（ ※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 2017/2/28 承認者 Approver Takanori Hattori 提出日 Date of Report 2017/2/28
課題番号 Project No. 2016B0179 実験課題名 Title of experiment High-pressure neutron diffraction study on ammonia borane ND_3BD_3 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 2017/01/31-2017/02/4

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) 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.

The purpose of the present study is to determine the hydrogen positions in some HP phases of ammonia borane, ND_3BD_3, and to observe the behavior of dihydrogen bonds in their densification.

The sample (Sigma-Aldrich, 97%) were crashed with a silica-glass agate and loaded into an encapsulating gasket of a Ti-Zr alloy as a sample capsule in an Ar atmosphere of a glove box. The capsule was clamped with a couple of sintered diamond double-toroid anvils of 4mm in diameters, and set into Paris-Edinburgh (PE) high-pressure cell. The sample was compressed to 18.3 GPa and usual 90-degree neutron diffraction measurement was performed in the duration of 10 hours and 40 min. A focusing mirror and hBN collimator of the incident beam and a radial collimator are used for the diffraction measurement. After the neutron diffraction measurement, the pressure value of the sample was estimated from the relationship between the volume of the sample and pressure, which has been obtained using an x-ray diffraction measurement in advance.

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

The sample ND_3BD_3 has already transformed to the first high-pressure phase (HP1) of $Cmc2_1$ structure at 1.7 GPa. The 111 and 112 peaks of HP1 gradually began to split above 9.7 GPa as shown in figure 1. It suggests that the next pressure-induced transformation from HP1 to the second high-pressure phase (HP2) of $P2_1$ (Z=2) structure took place.

Regarding the HP2, some main peaks of the phase (100, 011, 101) have shoulders or a broad base line, as shown in the figure. It might suggest that the crystal structure model ($P2_1$, Z=2) should be reconsidered.

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.

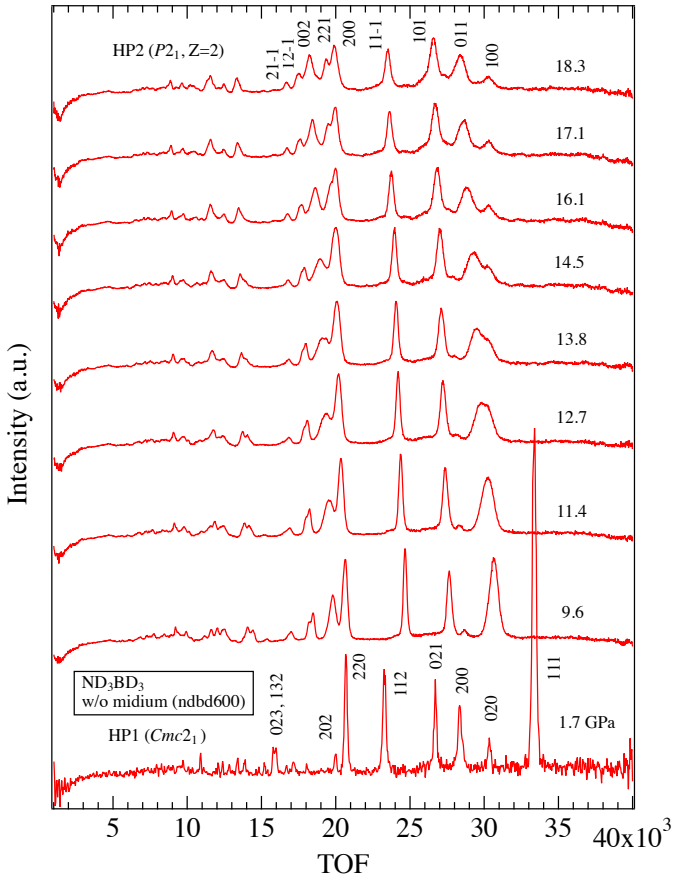

Fig.1. Neutron diffraction patterns of ND_3BD_3 at various pressures.