ESRF	

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Report:

The aim of the proposed experiment was to induce a metamictization of the α -quartz and monitor the dynamics of the transition by means of the X-ray Photon Correlation Spectroscopy (XPCS). The proposal wanted to investigate the beam-induced dynamics, the fast relaxation activated by an intense x-ray beam in oxide glasses. To pursue this aim we have prepared two samples: a SiO₂ α-quartz single crystal (MaTeck GmbH) and a commercial grade v-SiO₂ sample (Spectrosil from SILO company). Prior to the experiment, the α-quartz has been aligned at the department of Geoscience in the University of Padova. The two samples have been prepared in the shape of disks, optically polished with abrasive sheet.

The entire experiment has been performed in the WAXS configuration, mainly at 19.2°. The EigerX4M detector for the photon correlation has been mounted at 5.01 m distance to the sample stage. The setup geometry allowed to have the speckle size on the order of the pixel dimensions, 75 µm. A second detector (Pilatus300K) was mounted close to the sample stage to record the scattered intensity on a wider Q region.

As the first step we have calibrated the wavevectors on the Pilatus300K using a LaB6 sample.

We have studied firstly the silica sample because we had done several previous XPCS experiments on it. This has shown a significant change in the structure for high doses meaning that we were testing a different range of the beam induced dynamics.



Figure 1. Scattered intensity as a function of the wavevector for the vitreous silica sample recorded by the Pilatus300K. The detector covers up to 3.3 $Å^{-1}$. The long irradiation induces a damage clearly visible by the broadening of the first sharp diffraction peak.

Regarding the dynamics extrapolated from XPCS analysis, the vitreous silica showed an initial fast decorrelation of about 3s which slowed down progressively as a function of the dose. The EigerX4M detector allowed us to get the complete decorrelation curve with high resolution because of its nearly absent readout time and a speed of up to 750 Hz.

Next, we have studied the α -quartz. We have put the [110] reflection on the Pilatus300K and we inserted all the attenuators along the beam path to avoid saturation damage on the detector. During the irradiation we have seen a reduction in the intensity of the Bragg peak. Next, we rotated the sample in a way that no Bragg peaks were present neither on EigerX4M detector nor on the Pilatus300K. After a TGy of dose, the sample started to present a decorrelation with relaxation times typical of the amorphous silica together with the rise of the ring at 1.5Å⁻¹, clearly visibile on the Pilatus300K detector.

Figure 2. Correlation curves of both the investigated samples: α -quartz (yellow squares) and vitreous silica (blue dots). The two correlation curves have been done at different doses: vitreous silica at 1.5 TGy and α -quartz at 8TGy. Both of the curves show a compressed shape and in the case of the quartz, the value is bigger than the vitreous sample even if the dose is nearly quadruplicated.

We tested the correlation at different photon fluxes to check the beam induced dynamics for both the samples. At the end we studied the dynamics and the structure at different positions along the previously irradiated region



Because of the large amount of data collected, the detailed data analysis is still ongoing. We are confident that the excellent data quality, togheter with the interesting phenomelogy will result in a pubblication on a high impact journal in the next few months.