MLF Experimental Report	提出日 Date of Report 2010/5/17
課題番号 Project No. 2008A0027 実験課題名 Title of experiment Development of high pressure devices for neutron powder diffraction study 実験責任者名 Name of principal investigator 内海 渉 所属 Affiliation 日本原子力研究開発機構 量子ビーム応用研究部門	装置責任者 Name of responsible person 相澤一也 装置名 Name of Instrument/(BL No.) BL19 実施日 Date of Experiment 1 月 24 日～1 月 27 日 1 月 30 日～1 月 31 日 2 月 13 日～2 月 14 日 2 月 17 日～2 月 19 日

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.

A Paris-Edinburgh (PE) press as a high pressure device

Pb (powder sample: compacted into tablet shape (6mm in diameter, 4mm in height)

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.

<Experiment>

A VX4 Paris-Edinburgh Press (maximum load: 230 ton) was placed on the translation stage of TAKUMI. A tungsten carbide (WC) alloy with Ni binder was used as the anvil material and Duralmin or null-scattering TiZr alloy were used as a gasket. A tablet shape specimen (Φ6 mm x 4 mm) made by pre-compressing Pb powder was sandwiched by the opposing anvils together with the gasket. The incident beam slit (aperture size: 15 x 11 mm) was placed at 230 mm from the sample. Neutron experiments were performed with no load applied.

Two geometries were used in the neutron diffraction with the PE press (Figure 1(a)(b)): a) Vertical scattering geometry; the standard generally used in pulsed neutron experiments; The incident neutron beam was parallel to the compression axis, and should therefore have passed through the anvil before irradiating the sample. b) Horizontal scattering geometry; Incident neutrons were introduced perpendicular to the compression axis, and the neutron passed through the gasket material. The power of the proton accelerator used during the experiments was ca. 20 kW. Exposure times of 6-13 hours for the vertical scattering geometry and 9 hours for the horizontal scattering geometry were used.

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

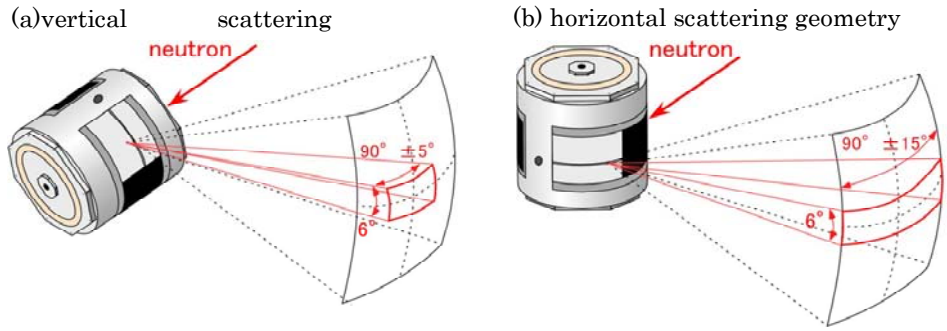

Fig..1 Schematic illustration of the experimental geometry of the PE press.

<Results>

Figure 2 plots the diffraction profiles obtained using the vertical scattering geometry where three set of experimental data, a) without any collimators, b) with Cd shielding and c) with a radial collimator, are compared. Diffraction peaks of Pb (200, 220, and 311) can be clearly observed together with several peaks originating from the anvil at all experiments. The three Pb peaks were fitted to a Gaussian function and their d-values calculated to be 2.48 Å, 1.75 Å, and 1.49 Å, with a resolution ($\Delta d/d$) of ca. 0.33 %. In the experiments where any collimators were not used i.e. (a), the signal intensity of the 311 peak was 10.1 count/hour and the background intensity 5.5 count/hour. When the Cd shielding was used i.e. (b), the signal was kept at basically the same intensity (11.0 count/hour), but the background considerably dropped to 0.8 count/hour. Background noise caused by scattering from high-pressure device is a serious problem that needs solving. The use of Cd shielding resulted in the detectors only being capable of viewing the sample position, and therefore the scattering neutrons from a high-pressure device and air were greatly discriminated.

Figure 3 plots the diffraction profile obtained using the horizontal scattering geometry. Neither Cd collimators nor radial collimators were used in this experiment. Clear Pb diffraction peaks in the PE press were also successfully observed using the horizontal geometry. The intensities of the signal and the background were 175.6 count/hour and 49.7 count/hour, respectively. The signal intensity was larger than that with the vertical scattering geometry, which can be ascribed to the difference in the effective solid angle of detectors and absorption of neutrons by the anvil.

The present study confirmed that in situ high-pressure neutron powder diffraction using a PE press was feasible with the Engineering Materials Diffractometer “TAKUMI” at J-PARC.

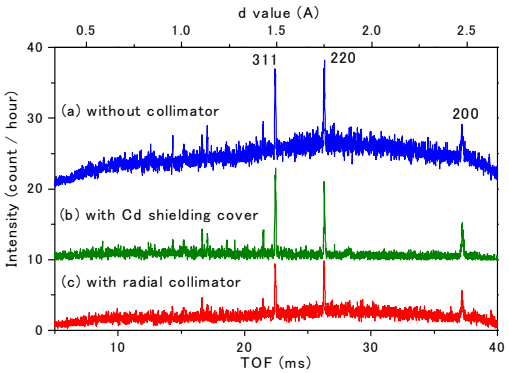

Fig.2 Pb profiles embedded in PE press for vertical scattering geometory

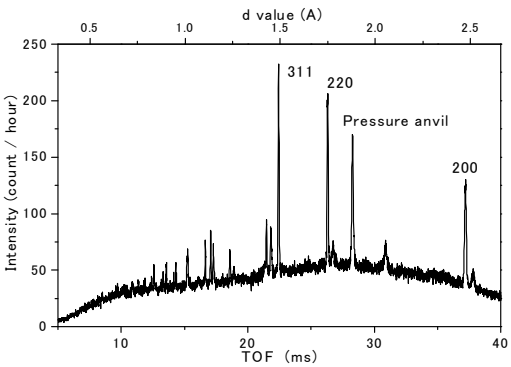

Fig.3 Pb profile for horizontal scattering geometry