実験報告書様式（一般利用課題・成果公開利用）

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CROSS TOKAI **Experimental Report** J-PARC	承認日 Date of Approval **2015/03/23** 承認者 Approver **Masayasu Takeda** 提出日 Date of Report **2014/12/08**
課題番号 Project No. **2014A0141** 実験課題名 Title of experiment In-situ neutron reflectivity measurements for photodoping of silver in Ge-chalcogenide films IV 実験責任者名 Name of principal investigator Yoshifumi Sakaguchi 所属 Affiliation CROSS	装置責任者 Name of Instrument scientist Masayasu Takeda 装置名 Name of Instrument/(BL No.) *SHARAKU* (BL17) 実施日 Date of Experiment April 20 10:00 – April 22 10:00, 2014 April 26 10:00 – April 28 10:00, 2014 April 29 10:00 – May 1 7:00, 2014

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.

Ag/Ge_xS_{1-x} films (x=20, 33, 40)

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.

Experimental method:

In order to clarify the change on the depth profile in Ag/amorphous (a-) Ge chalcogenide films by silver photo-doping, (silver diffusion) we investigate on the neutron reflectivity of Ag/a-Ge-S films under a light illumination. In the previous experiments, we measured time-resolved neutron reflectivity profiles of Ag/a-$Ge_{40}S_{60}$ films (2012A), Ag/a-$Ge_{20}S_{80}$ films (2013A), and Ag/a-$Ge_{33}S_{87}$ films (2013B). The neutron reflectivity data were analyzed by Fourier transformation technique and a reliable result on the silver diffusion kinetics was obtained without assuming any models. However, due to the limitation of Q-region in the time-resolved reflectivity profiles, the peak around 100 Å was not clear. The peak indicates the thickness of silver layer, and it clearly appears in the Fourier transform of the full set of the reflectivity profile which was obtained from two reflectivity measurements at two different angles. To improve the Fourier transform of the time-resolved reflectivity profile, which is obtained from the reflectivity measurement fixing at one angle, we try to obtain reflectivity profiles by changing the time window of incoming neutrons. This is realized by changing the phases of rotating three choppers. On BL17, the shortest neutron wavelength is usually set to be 2.2 Å. In the present measurement, the shortest neutron wavelength is set to be 1.0 Å. Applying this technique, the Q-region in the reflectivity profile fixing at one angle is considered to be wider, and this is the purpose of this experiment.

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

Results:

First of all, we have faced the problem that the neutron reflectivity profiles were not properly obtained as shown in Fig.1and 2. The profiles in the figures apparently show that the Q-resolution is different according to the angle. In all the measurements, the slit conditions were set to be $\Delta\theta/\theta$ = 0.05. Indeed, the wavelength resolution depends on the wavelength in the data set. However, in the data reduction (bin width: 100), the total Q-resolution at 8.8 Å is calculated to be 0.05 while that at 1.1 Å is calculated to be 0.06, and the profiles with different angles should basically be overlapped. Therefore, there was a doubt that the slits did not work properly.

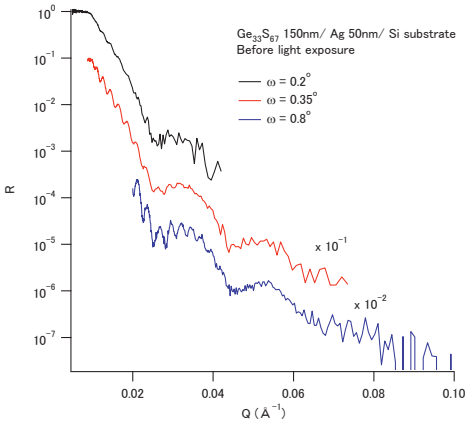

Fig.1

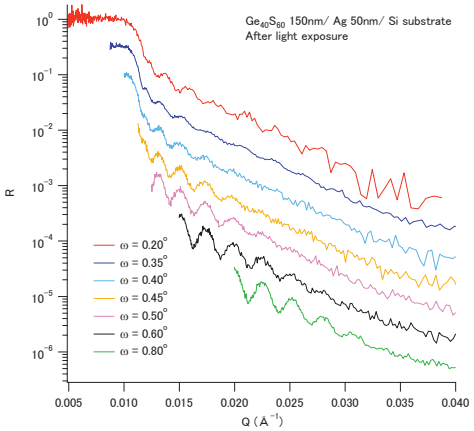

Fig.2

To check the actual slit condition, we set the slit width to be 0. The result was Fig.3. Surprisingly, there were considerable amount of neutrons even if the slit width is 0. This means that there is a discrepancy between the actual slit position and the reading value. Assuming the opening offset, it is easy to understand the difference of Q-resolution depending on the angles in Fig. 1 and 2.

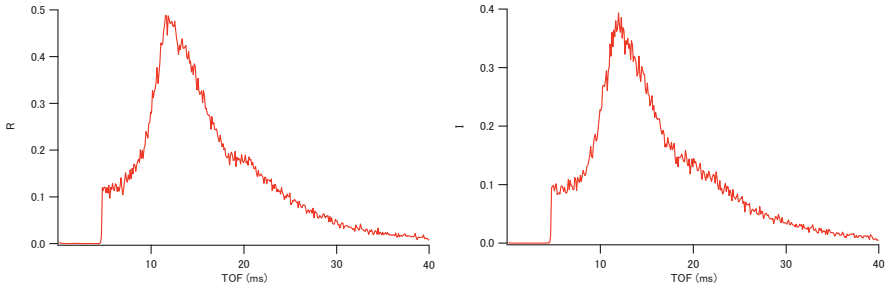

Fig.3 (a) width～40μm (b) width = 0 μm.

Actually, it seemed that the slit condition was not stable; some reflectivity profiles looked better (“better” does not mean “good”), and some were very bad without proper fringes. We tried to get meaningful results again and again from the better data, because that was the only thing which we could do after the measurements. However, the roughness of the film was not good for the data. As a result, the intensity at higher Q-region was very weak and the time-slicing data were very poor, which cannot provide useful information on silver diffusion kinetics.

In conclusion, we tried to refine the time-resolved reflectivity data by extending the Q-region, but failed due to the experimental conditions mentioned above.