実験報告書様式（プロジェクト利用）

Experimental Report	承認日 Date of Approval 2013/06/27 承認者 Approver TAKEDA Masayasu 提出日 Date of Report 2013/06/25
実験課題番号 Project No.: 2012P0903 実験課題名 Title of experiment Project research on structure and dynamics of proton, superionic and amorphous functional materials – Investigation on silver photo-diffusion into amorphous chalcogenide films by means of neutron reflectometer 実験責任者名 Name of principal investigator Yukinobu Kawakita 所属 Affiliation: JAEA	装置責任者 Name of Instrument scientist Masayasu Takeda 装置名 Name of Instrument/（BL No.） SHARAKU (BL17) 利用期間 Dates of experiments 2013/02/27-2013/03/09

１．研究成果概要（試料の名称、組成、物理的・化学的性状を明記するとともに、実験方法、利用の結果得られた主なデータ、考察、結論、図表等を記述してください。

Outline of experimental results (experimental method and results should be reported including sample information such as composition, physical and/or chemical characteristics.

Experimental method

In-plane structure is of technical as well as fundamental interest in the field of thin films and it can be clarified by grazing incidence diffraction (GID) technique. So far, GID with X-ray beam is now a conventional tool while GID with neutron beam is still challenging due to the intensity of the probe beam. In J-PARC, proton beam power increases to be 300kW, which improves the neutron beam flux condition, and GID option has been installed on polarized neutron reflectometer beam line, SHARAKU (BL17). These situations encourage us to perform GID experiment in J-PARC. Of course, in superionic and amorphous functional films research, GID measurement using neutron beam is very attractive and could be pioneering work.

In this project, we have performed fundamental GID measurements to see the performance of the instrument first. After the measurements, we have measured GID for silver photo-diffusion in Ag/$Ge_{40}S_{60}$ films under light exposure. Since brilliant X-ray beam of synchrotron radiation can induce silver diffusion in the system, use of neutron beam is preferable. In the measurements, 5-axes goniometer was used for accurate alignment of samples, incident beam and a detector, and RPMT 2-dimensinal detector was used as shown in Fig.1.

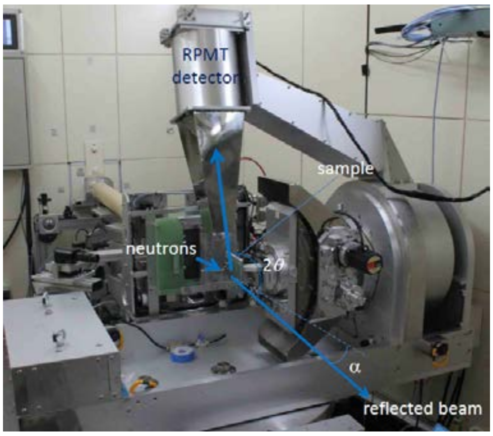


Fig.1

Results

1. Diffraction measurements for bulk samples

In order to check the performance of the diffraction measurement system, diffraction measurements have been performed for several standard samples. Fig.2 shows the 2-dimensional neutron intensity map (left) and the diffraction curve plotted on Q-I plane (right) for a bulky nickel. From the result, we could confirm that the

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

instrument works properly as a diffractometer.

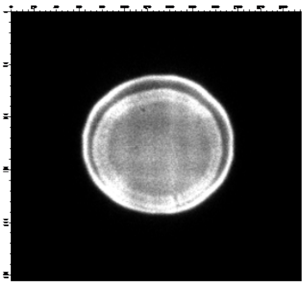
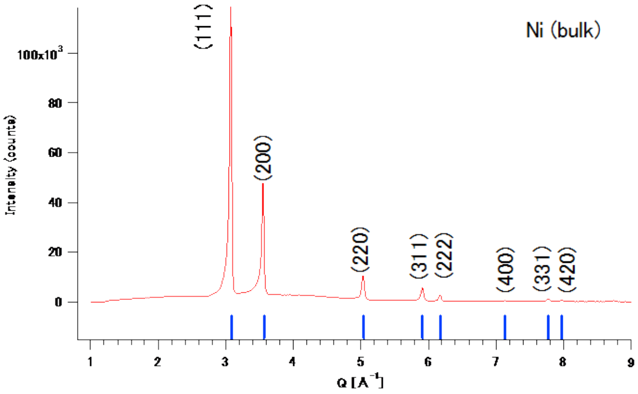
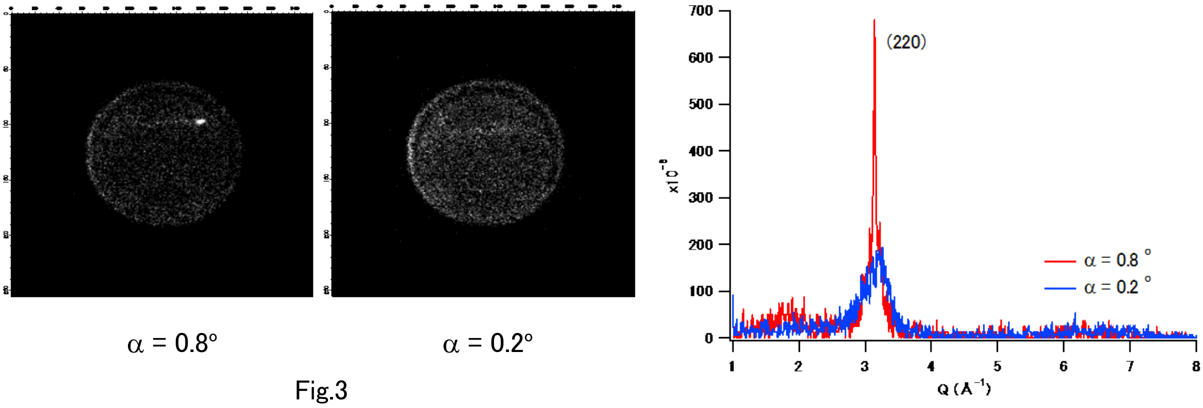


Fig. 2



2. Grazing incidence diffraction measurements

Next, we have measured grazing incidence neutron diffraction for a silicon wafer, a nickel film, and a silica glass plate. Fig.3 shows the 2-dimensional neutron intensity maps (left) and the diffraction curve plotted on Q-I plane (right) for a silicon wafer. Only broad peak is observed for grazing incidence beam, α = 0.2°, close to a critical angle for total reflection, while both a sharp Bragg peak and a broad peak are observed for incidence beam with larger angle, α = 0.8°. Obviously, the sharp Bragg peak comes from the single crystalline silicon wafer. The broad peak seems to indicate a diffused scattering from natural oxide layer on top.



α = 0.8°

α = 0.2°

Fig.3

3. GID measurements for silver photo-diffusion in $Ag/Ge_{40}S_{60}$ films under light exposure

Finally, we have measured GID for silver photo-diffusion in Ag 50nm/$Ge_{40}S_{60}$ 150nm film under light exposure. In the measurement, we used a xenon lamp unit (MAX-303, ASAHI SPECTRA) as an excitation light source. The illumination area on the film was 30mm x 30mm. The light was exposed from $Ge_{40}S_{60}$ layer side. Fig.4 shows the integrated time-of-flight (TOF) spectra for the samples before light-on and under the light exposure. As shown in the figure, a change has been observed by the light exposure. However, the change is not related to the diffraction peak from amorphous layer. It would probably be related to Bragg peaks from silver crystal. We are now making further analysis to identify the origin of the change.

Through the measurements, we have realized the characteristics of the time-of-flight instrument in GID measurement. We wish that our experience is utilized in the following GID experiment on the beam line.

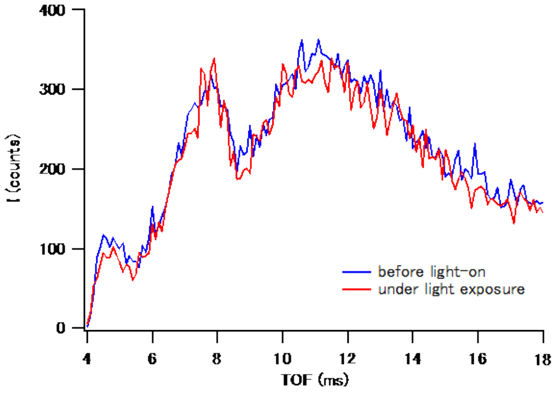


Fig.4