5 Extreme condition project

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5-1 Introduction

Materials under pressure and temperature show many interesting behaviors unlike those under ambient conditions. Such extreme conditions in nature are found inside our planet Earth and other planets. Regarding the materials in the Earth, basic information for the mantle minerals and core materials is the phase relations, on which much data already exists for various minerals and iron alloys using large-volume presses and diamond anvil cells (DAC) combined with in-situ X-ray diffraction at synchrotron facilities. However, the physical properties of each phase under the conditions in the mantle are not always known. Iron and hydrogen are particularly important elements for understanding the dynamics and evolution of the Earth. Iron is the heaviest element among the major mantle minerals and greatly affects gravitational phenomena in the mantle. Spectroscopy measurements have recently shown that iron exhibits complicated behaviors at high P-T [1-3]. Valence change, high-spin to low-spin transition, optical characteristics, and partitioning behavior among co-existent phases are important properties of iron in the Earth's deep interior. The changes of state of iron cannot, however, be obtained directly from conventional diffraction experiments developed so far. Therefore, we need to design a new beam-line that can trace the electronic state and spin state of iron as well as structural information from the same sample under the same conditions. For this purpose, Mössbauer measurement was chosen as the first step. X-ray imaging of DAC samples, absorption experiments and high-speed diffraction by CCD detector will also be promising for further studies on, for example, phase separation, rheological properties, valence change, and kinetics of phase transition.

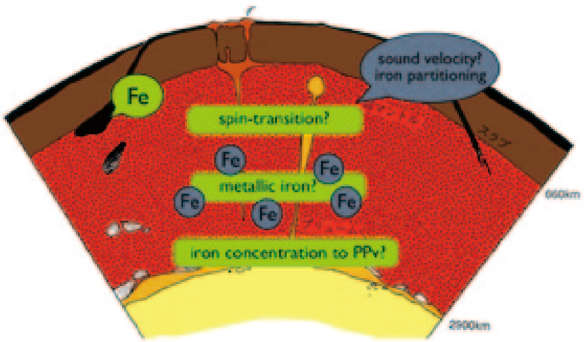

Fig. 1 Possible changes of state of iron in the mantle minerals and their effect on the structure and dynamics in the Earth.

On the other hand, water and hydrogen are major components in the universe. The stability fields of hydrous minerals, hydrous magma, and hydrogen in crystals are important problems in the Earth. A small amount of water has a strong effect on the phase boundary, melting temperature, electrical properties and rheological properties of the Earth's materials. These phenomena are considered to be essential in some of the dynamic processes in the mantle (Fig. 2). We have attempted direct observation of hydrogen because it is almost invisible by X-rays, and are starting a new project to perform neutron studies under high P-T conditions.

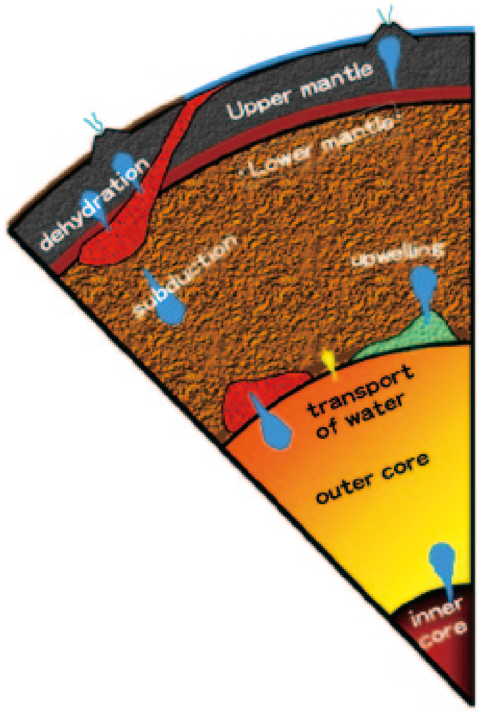

Fig. 2 Water (hydrogen) circulation, transport, and storage in the Earth.

In this project, we will develop a new in-situ technique to investigate physical and chemical properties of Earth and planetary materials including iron and hydrogen under extreme conditions. Projects for both iron and hydrogen were in the construction phase in FY2010 except for X-ray diffraction experiments, and both X-rays and neutrons, and in some cases also muons, will be utilized complementarily with high-pressure apparatus in this project.

5-2 Construction of the multi-purpose high-PT station at AR-NE1

The laser-heated diamond anvil cell (LHDAC) is a convenient and powerful tool for generating the high pressures and temperatures that exist in the Earth's core and was developed originally at BL-13 in KEK-PF for in-situ X-ray diffraction experiments. After the system was moved to AR-NE1A in FY2009, construction has progressed under Dr. Kikegawa to create a multi-measurement system consisting of a diffraction unit and Mössbauer spectroscopy unit under high pressure. Other combinations of measurements, such as imaging and diffraction, diffraction and absorption, imaging and Mössbauer, will be feasible because the diamond for an anvil material also works as an optical window to introduce a wide range of light into the compressed sample. Some of the combinations are expected to enable simultaneous measurement of the same sample. Station NE1A has two monochrometers: one is a micro-channel water-cooled Si(111) type for diffraction measurement and the other is a high-resolution type for high-resolution type. The X-ray source introduced from the insertion device (MPW) is monochromatized and focused by K-B type optics. The system is suitable for both Mössbauer and diffraction studies in alternative mode (Fig. 3). However, these optics need to be finely tuned every time after changing the experimental mode, which used to take several days in the initially installed condition. We have improved the system in FY2010 to allow fast switching of X-ray optics in the upper stream and experimental stages. After installation of the rail base to escape and fix the position of the experimental stage and digital axis control system, the switching time was dramatically shortened to half a day.

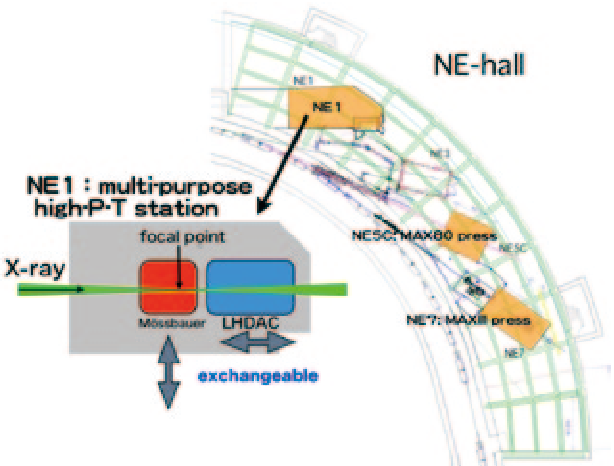

Fig. 3 Multi-purpose high P-T station, NE-1A, at the AR-NE hall. The experimental stage of the low temperature or large-volume compression system for Mössbauer spectroscopy can be exchanged to a high P-T DAC system in a few hours.

The system will be developed further in the near future to add a new measurement system to obtain independent information for the same sample. The planned capabilities of this experimental module are shown in Fig. 4.

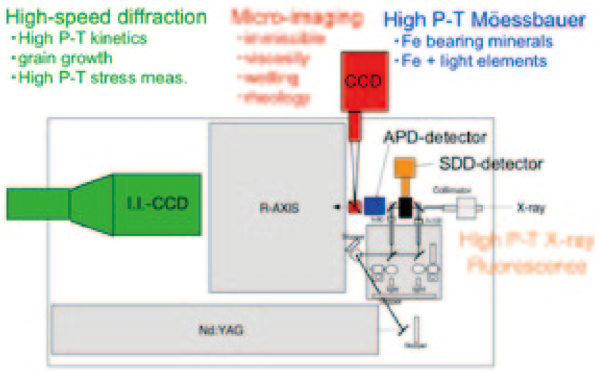

Fig. 4 New design for high P-T experimental stage for LHDAC at the planning phase. At present, the Mössbauer spectroscopy and micro-imaging system are under construction with a slightly modified laser-heating system for in-situ diffraction experiments.

5-3 Hydrogen in iron-nickel alloy

Iron-nickel alloy with light elements is considered to be the main constituent of the Earth's core. Hydrogen is one of the important candidates of the light elements, considering the reaction between iron and water in the early Earth. The effects of nickel, however, have not been evaluated yet. Nickel takes an fcc structure at the temperatures and pressures studied, compared with the several polymorphs of iron. Furthermore, the solution mechanism of hydrogen into metal is different in a lattice structure. Thus, it is important to study the reaction of water and iron-nickel alloy. We performed an in-situ X-ray diffraction study to elucidate the effect of nickel on the water-iron system. The sample configuration of the typical LHDAC experiment is shown in Fig. 5.

The experimental results are summarized in Fig. 6. (Fe,Ni)OOH, the structure of which is the same as that of delta-AlOOH, was observed up to 32 GPa and 1400 K, while (Fe,Ni)O was produced above 40 GPa at high temperature. The stability field of the oxyhydroxide plus oxide expands significantly to the higher pressure region, which covers a 1000-km depth of the magma ocean. If iron-nickel alloy were to react with water in the deep mantle, the reaction products would be oxyhydroxide rather than oxide, which would easily react with the surrounding hydride minerals. In that case, the total hydrogen in the core would be much less than in the case of pure iron. Moreover, as the in slope of the reaction boundary is steeper, the iron-nickel alloy cannot easily react with water at high pressure. This also influences the amount of hydrogen.

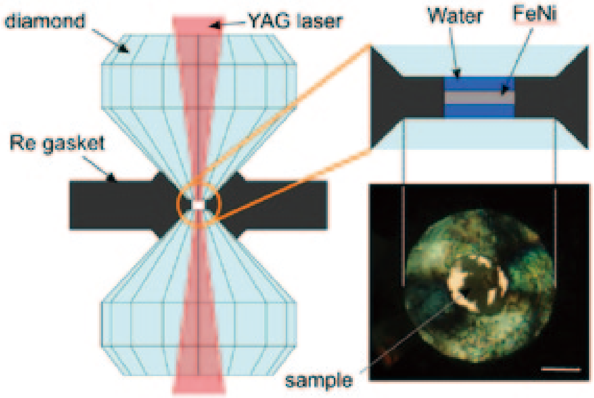

Fig. 5 Sample configuration of the LHDAC experiment for this study. Water is actually solid (ice) in this condition.

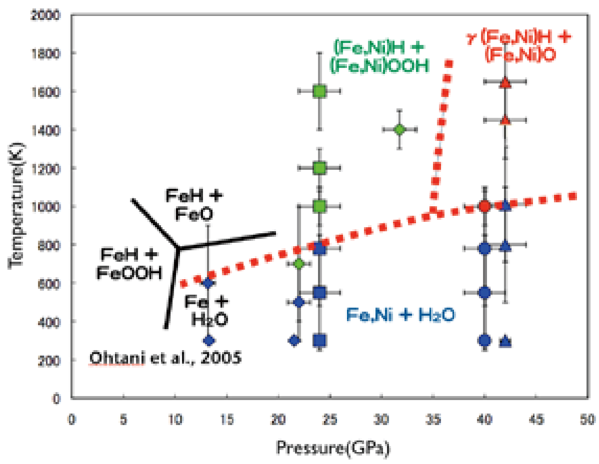

Fig. 6 Reaction diagram of water-Fe-Ni system. The phase boundary between hydrate and hydride has shifted to the high-pressure region, meaning enlargement of the stability field of (Fe,Ni)OOH, which is compatible with the dense mantle phase.

5-4 Possible water-rich phase in the lower mantle

Mid-ocean ridge basalt (MORB glass) is the primary component of subducting slabs which transport water into the deep mantle through oceanic trenches. We investigated the hydrous phase in the MORB-water saturated system to clarify the hydrous phase with maximum water content in the crystal structure under the lower mantle conditions using LHDAC with the in-situ X-ray diffraction method at AR-NE1A. The typical change in X-ray pattern is shown in Fig. 7.

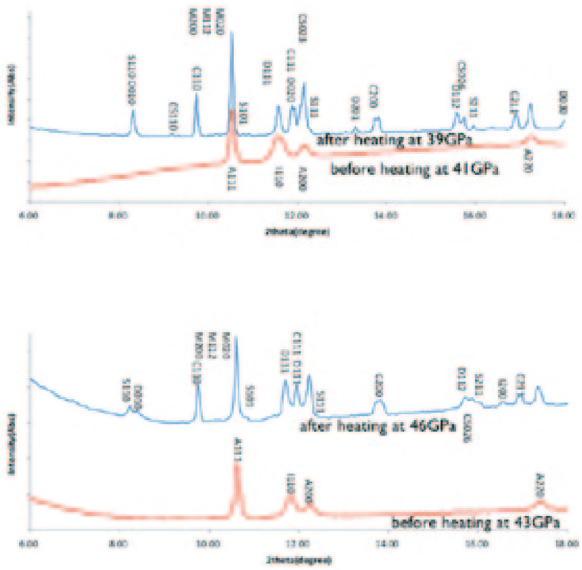

Fig. 7 Examples of in-situ X-ray diffraction obtained from water-MORB reaction under high pressure and temperature.

The nominally hydrous phase formed in this system was phase D, which includes almost 20% water. The decomposition boundary in this study was located at a significantly higher pressure than that suggested from early work by Shieh et al., 1998 (Fig. 8). Our results indicate that the water content may be enhanced greatly if some hydrous phase survives through the subduction process and generates a locally water-rich condition in the lower mantle.

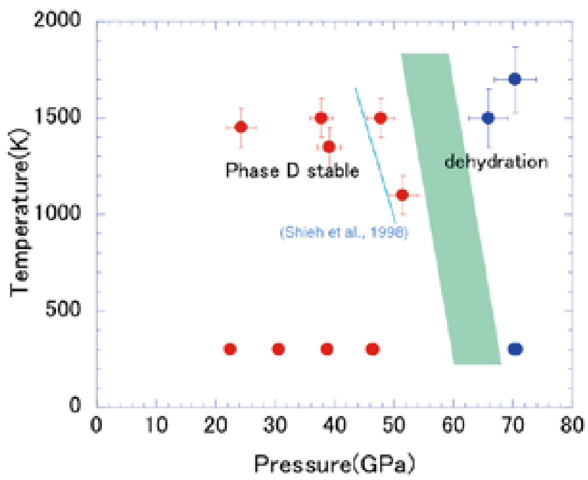

Fig. 8 New design for high P-T experimental stage for LHDAC at the planning phase. At present, a Mössbauer spectroscopy and micro-imaging system is under construction with a slightly modified laserheating system for in-situ diffraction experiments.

5-5 New high-pressure apparatus for neutron diffraction study

For Earth and planetary hydrogen-related studies, the high-pressure joint team led by Prof. Yagi and Prof. Kagi of the University of Tokyo in Japan has collaborated to construct a new apparatus to enable direct observation of hydrogen in materials under high pressures and high temperatures corresponding to the conditions in the Earth's mantle. This project includes precise determination of hydrogen position in crystals, the structure of wet magma and other liquids, and neutron radiography. The experimental station has been built [4-6] at MLF-BL-11 at J-PARC, and is called "PLANET: Pressure Leading Apparatus for NEuTron diffraction" (Fig. 9). In 2010, a newly designed high-pressure press was constructed and will be installed at PLANET. The press, named "ATSUHIME", has a six-axis loading system to generate ultra-high pressures in sufficiently large volume to conduct diffraction experiments. Each axis is an independent 500-ton press and therefore the press can be used not only to generate high pressures but also for uniaxial compression or deformation experiments. Actual operation will start in 2012, because the 3/11 earthquake hit this area just before the ceremony for introducing the first beam to the station with primary constructing members, although fortunately no serious damage was found in the system.

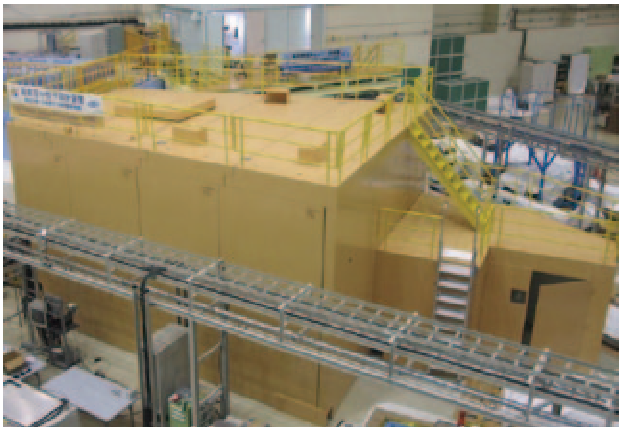

Fig. 9 New high-pressure neutron beamline PLANET, in which the six-axis large volume press will be installed in FY 2011.

5-6 Abnormal compression behavior of silica in helium

SiO2 glass has a significant amount of interstitial voids in the structure. Gas solubilities in silicates are usually expected to be small under high pressure due to compaction of voids. Professor Funamori's group performed unique experiments on the silica-helium system using a diamond anvil cell at BI-18C of the Photon Factory [7]. Volume measurements of SiO_2 glass indicate its less compressible nature than normal when compressed in helium. The volume in helium at 10 GPa is surprisingly close to the normal volume at 2 GPa. Their X-ray diffraction and Raman scattering measurements indicate that there is a relation between voids in the structure and helium, suggesting that helium penetrates into the sample. Details of this study can be found also in the highlight section of Photon Factory Activity Report 2011.

References

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