ESRF	**Experiment title:** X-ray induced amorphous-amorphous transition in vitreous silica	**Experiment number**: HC-4789
Beamline: ID22	**Date of experiment**: from: 08/06/2022 to: 13/06/2022	**Date of report**: 13/09/2022
Shifts: 12 (+3 added onsite)	**Local contact(s)**: Ola gjonnes Grendal	*Received at ESRF:*

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Report:

The aim of the experiment was to investigate the structural evolution of v-SiO_2 under x-ray irradiation by means of high-resolution x-ray diffraction. Recent x-ray photon correlation experiments (XPCS) have revealed that the x-ray beam simultaneously pumps and probes the dynamics of oxide glasses at temperatures where the structural relaxation is frozen. The observed dynamics is accompanied by a progressive rearrangement of the glass structure, which evolves towards a different amorphous configuration. The data were essential to study the structural details of this amourphous-amorphous transition. The sample was v-SiO_2, a commercial grade Spectrosil purchased by Silo company (Florence). It has been optically polished via carbon grinding paper to achieve a thickness of about 50 μm (the same used in our XPCS experiments). The sample holder was a parallelepiped made of plexiglass with a hole in the centre for fixing the sample.

The standard setup for powder diffraction of the ID22 beamline has been adapted to our bulk sample to go in transmission geometry. The diffraction pattern has been collected by a Perkin Elmer XRD 1611CP3 detector 40 cm far away from the sample stage. The standard PDF routine at ID22 requires x-rays at 72 keV. In this high energy condition the attenuation length of v-SiO_2 is above several mm meaning that it is completely transparent. So the delivery of the dose [absorbed energy/mass] have been done at 8keV to induce the amorphization and the structure has been consequently analysed at 72 keV on the same spot.

As a first step we have calibrated the wavevectors on the detector using LaB6 sample for both the energies, 72 and 8 keV. Consequently, we acquired firstly the empty sample holder for collecting the background and we

mounted after the sample for the measurement of the pristine glass. By looking at the intensity of the diffraction profile we decided an exposure of 1 hour for every data acquisition at 72 keV. Next we started to irradiate at 8 keV. Every switch in energy involved a misalignment in the position of the focal spot which was 50 µm big. The technical difficulty of the experiment was to get a micrometric control of the spot position to be sure to irradiate and probe always the same region. The starting idea was to put a gasket before the sample but we observed its diffraction rings in the measurement, so we decided to put this gasket on the edge of the sample holder.
For every energy change we adjusted the relative position of the two x-ray beams, 72 and 8 keV, by looking it with the gasket.

We acquired the structure with 72 keV x-rays after every 15 hours of irradiation at 8 keV, approximately.

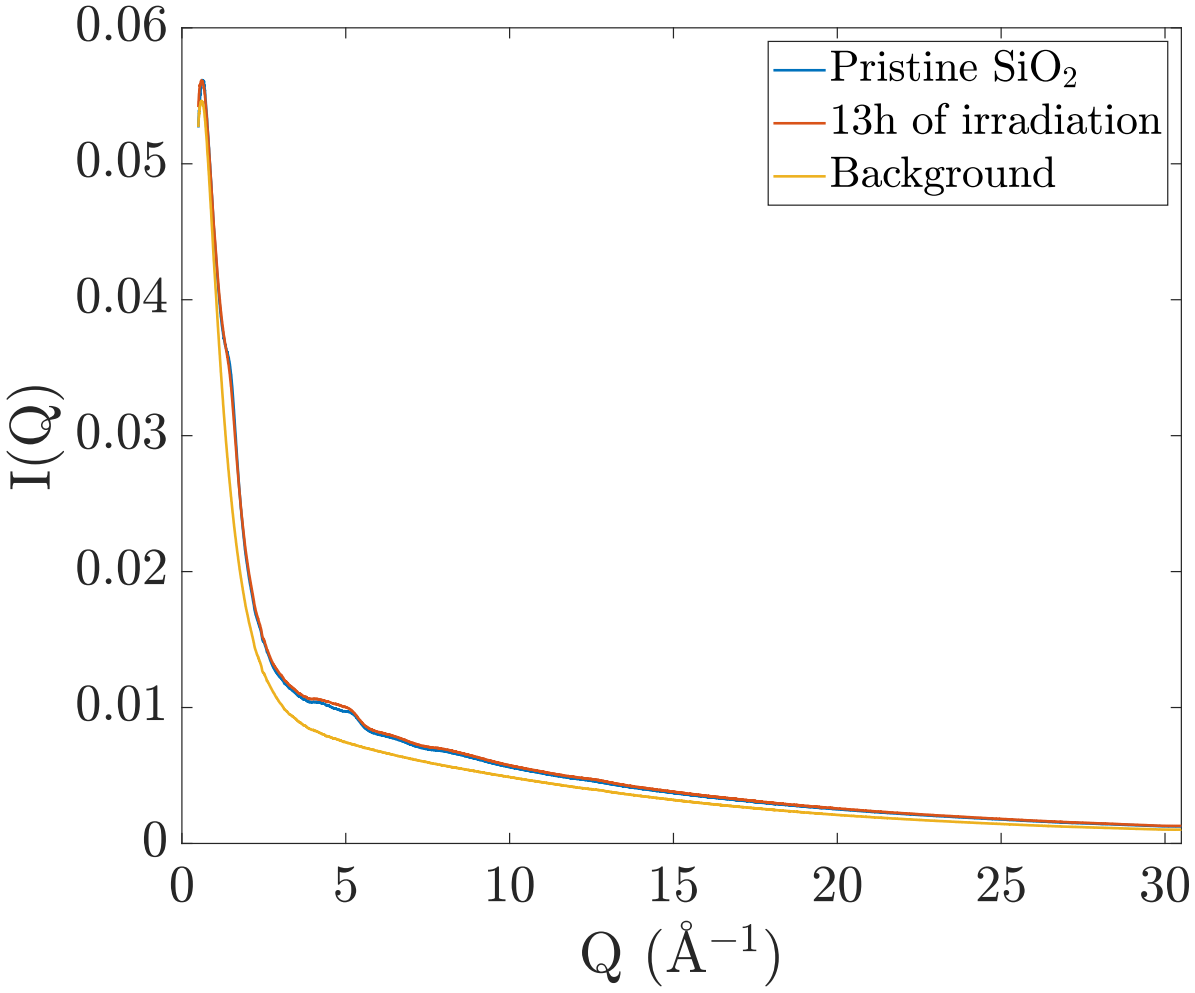

Figure 1: Intensity diffraction pattern as a function of the exchanged wavevector acquired with x-rays at 72 keV. The blue curve is the data for the pristine silica, the red curve is the data on the same sample region acquired after 13 hours of irradiation at 8 keV and the yellow curve is the background signal acquired before mounting the sample on the sample holder. The prolungated irradiation has induced a damage in the sample visible by the reduction in the intensity of the first diffraction peak (the shoulder at ~1.6 Å^{-1}) and a change in the shape of the peaks at higher orders
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We succeded in studying the structural features of the induced amorphous configuration. The data analysis is still ongoing and we plan to use both the pyFAI and the pdfgetx3 packages to recover the pair distribution function.