Kyushu Synchrotron Light Research Center

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Experiment Report for Prefectural Beamline

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Local structures in Ga-Ge-Se glasses used for high-speed telecommunications

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1．**Summary**（Note: Please include conclusions）

We conducted a first experiment of anomalous x-ray scattering (AXS) at BL15. The aim of our research project is the investigation of Er-doped $(GeSe_2)_{0.8}(Ga_2Se_3)_{0.2}$ chalcogenide glasses. For this first experiment, we limited the sample choice to the undoped $(GeSe_2)_{0.8}(Ga_2Se_3)_{0.2}$ to establish the method and to obtain reference data for future AXS experiments. The final information gathered in this project are expected to elucidate the role of Er and Ga in the amorphous structure and their impact on the photoluminescence properties. Since AXS is sensitive to the short- and intermediate-range order, we expect to obtain information on the possible formation of Er clusters, which are expected for higher concentrations of Er, and about the role of the Ga atoms, which are a necessary requirement to solve Er in the glass matrix [1, 2].

2．**Purpose of experiment and background**

Chalcogenide glasses are characterized by remarkable physical properties, such as high ionic conductivity or large photosensitivity. In general, their properties can be fine-tuned by varying the composition of the constituent elements. Specifically, the photoluminescence properties in rare earth-doped Ga-Ge-Se glasses can be largely varied by the concentration of Ga and the dopant. Er doped $(GeSe_2)_{0.8}(Ga_2Se_3)_{0.2}$ glasses can be used for IR optical wave guide applications and are thus important materials for high-speed telecommunications [1]. It was found that Ga is extremely important for the Er^{3+} activation, and secondly, that the IR photoluminescence (PL) shows a complex dependence on the Er content [1,2]. The latter results in a peak of the PL for an Er content of about 2%. The decrease of the PL above 2% has been explained as being connected with the formation of Er clusters. However, such clusters could so far not be confirmed by experiments.

Major problems to answer this question with usual x-ray techniques are
- the low concentration of the rare earth element
- the very similar atomic numbers (Z = 31, 32 and 34) of the elements of the base material
- the proximity of several absorption edges (for extended XAFS investigations).
- even the application of neutron scattering is difficult, as the respective scattering lengths are very similar (Ga: 7.288 fm, Ge: 8.185 fm, Se: 7.97 fm).

Anomalous x-ray scattering (AXS), however, can distinguish element-specific contributions [3]. The AXS contrast is mainly given by the difference in the atomic form factors at two energies near an absorption edge and the relative abundance of the element in the material. However, this implies that even a dopant can be analyzed by AXS if the form factor contrast is large enough. We therefore choose the L_{III}-absorption egde of Er, which provides a much larger contrast than the K-edges that are usually used for AXS experiments.

For this project, we already prepared samples with various dopant levels of Er. For the first experiment at Saga-LS, we used the undoped sample of $(GeSe_2)_{0.8}(Ga_2Se_3)_{0.2}$ glass, which should serve as a reference for future experiments of the Er doping.

３． **Experimental** (Note: Description of sample, method of experiment and analysis, etc.)

The AXS experiment was carried out using a standard diffractometer installed at BL15. An energy-dispersive SDD detector was used to discriminate the elastic scattering signal from spurious contributions of fluorescence lines and inelastic contributions. The energy resolution of the detector was about 300 eV at 10 keV. We also used a second SDD detector to monitor the fluorescence signals independently during the entire experiment, which greatly facilitates the data analysis.

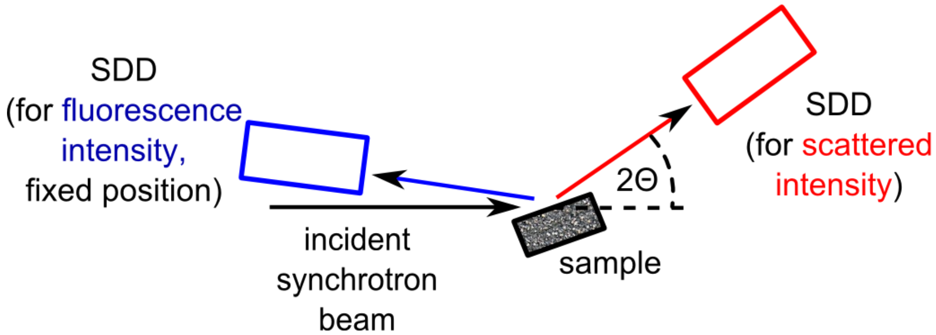

Fig. 1: Schematic setup for the AXS experiment. One SDD detector is used to collect the scattered intensity in a 2θ range, the other detector is fixed in a backscattering position and mainly monitors the fluorescence signals during the entire measurement.

The experiment itself was conducted at two incident x-rays energies of about 20 eV (**near** edge) and 200 eV (**far** edge) below the *K* absorption edge of Ga (10.386 keV) and Ge (11.104 keV). The scattering intensity was measured by a SDD detector in an angular range of about $2\theta = 3° \sim 130°$, in steps of about 0.5°. Each scan took approximately 4 h, so that a pair of incident energies could be measured during one day of beamtime.

The data were corrected for absorption effects and Compton scattering, and normalized using the Norman-Krogh-Moe method [4,5]. Further details on the theoretical and experimental background of AXS can be found elsewhere [3, 6-9].

4. Results and Discussions

In this experiment, we investigated the chalcogenide glass $(GeSe_2)_{0.8}(Ga_2Se_3)_{0.2}$ by AXS. We have thereby established the AXS method at the Saga-LS, proved the possibility to gain element-specific information and demonstrated the feasibility of the current AXS setup at BL15.

The differential structure factor of Ga, $\Delta_{Ga}S(Q)$, is displayed in Fig. 2 along with the experimental raw data. The results provide a basis for subsequent experiments (see also section 5 below) aimed at gathering information about whether or not Er clusters are forming in the glassy network, and about the relationship between Ga and Er atoms. These results are important to understand the role of each element and thus to develop optimized wave guides for telecommunications.

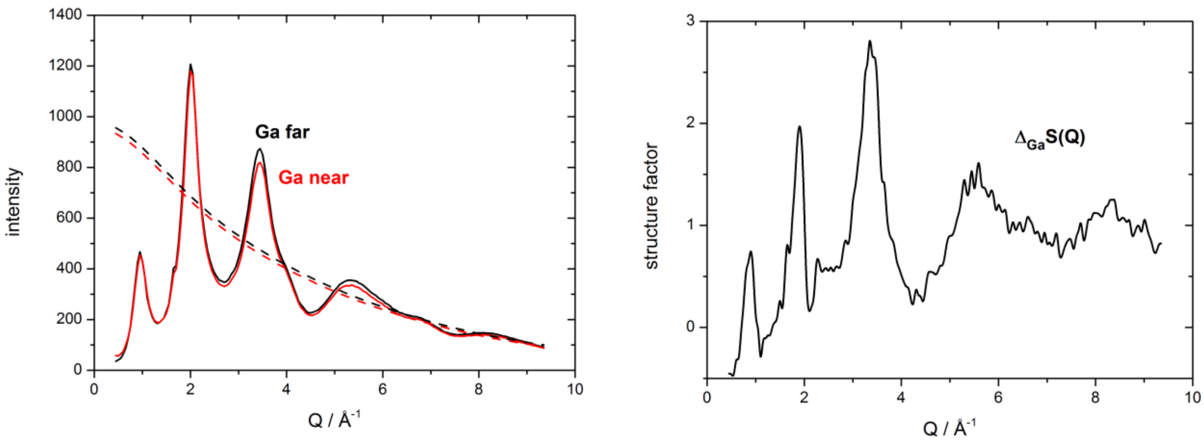

Fig. 2: Experimental scattering data of the $(Ga_2Se_3)_{0.25}(GeSe_2)_{0.75}$ chalcogenide glass collected at the Ga edge (left) and the corresponding differential structure factor of Ga (right).

5． **Future issues**

Future experiments will investigate the Er dopant in Ga-Ge-Se glasses and its impact on the photoluminescence properties. Since AXS is sensitive to the short- and intermediate-range order, we expect to gain information on the possible formation of Er clusters, which are expected for higher concentrations of Er, and about the role of the Ga atoms, which are a necessary requirement to solve Er in the glass matrix [1, 2]. Furthermore, we will acquire vital information on the feasibility of AXS for doped systems, and can thus expand the application of AXS to a whole new field of research.

6． **References**

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7． **Publications, patents**（Note: Typical deliverables related to this proposal.）

8． **Keywords**（Note: 2-3 words about samples and experimental methods.）

Ga-Ge-Se, chalcogenide glass, anomalous x-ray scattering, photoluminescence

9． **About the publication of research results**

Please delete either item ① or item ② indicated below, according to a user’s choice of publications as described in ※2. Also, please fill in an expected date of publication of a refereed journal article or an expected date of submission of a SAGA-LS Research Report. For example, deadline of both publications corresponding to the proposals performed in the fiscal 2017 is by the end of fiscal 2019 in both ① and ②.

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