CROSS TOKAI **Experimental Report** J-PARC

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課題番号 Project No.
2016A0021

実験課題名 Title of experiment
In situ neutron diffraction investigation on the formation process of superabundant vacancies in NiD_x

実験責任者名 Name of principal investigator
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装置責任者 Name of Instrument scientist
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装置名 Name of Instrument/(BL No.)
PLANET(BL11)

実施日 Date of Experiment
15-21, September, 2016
19-23, January, 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 nickel, Ni, and magnesium oxide, MgO, compacted to a disc shape 3.0 mm in diameter and 2.5 mm in thickness.

A bulk nickel 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 nickel and magnesium oxide was pressurized in a hydrogenation cell containing inner hydrogen source of AlD_3 to 3.0 GPa at room temperature and then heated successively to 800°C in order to synthesize hydride fcc-NiD_x and further to form superabandant vacancy state, SAV, in it. Neutron diffraction measurements, however, revealed that fcc-NiD_x remained unchanged with several hours holding at high temperatures at an expected SAV formation temperature: SAV formation was not observed. Vacancy formation at the interface between nickel and magnesium oxide appeared to be inhibited.

We have determined experimentally a hydrogen induced volume expansion for Ni metal, for the first time, by neutron diffraction measurements. In addition, a saturated maximum hydrogen concentration has been determined as well. These experimental data are essential in discussion of the miscibility gap in the hydrogen solubility of nickel metal in combination with pressure – volume – temperature data obtained by X-ray diffraction in early works.

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-NiD_x by neutron diffraction.

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

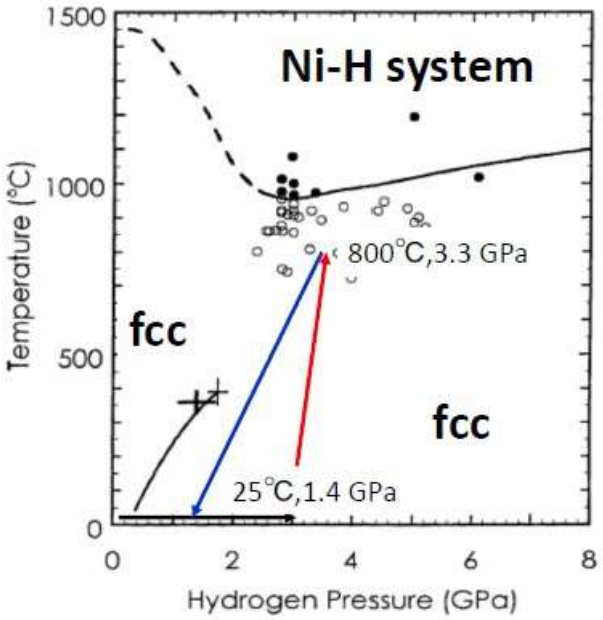

Fig.1 Heating and cooling paths for neutron diffraction measurements on a bulk nickel deuteride. Diffraction profiles were collected at a 150℃ interval along the cooling path.

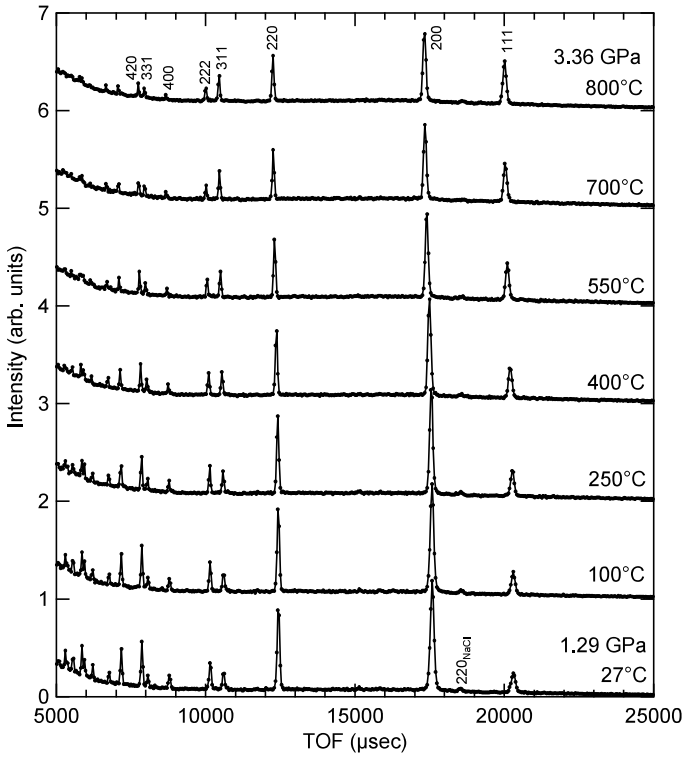

Fig. 2 Evolution of diffraction profile on cooling. $P-V-T-x$ relation will be derived for the Ni-D system by refinement analysis of the measured diffraction profiles, allowing future analysis of the miscibility gap and critical point in combination with the $P-V-T$ relation to be obtained by synchrotron X-ray diffraction.