ESRF	**Experiment title:** Heterogeneous elasticity of permanently densified silica glass probed by XPCS at wide angle.	**Experiment number**: HC-3441
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Report:

The aim of the experiment was to measure the beam induced dynamics of vitreous silica as a function of the glass density at room temperature. The idea was to try to better understand the mechanism underlying the beam induced dynamics and to relate it to the known presence of heterogeneities in the elasticity of glasses.
To this aim, we have prepared a set of three permanently densified silica samples. The permanent densification was performed at the Institute of Materials for Electronics and Magnetism of the Italian CNR in Parma. The samples were obtained from a commercial-grade Spectrosil block glass, v-SiO_2, purchased from SILO (Florence). The starting block was drilled in cylindrical pieces of two sizes, 5.6 mm diameter and 5 mm length for the lowest densities and 3 mm diameter times 3 mm length for the highest density. A high-temperature high-pressure multianvil apparatus was used to obtain the permanent densification at pressures of 4, 6 and 8 GPa. The samples were heated at T=500 °C for 10 min with a heating rate of 50 °C/min; the pressure was released after quenching. The final products were homogeneous and permanently densified.
We performed the XPCS experiment at 8 KeV incident energy. For this reason we had to reduce the thickness of the samples down to approximately 50 μm, in order to optimize the sample transmission and the contrast of the correlation curves. The thin samples were prepared by a polishing procedure, using abrasive sheets, resulting in flat and transparent samples.

We performed the experiment on four samples: the three permanently densified ones and a reference sample of normal SiO_2. We started the experiment using the standard setup fo XPCS at wide angles, with an Andor CCD placed at 70 cm from the sample holder. The setup is designed in such a way that the speckle size on the detector matches the pixel size, which is 13 μm for the CCD. The Andor camera has 1 million pixels and a readout time of approximately one second, limiting the accessible time window to longer times.

Differently from the normal silica sample, the densified glasses show a beam induced dynamics that is time dependent. This kind of "aging", or evolution with experimental time of the dynamics is visible already from the two-time correlation function, as shown in Figure 1 for the case of the 4 GPa sample.

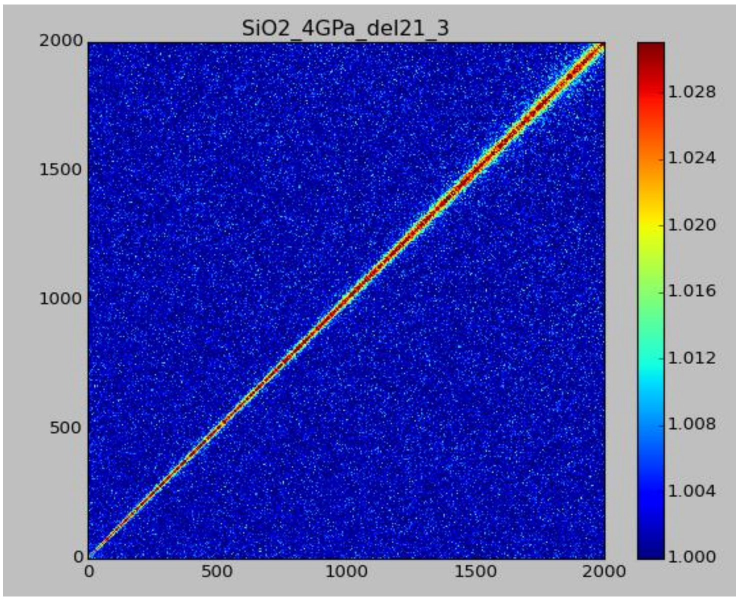

Figure 1: two times correlation function measured with the Andor CCD on the structure factor peak of the 4 GPa sample. The intensity values are indicated by the color bar. The width of the diagonal increases with time indicating an increase of the decorrelation time with the age of the system. The axes are in seconds.

The decorrelation time increases during the acquisition and after 1000s is of the order of 50s. In the samples with higher densities the decorrelation time is smaller, making it difficult to measure the correlation curve with the Andor CCD. For this reason, we decided to change the experimantal setup and to use the Eiger detector. This is a 500K pixel detector (500 x 1000) with single photon counting capabilities and a very small readout time (of the order of 10^{-5} s). The pixel size is 75 mm, so that the detector has to be mounted at 4 meters from the sample. The setup at wide angles was composed of a small translation stage, with an extension of approximately 60 cm, where we mounted the detector and a second translation stage to hold the evacuated pipe of the appropriate length (4 m). This setup allowed us to scan an angular range of 8 degrees around the structure factor maximum, corresponding to a q range between 11.5 and 17 nm^{-1}. The smaller number of pixels of the Eiger detector compared to the CCD gave rise to a reduction by a factor of two of the measured solid angle.

Nevertheless, the quality of the measured correlation curves is very high, as can be seen in figure 2 for the 4 GPa sample. Specifically, we obtained an extension of the experimental time window by more than one order of magnitude towards the fast times. The curves in figure 2 are obtained with an integration time of 0.3 s but the signal was sufficient to allow us to use also smaller integration times, of the order of 0.1 or even 0.05s.

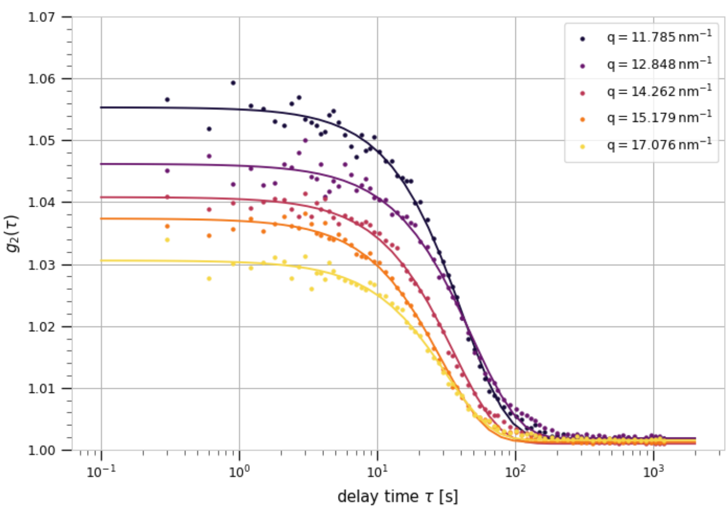

Figure 2: Correlation functions of the 4 GPa sample obtained with the Eiger detector at wide angles. The colors correspond to different values of the exchanged wavevector as indicated in the legend. The curves are the best fitted function with a Kohlrausch-Williams-Watts shape. Unpublished data.

We used the Eiger detector to measure the dynamics of the four samples as a function of exchanged wavevector and at different values of the incident flux, to monitor the beam induced effect. We also studied the evolution of the dynamics with acquisition time. The analysis of the data is still ongoing but we are confident that these results will strongly improve our knowledge on the glassy state and will surely deserve to be published.