（ ※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 2014/6/3 承認者 Approver Takanori Hattori 提出日 Date of Report 2014/5/28
課題番号 Project No. 2014A0015 実験課題名 Title of experiment In Situ Observation of Superabundant Vacancy Formation and its Ordered Structure in Fe-D Solid Solution 実験責任者名 Name of principal investigator Katsutoshi Aoki 所属 Affiliation Tohoku University	装置責任者 Name of Instrument scientist Takanori Hattori 装置名 Name of Instrument/（BL No.） BL-11 High Pressure Neutron Diffractometer (PLANET) 実施日 Date of Experiment 2nd – 7th of May, 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.

Sample: Mixed powder of iron deuteride FeD_x and magnesium oxide MgO

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.

A mixed powder of iron Fe and magnesium oxide MgO was compressed and heated with internal deuterium sources AlD_3 to prepare solid solution of iron deuteride FeDx with an fcc metal lattice. After formation of fcc FeDx, the temperature and pressure were kept at ~1000 K and ~5 GPa, respectively, and time resolved diffraction profiles were collected over ten hours. The lattice constant of the fcc metal lattice showed gradual shrink likely due to vacancy formation and reached a steady value after several hours (Fig. 1). The behavior of the metal lattice is in agreement with those reported for transition metals hydrides and indicative of formation of superabundant vacancies, SAV. Two dimensional diffraction images showed no brilliant spots through the time-resolved observation of ten hours; MgO powder mixed with the specimen worked well to prevent grain growth of fcc FeDx. High quality of powder diffraction profiles were thus obtained and the refinement analysis is in progress.

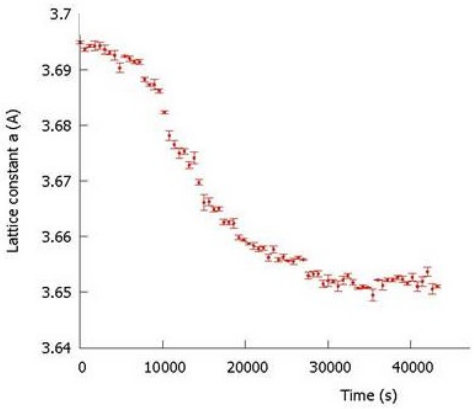

Fig.1 Lattice shrinkage due to SAV formation in FeD_x at 1000 K and 5 GPa.

We tried to prepare the ordered SAV state of fcc FeDx at high temperatures beyond 1000 K. No change was observed in diffraction profile; the ordered

structure was not developed.

According to the literatures previously reported, the ordered structure of SAV appears extremely rarely and the temperature and pressure conditions of the ordred SAV formation is unknown.

The results of the present experiment around 1000 K and 5 GPa indicates that higher temperature and pressure conditions are required to produce the ordered SAV state of fcc FeDx.

We made two experimental runs for the SAV formation of iron deuteride and faced trouble of the pressure control system every time. These system trouble cost enormous waste of time.