（ ※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 2016/09/13 承認者 Approver Takanoti Hattori 提出日 Date of Report 2016/06/21
課題番号 Project No. 2015A0180 実験課題名 Title of experiment High-pressure neutron diffraction study on high-pressure structures of ternary low-Z hydrides 実験責任者名 Name of principal investigator Satoshi Nakano 所属 Affiliation National Institute 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 2016.6.8–6.15

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 ($Li^{11}BD_4$), powder

(2) Sodium borodeuteride ($Na^{11}BD_4$), 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.

Low-Z hydrides are attractive as novel hydrogen-storage materials for their high hydrogen density, and novel superionic conductors or superconductors. It is important to clarify the accurate crystal structure including hydrogen positions to understand the physical properties and chemical reactions of the hydrides.

We have investigated the relationship between the structures and properties on low-Z hydrides using in-situ x-ray diffraction and Raman spectroscopy. In the present experiment, we tried to obtain structural data of high-pressure phases of high hydrogen-density low-Z hydrides, lithium borodeuteride ($LiBD_4$) and sodium borodeuteride ($NaBD_4$) using neutron diffraction under high-pressure.

The samples are loaded into a Ti-Zr alloy capsule (an encapsulating gasket) in an inert atmosphere with an Ar glove box. No pressure medium was used. The capsule was clamped with toroid-type high-pressure anvils (WC-Co alloy or sintered diamond) and set into Paris-Edinburgh (PE) high-pressure cell.

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

Since the sample volume is small, a focusing mirror and hBN collimator for the incident beam and a radial collimator for the scattered beam are used. Usual 90-degree neutron diffraction measurement is performed in the duration of 1-8 hours. Pressure value was estimated from lattice parameters of the sample, which has been obtained using x-ray diffraction measurement.

(1) Lithium borodeuteride ($Li^{11}BD_4$)

In a previous experiment for $LiBD_4$, no satisfactory result was obtained because of absorption by ^{10}B boron in the sample. Therefore, we prepared a sample whose ^{10}B boron has been substituted to ^{11}B boron for the present experiment. $LiBD_4$ was transformed to the first high-pressure phase (phase-III) at 1.1 GPa. Namely we obtained better diffraction patterns of the phase-III at 1.7 and 3.0 GPa as shown Fig.1. Using the present data, the crystal structures of the phase-III, especially the positions of BD_4^- ions and hydrogen atoms, will be analyzed in detail.

(2) Sodium borodeuteride ($Na^{11}BD_4$)

The first high-pressure phase of $NaBD_4$ (β-$NaBD_4$) appeared at 5.3 GPa and the transformation was completed at 6.9 GPa. Next transformation from the β-phase to the second high-pressure phase (γ-$NaBD_4$) occurred at 8-11 GPa as shown in Fig. 2. Typical diffraction patterns of the phases was obtained for the duration time of 8 hours. Using the collected data, the crystal structures of β-$NaBD_4$ and γ-$NaBD_4$ will be analyzed in detail.

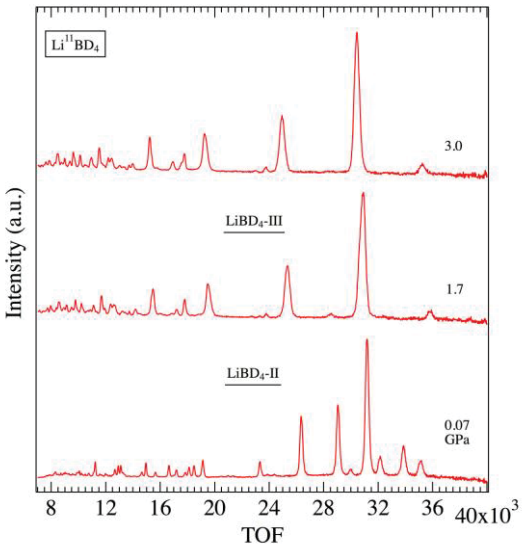

Fig.1. Pressure dependence of neutron diffraction

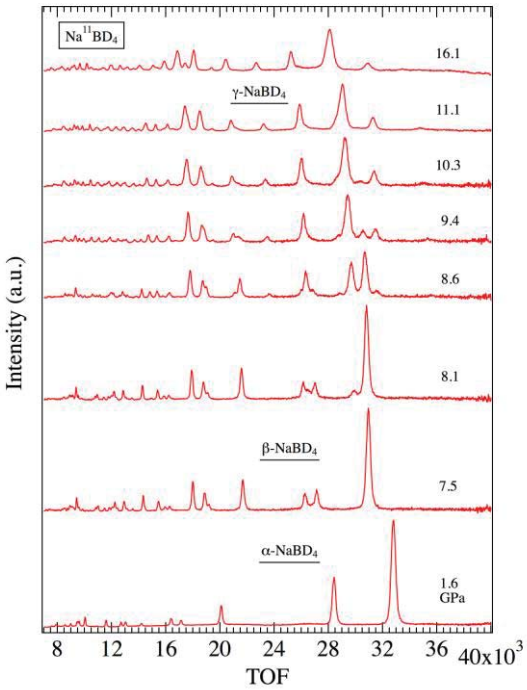

Fig.2. Pressure dependence of neutron diffraction patterns of $NaBD_4$.