（ ※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。 ）

CROSS TOKAI Experimental Report J-PARC	承認日Date of Approval 2014/03/24 承認者Approver Takanori Hattori 提出日Date of Report 2014/03/24
課題番号 Project No. 2013B0030 実験課題名 Title of experiment In Situ Neutron Diffraction Study of the Crystal and Magnetic Structures of FeD Synthesized by Deutration of Fe at High Temperature and High Pressure 実験責任者名 Name of principal investigator Katsutoshi Aoki 所属 Affiliation Tohoku University	装置責任者 Name of responsible person Takanori Hattori 装置名 Name of Instrument/（BL No.） PLANET(BL11) 実施日 Date of Experiment 8-12th of March, 2014

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

Iron, Fe (2 mm in diameter, 1.9 mm height)

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

Ferromagnetic dhcp-FeDx was synthesized by deuterization of fcc-Fe at about 1000 K and 7 GPa, and then cooling synthesized fcc-FeDx to convert it to dhcp-FeDx. The specimen obtained at 300 K and 6.5 GPa provided diffraction profiles from dominant component of dhcp-FeDx, minor component and residual hcp D2. The high temperature and high pressure cell used for iron hydride formation is shown in Figure.

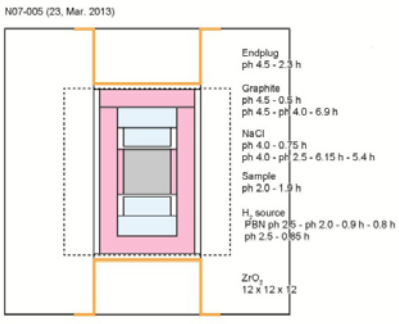

Fig. 1 Cross sectional view of deuterization cell

Byproduct of hcp-FeDx was obtained unexpectedly. The phase diagram of Fe-H(D) system shows presence of bcc, fcc, and dhcp phases as iron hydride and we never observed hcp hydride formation in preliminary experiments on Fe-D system by synchrotron radiation X-ray diffraction. We hence tried to convert a metastable hcp-FeDx to stable dhcp-FeDx by heating the specimen up to 573 K at a fixed pressure of 6.5 GPa. However, hcp phase persisted during thermal annealing and

2. 実験方法及び結果（つづき） Experimental method and results (continued)

a single phase of dhcp-FeDz was failed.

The starting material was an iron rod (Nilaco, purity 99.5%) 2.0 mm in diameter and 1.9 mm in height. The sample was placed in a deuterium sealing capsule made of NaCl along with disks of AlD_3, internal deuterium source. The dimensions of the sample and the disks of internal deuterium source were designed so that the molar ratio D/Fe was1.4; excess amount of deuterium was confined in the sealing capsule. The sample and the disks of internal deuterium source was separated by pyrolytic boron nitride disks which can permeate deuterium and avoid contamination of the sample. The deuterium sealing capsule was located in a cylindrical graphite heater which generates high temperature by supplying electric current through top and bottom electrodes. The cubic pressure medium made of ZrO_2 with 12 mm in edge length was used to contain the above motioned cell parts. The high temperature and high pressure cell used for iron hydride formation is shown in Fig. 1.

We collected a diffraction profile of the mixed state of iron hydride at 300 K and 6.5 GPa by about 8 hours accumulation. The statics turned insufficient to analyze the crystal structure of dhcp-FeDx including magnetic moments. Longer accumulation time is required for structural analysis. During diffraction measurement with a multi anvil press, we faced malfunction in press operation and were forced to stop measurement. A profile measured at 300 K and 6.5 GPa is shown in Fig.2.

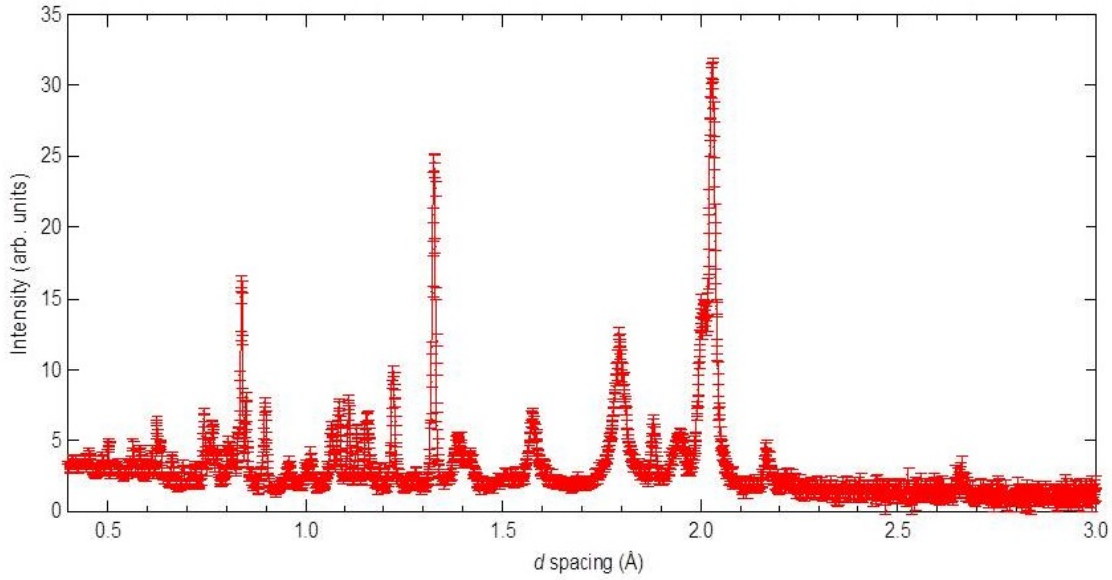

Fig. 2. Neutron diffraction profile of FeDx at 300 K and 6.5 GPa