EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:
https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report")*,
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round **- 5th March**
- 10th September Proposal Round **- 13th September**

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Structural Origin of the Enhanced Thermal Stability of Amorphous Ge45Cu5Te50	**Experiment number**: HC-4686
Beamline: BM02	**Date of experiment**: from: 25th January 2022 to: 3rd February 2022	**Date of report**: 4th August 2022
Shifts: 18	**Local contact(s)**: Gilbert Chahine, Nils Blanc	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Amorphous Ge45Cu5Te50 (GCT-9110) exhibits an increased crystallization temperature compared to binary GeTe [1]. EXAFS measurements revealed the formation of Cu clusters emebedded and linked to the surrounding Te enriched GeTe matrix in GCT-9110 and also GCT-7310 (Ge35Cu15Te50) [2]. In order to investigate possible structures of the Cu clusters and the structural effect of the clusters on the amorphous matrix it is necessary to have information not only about the short range order but also about the intermediate and extended range ordering in GCT-9110. This information can be obtained by anomalous x-ray scattering (AXS) which utilizes the change of the dispersion form factors f' and f'' near an absorption edge of an element of the sample to enhance the structural information around this element using the difference of two scattering laws, $\Delta S(Q)$.

The beamtime was divided into two parts. During the first part we used primary energies near the K-edges of Cu, Ge, Ni and near the Te K-edge during the second part. The experimental setup was as proposed. The samples were measured at room temperature under vacuum using a Be-dome to reduce air scattering. The scattered intensity was reflected by a graphite analyzer crystal to discriminate against fluorescence radiation. The detector for the first part was a two dimensional pixel detector with whom it is possible, in combination with the analyzer crystal, to analyze inelastic Compton scattering. This makes it possible to subtract the unwanted Compton intensity from the elastic line.

For the second part at high energies the detector was changed to a point-detector since the effectivity of the pixel detector strongly decreases at these high energies. Additionally, slits were mounted in front of the detector.

In the first part we measured successfully the differential structure factors $\Delta S(Q)$ at the Cu and the Ge K-edge of the proposed material GCT-9110. Therefor we measured the scattering intensity of the sample ($3° < 2\theta < 110°$) at 8.779 keV and 8.959 keV to obtain $\Delta S_{Cu}(Q)$ and at 10.903 keV and 11.083 keV to obtain $\Delta S_{Ge}(Q)$. The primary beam intensity at these energies was much higher than expected, sufficient intensities were collected for one measurement within 1 or 2h. These measurements were also conducted for GCT-7310.

Additionally, we changed the primary energy to the Ni K-edge to measure the backup samples GdNi and La55Ni20Al25 before changing to higher primary energies. These samples are metallic glasses which can be rejuvenated by cryogenic treatment changing the mechanical properties strongly. Thus, to understand the effect of the treatment the knowledge of how the amorphous structure changes upon treatment is crucial.

After finishing the scattering measurements at the low primary energies, the monochromator in the optical hutch was changed from the Si(111) to the Si(311) reflection for the usage of high primary energies. After the mentioned setup changes the primary energy around the Te K-edge was used. We measured the scattering of GCT-9110 at 31.513 keV and 31.783 keV. Since the bunch mode was changed (16b, 35mA beam current) the intensity was lower and longer measurements were scheduled by us. However, the intensity drop was stronger than expected, thus, to meet better statistics the number of points was reduced, the q-range shortened. Nevertheless, the statistics were not entirely satisfactory. For future measurements the 16b mode with 35mA beam current can not be recommended for AXS measurements.

The resulting ΔS(Q)s of GCT-9110 for each edge are depicted in Fig. 1. In reverse Monte Carlo simulations (RMC) they can be successfully fitted together with EXAFS data. Analysis of the RMC model shows that the Te enriched GeTe matrix of GCT-9110 is built of tetrahedral and defect-octahedral units whereas in comparison

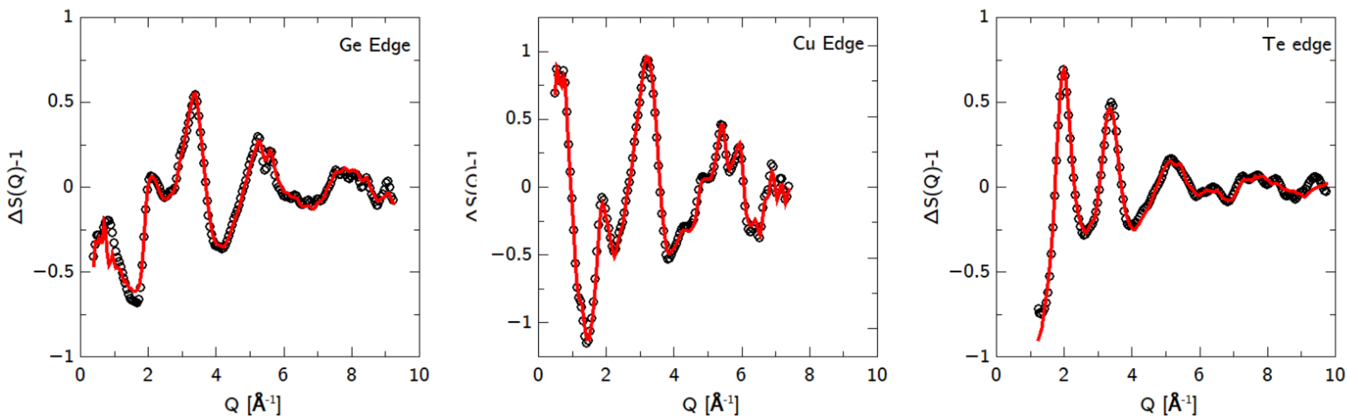

Figure 1 : Differential structure Factors ΔS(Q)-1 of GCT-9110 measured (black circles) near the Ge edge (left), near the Cu edge (center) and near the Te edge (right) and the corresponding RMC fit (red line).

with GCT-7310 the fraction of defect-octahedral sites diminshes with increasing Cu content. Furthermore, the RMC simulations indicate the preferred formation of disk-like Cu clusters.

Literature

[1] Y. Saito, Y. Sutou, J. Koike, J. Phys. Chem., 118, 26973, 2014, 10.1021/jp5066264.
[2] B. Paulus, J. R. Stellhorn, S. Hosokawa, B. D. Klee, Y. Sutou, W.-C. Pilgrim, Phys. Stat. Sol. B, 2100619, 2022, 10.1002/pssb.202100619.