



	Experiment title: Rigidity and stress transitions in GeSe glasses	Experiment number: HC1425
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Shifts: 18	Local contact(s): Beatrice Ruta	<i>Received at ESRF:</i>
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Report:

XPCS is the very only technique able to monitor the decay of the structural relaxation at the atomic length scale and on the second timescale that is relevant to glass transition phenomena. Previous XPCS data obtained on metallic and silicate glasses have indicated that markedly different slow dynamics can be found depending on the glass structure. The main aim of the proposed experiment was to evidence for the first time a composition-driven change of the structural relaxation when crossing the rigidity transitions in the canonical $\text{Ge}_x\text{Se}_{100-x}$ binary glass system.

As originally proposed, six homogeneous glasses with increasing Ge content were prepared for the experiments. However it was found very difficult to polish them as disks of 5 mm diameter, thickness ~ 25 μm to optimize the scattering intensity and the coherent properties of the X-rays at the fixed incident energy of 8 keV. They inevitably broke for thickness below ~ 50 μm in small pieces of μm size. Nevertheless it was possible to load them in the measurement cell, make the alignment and eventually get signal as the beam sizes only $\sim 10 \times 10$ μm^2 at sample position.

For three samples spanning the series we tried to measure at room temperature the ISF at the first maximum of the static structure factor $S(Q)$ where the intensity is maximum, then at lower Q where the dynamics is expected to be slower. We used the wide-angle setup, which become available a couple of year ago. To enhance as much as possible the weak measured signal, speckle patterns were recorded by using two IkonM CCD from Andor Technology (1024 \times 1024 pixels, 13 \times 13 μm^2 pixel size) installed perpendicularly to the horizontal scattering plane, ~ 67 cm downstream of the sample, and symmetrically with respect to the incoming beam. In this way, the two detectors cover the same solid angle and the same wave vector Q with a resolution of $\Delta Q = 0.04 \text{ \AA}^{-1}$.

In all the samples investigated, the dynamics was too fast to be properly measured by the XPCS setup. Only the tail of the ISF was obtained as plotted in Figure 1 for the $x=30\%$ glass. We further tried to slow down the dynamics using a classical annealing treatment just below the glass transition temperature for two samples but one crystallized after a couple of hours ($x=30\%$) and the other one was still too fast ($x=19\%$).

We used the remaining time to complete a previous experiment on vitreous silica as a function of temperature (Experiment number HC1140). The analysis of the data is still ongoing but we are confident that these results will strongly improve our knowledge on the glassy state.

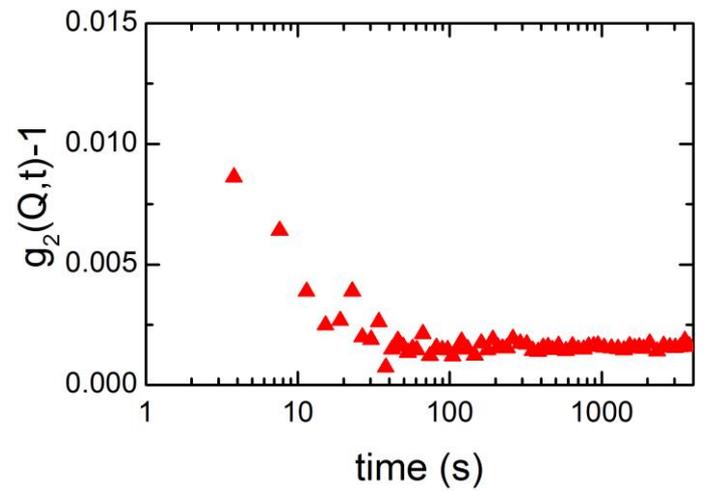


Figure 1: Room temperature unnormalized intensity auto-correlation function measured at the maximum of $S(Q)$ on $Ge_{30}Se_{70}$.