（ ※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。 ）

MLF Experimental Report	提出日 Date of Report 2011/07/04
課題番号 Project No. 2010B0075 実験課題名 Title of experiment: Bonding and disordering transformations of layered magnesium hydroxide at pressure 実験責任者名 Name of principal investigator Takuo Okuchi 所属 Affiliation Okayama University	装置責任者 Name of responsible person Stefanus Harjo 装置名 Name of Instrument/(BL No.) BL19 TAKUMI 実施日 Date of Experiment 2010/12/16 - 2010/12/17

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.

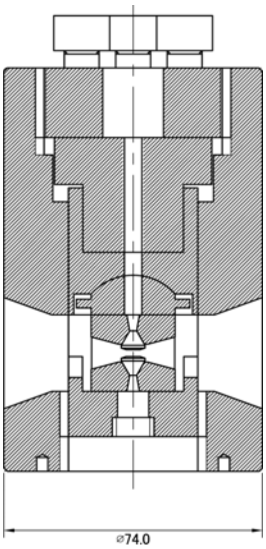
1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

Deuterated magnesium hydroxide (brucite): $Mg(OD)_2$

Deuterated glyceline: $C_3D_5(OD)_3$

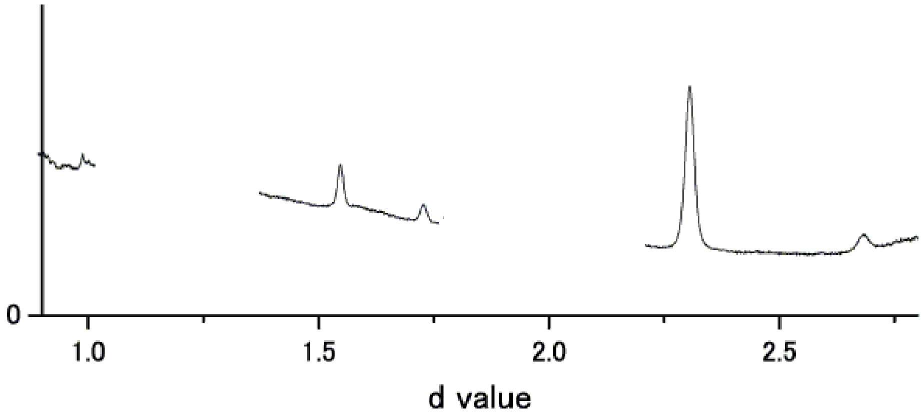
2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

The $Mg(OD)_2$ powder sample was synthesized at 250ºC and 4 MPa by reacting MgO powder and D_2O in an autoclave for a week. No significant OH residual peak was observed in its IR spectra. Our newly-developed opposed-anvil high-pressure cell is shown in the figure. $Mg(OD)_2$ powder of ~4 mm^3 volume (9 mg) was enclosed into the cell along with deutereated glyceline, which works as liquid pressure medium to alleviate uniaxial stress applied to the sample. It was slowly compressed by fastening the six screws, and then annealed at 80 ^oC for 1h. This high pressure cell was designed to generate 10 GPa for 3 mm^3 volume and 20~30 GPa for 1mm^3 volume.



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

At this time, we have successfully generated up to 8 GPa pressures and took neutron diffraction patterns of moderate quality. An observed pattern at 5 GPa is shown below (d-value shown in angstrom). Spurious peaks mainly coming from two anvils directly contacting with the sample; these are made of NPD (nano-polycrystalline diamond) and SD (sintered diamond), giving strong diffraction pekas of powdered diamond. These peaks have different d-values from the sample which were removed from the figure. All five peaks in the figure, including the most strong one at 2.3 A for 011 diffraction, were coming from the $Mg(OD)_2$. These were strong enough to determine the lattice constant variations at high pressure by Le Bail fit, although were still marginal to conduct a Rietveld analysis to determine the deuteron positions.



We have installed a microscope-based ruby fluorescence system at TAKUMI. Using this system, sample pressures were swiftly measured by optical ruby fluorescence technique through the transparent NPD anvil (Note: this system has now moved to the cabin close to BL11). We observed that the intensities of spurious diffraction were significantly reduced by the following procedures: (i) Shielding of neutrons using the combination of cadmium foil, hBN (boron nitride) tube collimator, boron rubber plate, and sinterd ^{6}LiF plate collimator. We have prepared a number of hBN collimators with different inner diameters to best-fit the sample size. We have also prepared sintered ^{6}LiF collimators with different inner diameters for use at the beam entrance to reduce the gamma-ray background to scintillation detectors. (ii) Fine tuning of neutron focusing device designed for TAKUMI. It works not only to increase the beam intensity at the sample position but also to reduce the background.

We have prepared for our second beamtime scheduled on March 14 to 15 to conduct the experiments at higher pressures than 10 GPa, for a reduced sample volume at ~1 mm^3. We were also ready to increase the pressure on-site to obtain the results at multiple pressures, all of which would be the first results of neutron diffraction at >10 GPa for this sample. However, the earthquake had made it impossible. We are expecting that after the recovelry of TAKUMI, we will continue the proposed experiments to complete the scientific mission described.