



実験報告書様式(一般利用課題・成果公開利用)

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 Experimental Report 	承認日 Date of Approval 2014/9/1 承認者 Approver Takanori HATTORI 提出日 Date of Report 2014/9/1
課題番号 Project No. 2014A0110 実験課題名 Title of experiment Structural analysis of the condensed phase of lithium borohydride using high-pressure neutron diffraction 実験責任者名 Name of principal investigator Satoshi Nakano 所属 Affiliation National Institution for Materials Science	装置責任者 Name of Instrument scientist Takanori Hattori 装置名 Name of Instrument/(BL No.) PLANET (BL11) 実施日 Date of Experiment 2014.5.8-5.13

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.
(1) Lithium borodeuteride (LiBD_4), powder (2) Deuterated ammonia borane (ND_3BD_3), powder

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.
<p>Low-Z hydrides are attractive as novel hydrogen-storage materials for their high hydrogen density, and novel superionic conductors or superconductors. In the present work, we investigated the densification behavior of two kinds of high hydrogen-density low-Z hydride, lithium borodeuteride (LiBD_4) and deuterated ammonia borane (ND_3BD_3) using neutron diffraction under high-pressure for studies on novel materials.</p> <p>The samples are loaded into an encapsulating gasket of a Ti-Zr alloy as a sample capsule in an inert atmosphere of an Ar glove box. The capsule was clamped with toroid-type high-pressure anvils and set into Paris-Edinburgh (PE) high-pressure cell. Since the sample is small, a focusing mirror and hBN collimator of the incident beam and a radial collimator are used. Usual 90-degree neutron diffraction measurement is performed in the duration of 8 hours. Pressure value was estimated from lattice parameters of the sample which has been obtained using x-ray diffraction measurement.</p>

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

(1) Lithium borodeuteride (LiBD₄)

In the experiment for LiBD₄, a diffraction pattern obtained was quite weak. Absorption by boron in the sample could be considered as a cause. Therefore, the sample was diluted with fluorinert as a pressure medium and the similar measurement was carried out. The diffraction intensity was obtained from the diluted sample, but it was not enough for structural analysis. For a future experiment, we will carry out an isotopic substitution of boron in LiBD₄ and try the similar measurement.

(2) Deuterated ammonia borane (ND₃BD₃)

The sample ND₃BD₃ transforms to the first high-pressure phase (HP1) at 1.1 GPa and the second high-pressure phase (HP2) at about 10 GPa. We have performed a neutron diffraction experiment for the first transformation last year. In the present work, we examined the similar experiment for the second transformation. Diffraction data was obtained at 7.2, 9.8, and 14.1 GPa as shown in Figure 1. The diffraction intensity obtained was enough to analyze the structure. Also, measurements of vanadium and empty cell were carried out at the same conditions for an absorption correction of the diffraction profiles. Using the present data, the crystal structures of HP1 and HP2 will be analyzed in detail and a pressure dependence of their dehydration bond length will be calculated.

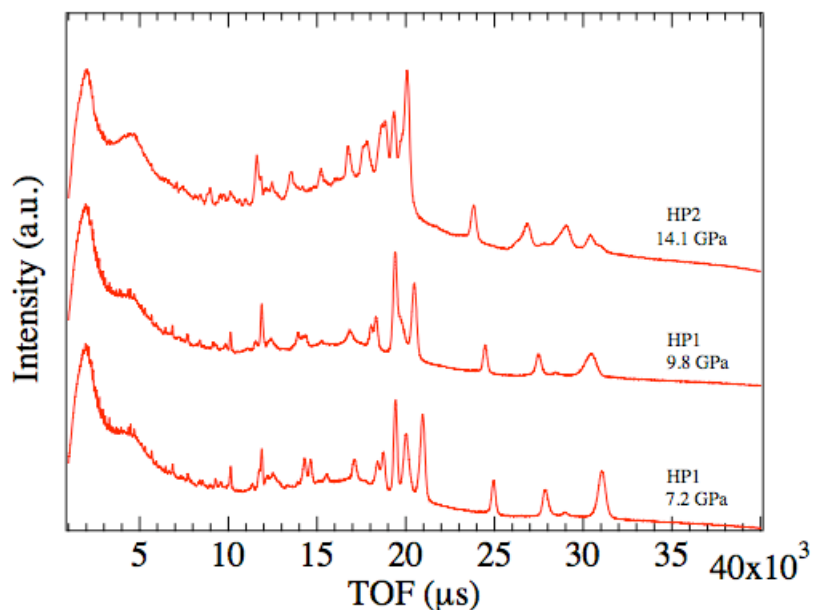


Figure 1. Neutron diffraction patterns of two kinds of high-pressure phase of ND₃BD₃ (before an absorption correction).