| CROSS TOKAI **Experimental Report** J-PARC | 承認日 Date of Approval 2017/03/27<br>承認者 Approver Takanori Hattori<br>提出日 Date of Report 2017/03/27 |
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| 課題番号 Project No.<br>2014B0017<br>実験課題名 Title of experiment<br>*In situ* neutron diffraction investigation on the formation process of superabundant vacancies in $FeD_x$<br>実験責任者名 Name of principal investigator<br>Katsutoshi Aoki<br>所属 Affiliation<br>The University of Tokyo | 装置責任者 Name of Instrument scientist<br>Takanori Hattori<br>装置名 Name of Instrument/（BL No.）<br>PLANET(BL11)<br>実施日 Date of Experiment<br>2-6, March, 2017 |

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.

A mixed powder sample of iron Fe and magnesium oxide MgO compacted to a disc shape 3.0 mm in diameter and 2.5 mm in thickness.

A bulk iron disc 3.0 mm in diameter and 2.5 mm in thickness.

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 sample of iron and magnesium oxide was pressurized in a hydrogenation cell containing inner hydrogen source of $AlH_3$ to 3.0 GPa at room temperature and then heated successively to 1050°C in order to synthesize hydride fcc-$FeH_x$ and further to form superabandant vacancy state, SAV, in it. Neutron diffraction measurements, however, revealed that fcc-$FeH_x$ remained unchanged with several hours holding at high temperatures far above an expected SAV formation temperature: SAV formation was not observed. Vacancy formation at the interface between iron and magnesium oxide appeared to be inhibited. We have observed a sequential structural transition for the bulk iron deuteride on cooling, fcc – [fcc+hcp] – [bcc+dhcp+hcp] – [bcc+dhcp]. This structural change was very contrast to the fcc – dhcp transition observed for the powder iron hydride in consistent with the phase diagram of the Fe-H system.

We will confirm the cause of SAV formation inhibition by synchrotron X-ray diffraction and examine another dispersed material instead of magnesium oxide. In a future experiment using PLANET at MLF, we will try to observe SAV formation process in fcc-$FeH_x$ by neutron diffraction.

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

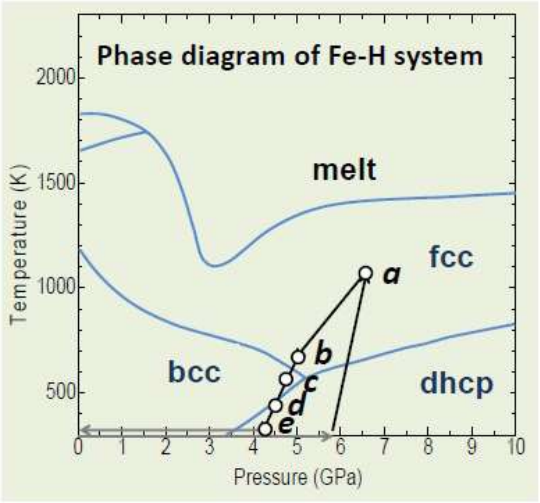


Fig. 1 A pressure–temperature path along which neutron diffraction profiles were taken for a bulk iron deuteride. a: 1073 K(800℃), 6.8 GPa, fcc-$FeD_x$, b: 673 K(400℃), 5.1 GPa, fcc and hcp-$FeD_x$, c: 573 K(300℃), 4.7 GPa, fcc and hcp-$FeD_x$, d: 473 K(200℃), 4.5 GPa, hcp, bcc and dhcp-$FeD_x$ e: 298 K(25℃), 4.2 GPa, bcc and dhcp- $FeD_x$

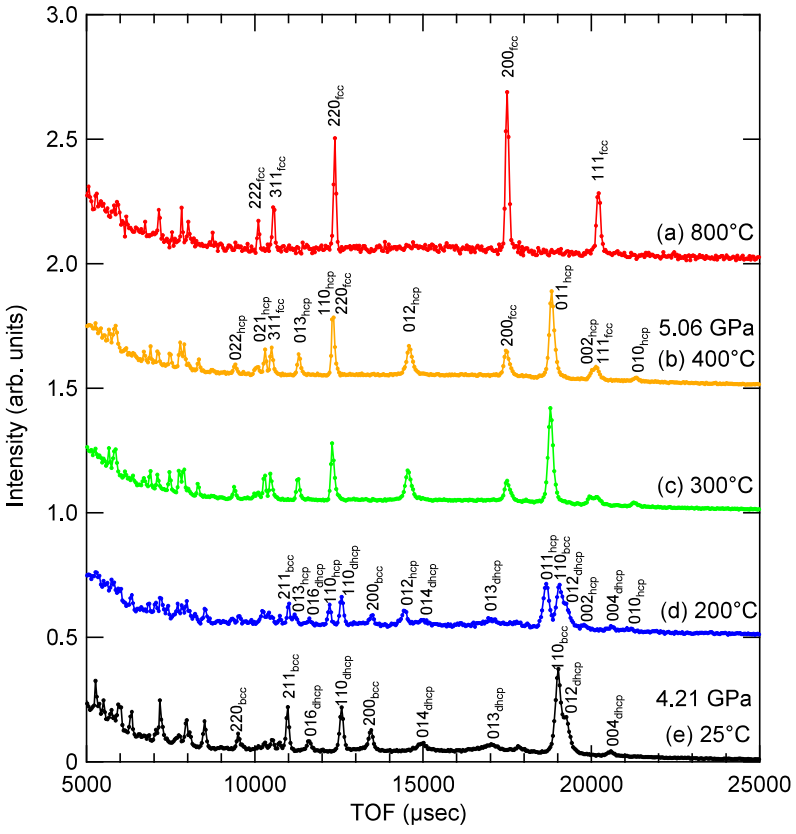


Fig. 2 Diffraction profiles of a bulk iron deuteride taken along the cooling path, showing a sequence of structural change, fcc – (fcc + hcp) – (bcc + dhcp).