CROSS TOKAI

MLF Experimental eport

J-PARC

承認日 Date of Approval 2015/5/7
承認者 Approver Takanori Hattori
提出日 Date of report 2015/5/7

実験装置名／BL番号 Name of Instrument/BL
PLANET/BL11
実験装置責任者 Name of the person responsible for the instrument:
Takanori Hattori
所属 Affiliation: Japan Atomic Energy Agency

1. 研究成果概要 (a)装置グループ内の成果、(b)ユーザー課題実装時における特筆すべきサポート、(c)ユーザー課題の執行状況について、まとめてください。A4 サイズ用紙使用のこと。

Outline of your activities. Following results at your instrument should be reported in A4 size papers: (a) results of your instrument group, (b) significant user support works, and (c) statistical summary of user experiments.

(a) Results of your instrument group

This year, we aimed at developing following items.

- Stable high-PT generation by the 6-6 compression system with 6-axis press
- Extention of accessible PT range by employing the 6-8 compression system
- Neutron diffraction at ultra-high pressure condition using a diamond anvil cell
- Simultaneous measurements of the neutron diffraction and acoustic emission

Some of them have been accomplished, and others remained untouched because of the unscheduled beam shutdown by fire accidents etc. Here, we introduce several results that have been conducted.

Development of the stable high-PT generation by 6-6 compression system with 6-axis press

The extension of attainable high-PT condition with keeping the sample size is basically difficult. Therefore, the sample size usually needs to be reduced when one wants to access a higher PT condition. So far, the attainable pressure and temperature condition is limited to 8 GPa at 1000 K and 10 GPa at RT when using standard design tungsten carbide anvils with truncation edge length (TEL) of 7 mm in the 6-axis press experiments. However, the "blow-out" often happened at such condition. To successfully conduct high-PT experiments, we tested feasibility of the anvils reinforced by a surrounding support ring (Fig. 1) and high-pressure cell which pregaskets are attached on. This is originally developed by Yagi's group in Tokyo University. Off-line tests confirmed the safe high-PT generation of 9.8 GPa and 1200 C^{o} without blowouts. In-situ neutron diffraction has revealed that the scattering intensity is reduced by the factor of 70 % due to the reduction of the horizontal opening angle. This study have shown that this type of anvils can be used to explore the higher PT state for the sample with

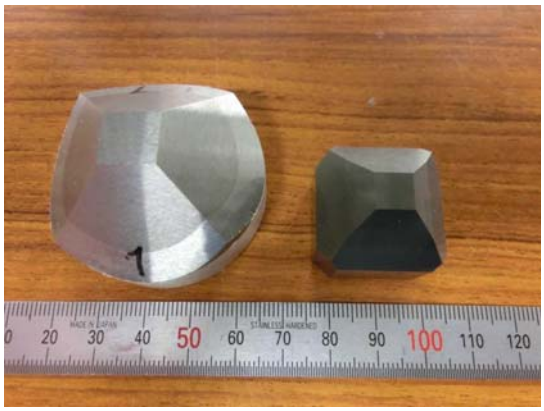

Fig.1 newly designed anvils reignforced by a surrounding support ring (left) and the original one (right)

intense signals. Since the exposure time for a typical sample is unrealistic (~24h per each pressure point), furthermore developments are needed.

Extention of accessible PT range by employing 6-8 compression system

In conventional 6-6 compression method, the accessible PT region is limits to about 10 GPa and 2000 K. To extend the region, we applied the 6-8 compression method to *in-situ* neutron diffraction experiments. In this method, the high-pressure cell of octahedron is compressed by eight anvils (Fig. 2). The anvils are cubes made of SiC sintered diamond with truncated edge length (TEL) of 5 mm. The initial volume of the sample was 6 mm^3. The test using internal pressure marker (NaCl) confirmed the pressure generation of 16 GPa at 1600 kN (Fig. 3). The heating test also showed the temperature generation of 1273K. But, on heating to 1073K, the pressure was decreased to 14 GPa due to the deformation of the gaskets that seals pressure.

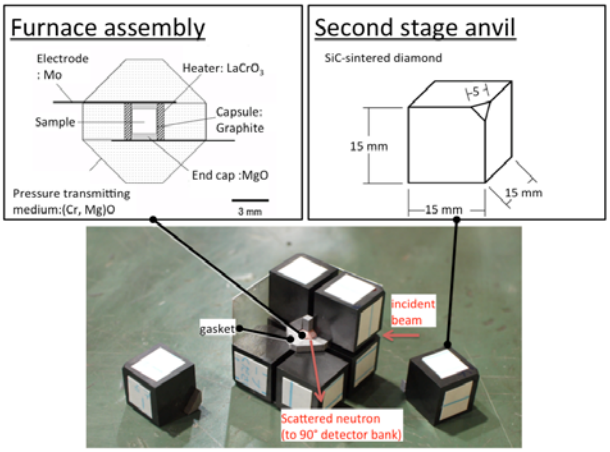

Fig. 2 Photograph of a 6-8 type cell and schematic drawings of the furnace assembly and the second stage anvil

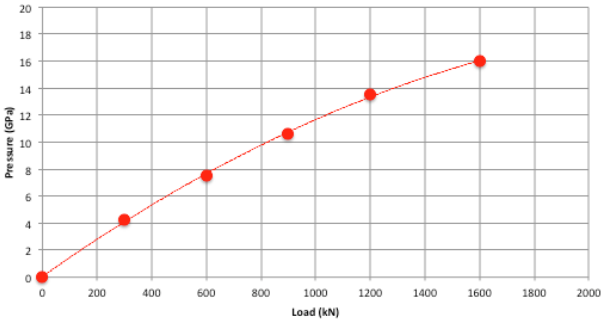

Fig. 3 Pressure generation curve for a 6-8 cell with TEL of 5 mm. The horizontal axis is the load applied to the

Figure 4 shows a typical example of the neutron diffraction pattern. The $CaSiO_3$ perovskite was synthesized at high-pressure and high-temperature from ambient phase (wollastonite). The diffraction pattern is taken at 15.4 GPa for 36 hours after cooling to room temperature. The pattern shows several new peaks that have not been reported in the previous x-ray diffraction, maybe due to the different scattering length. This ensures that the structural investigation above 10 GPa is possible by employing 6-8 compression system.

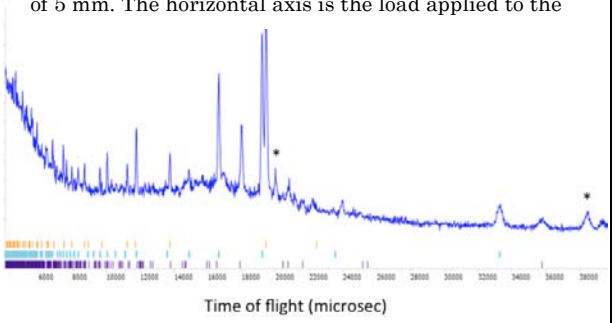

Fig. 4. Diffraction pattern of CaSiO3 perovskite obtained at 15.4 GPa with a 6-8 cell. The tick marks show the calculated peak positions for the cubic $CaSiO_3$ (cyan), MgO (orange) and $LaCrO_3$ (purple). The stars indicate the peaks that can be indexed with the orthorhombic cell

(b) Significant user support works

For the user experiments, we did the following developments.

・Feasibility test of aluminum cube as a high-pressure cell. It can seal liquid pressure transmitting medium, which is effective for sample easily deteriorated by shear stress during compression.

・Feasibility test of liquid container made of Teflon. This is also used for the same purpose as mentioned above.

1. 研究成果概要（つづき） Outline of experimental results (continued).

(c) Statistical summary of user experiments.

・Some user experiments are transferred to 2015A due to the unscheduled beam shutdown by fire accident.

・Other experiments have been conducted as scheduled.

・The statistics of the science fields of users from 2013 to 2014 is shown in Fig. 5

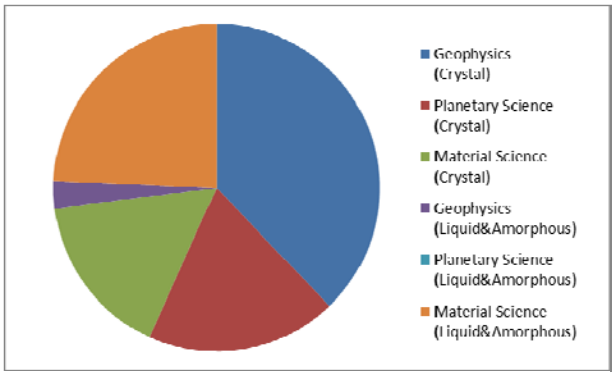

Fig. 5. Statistics of the users from various science fields from 2013 to 2014.

必要に応じて、A4 サイズの用紙に続きを記入して下さい。

Please use A4-size papers for further reporting, if necessary.