ESRF	**Experiment title:** **Effect of x-ray irradiation in structural glasses**	**Experiment number**: HC-4988
Beamline: ID15A	**Date of experiment**: from: 08/11/2022 to: 14/11/2022	**Date of report**:
Shifts: 18	**Local contact(s)**: Marco Di Michiel	*Received at ESRF:*

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Report:

With the experiment HC-4988 we aimed at characterizing the structural impact of X-ray radiation of different energies in some oxide and chalcogenide glasses. In fact, while pioneering studies with X-ray photon correlation spectroscopy have shown that ~10keV photons induce atomic scale rearrangements and bring glasses in new disordered configuration [1], a quantitative and complete characterization from a structural point of view (e.g., with pair distribution functions and at different photon energies), was still missing. The experiment HC-4988 performed at ID15A allowed us to explore accurately - through total diffraction scattering - the effects of different photon energies (8keV and 75keV) on the structure of oxides and chalcogenides.

Before the experiment, we prepared the samples (for each of the explored compositions) irradiated with 8keV photons at different total doses. During the diffraction experiment at ID15A, we collected the scattered intensity from different sample's spots: this procedure allowed us to obtain repeated measurements and statistically robust results. We successfully measured all the prepared samples.

We have been evaluating the induced differences in the diffraction patterns and the data analysis is in progress. In detail, the calculation of the pair correlation functions is still ongoing and, once completed, will allow us to better understand the type of structural photo-induced changes and compare oxide and chalcogenide glasses.

The second part of the experiment was dedicated to the characterization of the role of different energies, which can trigger different microscopic phenomena (for example, for high energy photons knock-on events start to

play a role), leading to quantitatively and/or qualitatively different structural effects. For this reason, we studied samples irradiated with photons at 75keV. In this part, we directly employed the diffraction beam for irradiation, in a pump and probe configuration. We followed the evolution of the diffuse scattering as a function of the exposure time to the X-ray beam. In this case, both the temporal variations of the structure on the irradiated spot and the spatial extent of changes in the amorphous network were observed.

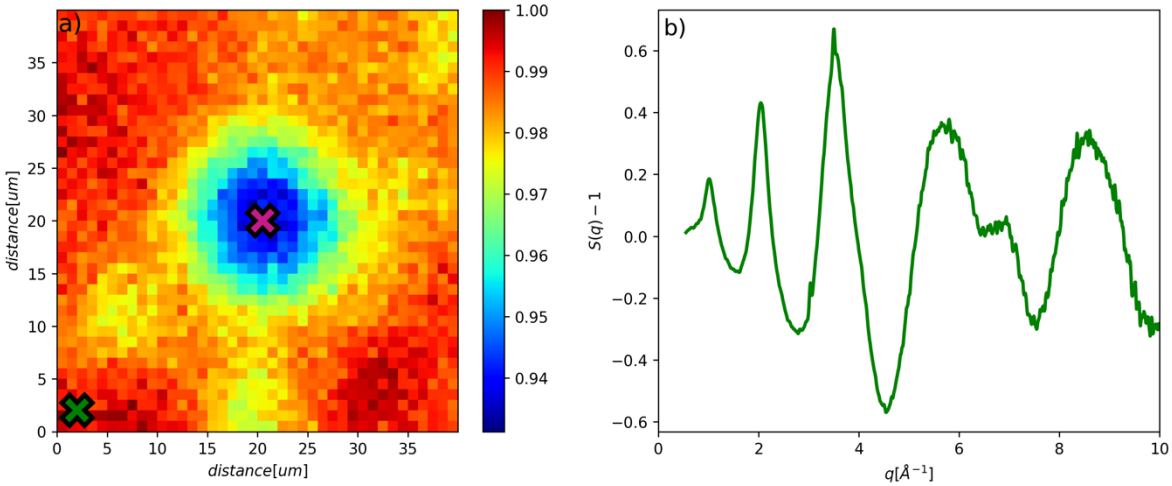

Figure 1: Binary Ge-Se glass irradiated at 75 keV. a) 2D mesh showing the normalized average intensity across the second peak of the diffraction pattern. The beam impinged at the center of the mesh (pink cross). b) Preliminary structure factor of a pristine glass. The curve was obtained at the edge of the mesh (green cross), where there are no irradiation effects.

In Fig.1 we show the effect of the 75keV beam on a sample of $GeSe_2$ after 360s of exposure. The 2D map reported in panel *a*) shows the normalized average scattered intensity across the second sharp diffraction peak of the structure factor as a function of the position on the sample around the irradiated region. The blue region, clearly visible in the proximity of the X-ray impinging beam axis, indicates that the irradiation causes a significant reduction of the scattered intensity. In Fig.1*b*) we show a preliminary S(q) obtained from a peripheral spot (green cross in panel *a*), where the sample is in its original conditions (as-prepared). Interestingly, we found out that the structural changes not only involve the first diffraction peak but extend to the entire structure. However, a deeper analysis is required to obtain quantitative information.

The large amount of scattering data collected (2.8 Tb) requires significant computational resources to be fully processed and analyzed. The data processing is currently underway, and it is expected to take a few more months to get the entire set of pair-distribution functions.

References

[1] B. Ruta *et al.*, Scientific Reports 7, 3962 (2017)