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 MLF Experimental Report	提出日 Date of Report 2011/09/06
課題番号 Project No. 2009A0082 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 Takanori Hattori 所属 Affiliation JAEA Quantum Beam Science Directorate	装置責任者 Name of responsible person Kazuya Aizawa 装置名 Name of Instrument/(BL No.) Engineering Materials Diffractometer (BL19) 実施日 Date of Experiment 2009-2010

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.
Lead, Pb, pellet. Ice, H ₂ O, solid 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.
<p>To check matching of several high-pressure devices with neutron diffraction and to develop the high-pressure devices based on the results.</p> <p>We are planning to use three kinds of high-pressure devices. We brought all these to the TAKUMI and performed neutron diffraction. The following results are obtained.</p> <p><u>P-E press</u></p> <p>The neutron diffraction was performed for a standard sample of Lead. We confirmed the detection angle of plus/minus 5 deg. in horizontal plane under the restriction of the opening angle of the press. We also found the incident beam loss of about 50% due to the neutron absorption by an anvil material. These are consistent with previous results at ISIS. In spite of these difficulties, we could manage to obtain the data of lead at 5 GPa.</p> <p><u>Palm cubic anvil cell</u></p> <p>This type of the cell was used for TOF experiments for the first time, and the data of lead was taken at 1.5 GPa. Despite the worry about its limited opening angle, we obtained data comparable to those taken by P-E press. Furthermore, the cell was attached to the cryostat and the data at low temperature and high-pressure</p>

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

(60K and 1 GPa) were successfully collected.

NPD cell

The NPD cell is still under development. For the test, we took the diffraction data of lead using this cell. In spite of the small size of the sample (1mm × 1mm), we could obtain the signal from the sample successfully. Besides the signal, huge parasitic scattering from the NPD anvils was observed, which showed the importance of the further development to reduce the background.

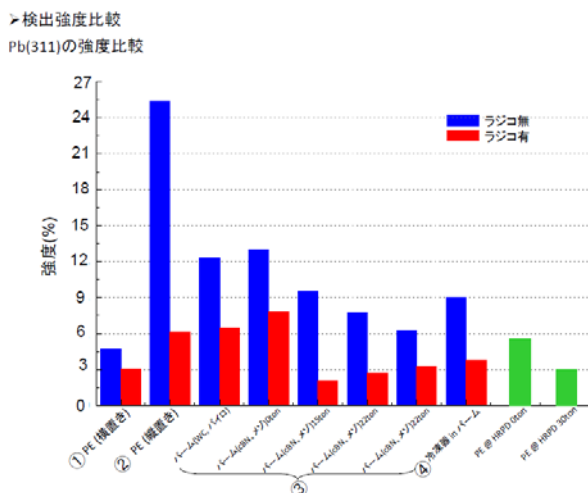


Fig. 1 Comparison of the intensity loss for Pb311 peak among several high-pressure devices with various scattering geometries. The horizontal axis represents the intensity decrease against the intensity for the naked sample without press.

Development of experimental setup to increase the signal intensity and to reduce background.

In the high-pressure experiments the sample is tiny, so the incidence beam is preferable to be intense. To gain the incident beam flux, focusing mirror was designed and installed in the TAKUMI. The results showed the gain of the designed value and remarkable reduction of the background due to the focusing of the incident beam. Furthermore, the origin of the background was inspected and the shielding was added to the experimental setup in order to reduce background. By these efforts, the signal to background ratio for high-pressure data increased from two to ten (Fig.2).

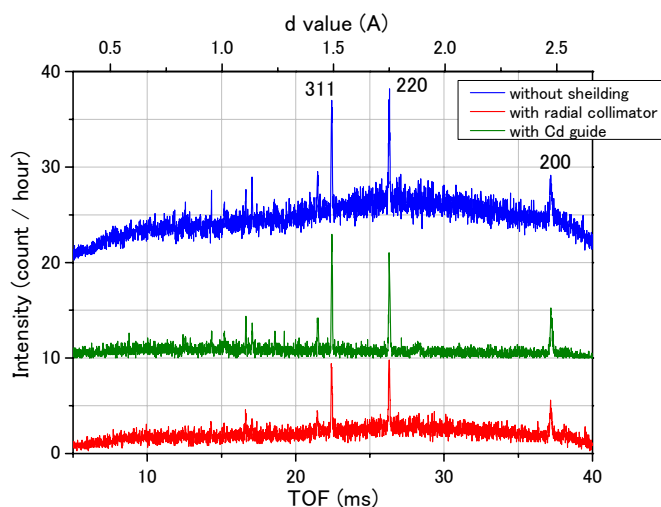


Fig. 2 Gain of S/B ratio by shielding the detector bank with Cadmium plates. The S/B ratio increased from 2 to 10.