FeH_x$, in contrast to the nonmagnetic hcp-Fe. By energy-domain synchrotron radiation $^{57}$Fe-Mössbauer spectroscopy technique [2], we observed the magnetic transition from ferromagnetic to nonmagnetic state on the dhcp-$FeH_x$ near 27 GPa without structural phase transitions [3]. In order to understand the electronic and magnetic properties of FeHx, it is important to know detailed *P-T* phase diagram and physical properties of each phase. Previously we have performed high-*P* and Low-*T* XRD experiments going through various *P-T* paths and reported the emergence of the new structural phase of hcp-$FeH_x$, by the hydrogenation of hcp-Fe on pressure unloading process in high-pressure $H_2$ condition.

In this study, we extended our study to high-temperature region, iron-deuteride ($FeD_x$), and electrical properties. The X-ray diffraction experiments were performed at SPring-8/BL10XU in combination with laser heating system and DACs. When dhcp-$FeH_x$ at 16.6 GPa was heated to 1400 K – 1500 K, the structure transformed to fcc via an unknown intermediate phase. The fcc phase was found to be quenchable to room temperature by rapid cooling. In the study of $FeD_x$, it was observed that dhcp- and hcp-$FeD_x$ could be synthesized at just the same conditions with the case of $FeH_x$. FeH was found to be a metal by electrical resistance measurements. We had also observed the changes of *P* and *T*-dependence of electrical resistance that can be thought to reflect the ferro-magnetism collapse reported at corresponding pressures. Currently, we are investigating the possible tuning of hydrogen composition (H/Fe ratio) by the application of electric current.

This work was supported by the New Energy Development Organization (NEDO) under Advanced Fundamental Research on Hydrogen Storage Materials.

[1] Y. Fukai, *The Metal-Hydrogen System*. Springer, Berlin, **2000**. [2] T. Mitsuii, M. Seto, N. Hirao, Y. Ohishi, Y. Kobayashi, S. Higashitaniguchi, R. Masuda, *Japanese Journal Applied Physsics* **2007**, *46,* L382-L384. [3] N. Hirao, Y. Ohishi, T. Mitsui, T. Matsuoka, M. Seto, K. Aoki, K. Takemura, H.Yamagami, in preparation.

**Keywords: iron-hydride, structural phase transition, high-pressure**

## MS.20.1

*Acta Cryst.* (2011) A**67,** C58

**Disordered and incommensurate crystal structures by transmission electron microscopy - some examples**

Cristiano Ferraris *Laboratoire de Minéralogie et Cosmochimie, UMR7202, Muséum National d'Histoire Naturelle, CP 52, 61 Rue Buffon, 75005 Paris (France).* E-mail: ferraris@mnhn.fr

Ideal crystals are defined as arrays of atoms that are periodically arranged according to a Bravais lattice; the atomic structures of these edifices are completely characterized by three basis vectors fixing the translational symmetry and the coordinates of the atoms in the unit cell. In this scenario Transmission Electron Microscopy (TEM), apart the possibility of investigating nano-crystals provides information which does not substantially differ from those obtained by X-ray and neutron crystallography.

However, crystalline 'order' can be achieved in different ways than by Bravais translational symmetry, resulting in aperiodic crystals. To investigate aperiodic structures, the stronger interaction of the electrons with matter allows obtaining information about subtle crystallographic details that are usually not available by X-ray diffraction. Using TEM many different types of information can be collected from very small volumes (even down to less than 1 nm) from one single grain. Apart from the structural data obtained via selected area electron diffraction (SAED), bright field (BF) and dark field (DF) imaging and high-resolution transmission electron microscopy (HRTEM), more information can be gathered from this small volume by other innovative techniques. Convergent beam electron diffraction (CBED) discloses information on local symmetry of the structure, and high spatial resolved spectroscopic techniques such as electron energy-loss spectroscopy (EELS) allow studies of local cell composition and light elements determinations, the electronic structure of the crystal and the oxidation state of the transition metals.

As example, an innovative TEM investigation on the co-existence of crystallographically commensurate and incommensurate domains within a $(Ba,Sr)_2TiSi_2O_8$ fresnoite structure is presented, particularly the dependence of modulation on local composition. It is shown that perturbations of the average fresnoite structure, determined from 'single crystals', are in some cases better described as nanometric domain intergrowths where departures from an ideal stoichiometry are cause of incommensuration. Evidences of chemical differentiation are obtained from both SAED patterns and HRTEM images. Simulations and Fourier reconstructions of HRTEM images show that modulation can change within relatively small crystal structure volumes.

**Keywords: incommensurate structures, electron diffraction, HRTEM**