Report

Introduction

In a first experiment at ESRF (ME1000) it was demonstrated that SAXS experiments are a good way to study the poling mechanism in Lanthanum-Boron-Germanium (LBG) glassy system. Poling is a technique used to break glass isotropy. We performed two kinds of poling on LBG glasses :

- thermal poling[1] which consists in applying an external electric field of several kV.mm^{-1} between two electrodes in physical contact with the glass. At the same time the glass is heated at several hundredths of degrees (depending on the glass transition temperature value) in order to facilitate orientations by release of the glass network.
- UV poling[2] which consists in irradiating glasses with UV and heating the sample at the same time.

In both cases birefringence has been observed with a polarizing microscope. SAXS experiments were performed to put in evidence the poling induced anisotropy in glass matrix. Since from X-Rays diffraction of the thermally poled samples there was no evidence of a cristalline structure, the anisotropy is supposed to concern the orientation of fragments of the amorphous matrix or nano-crystals dispersed in the glass. Stillwellite ferroelectric nano-crystals are assumed to be responsible for the observing thermal poling induced birefringence. However the mechanism of thermal poling is still debated. It can be due to a migration of mobile impurities towards the cathode. In this case the non linear properties are concentrated in a thin layer at the anodic surface. This is what is observed in thermally poled silica glasses[3],[4].

Experimental

During the experiment carried out from the 28^{th} of September to the 2^{rd} of October 2006, we built a new set up allowing us to perform in-situ thermal poling experiments : a DC high voltage supply was delivering until 10kV in a very small furnace allowing regulated temperature from 20°C to 520°C. A glassy sample was put inside the furnace between two inox electrodes and X-rays were sent perpendicular to the electric field in order to test anisotropy induced in the sample. Before the start of the experiment, technicians and engineers of LPCML came to ESRF to adjust the mechanical set up. The experimental set up was therefore ready on September 28^{th}. SAXS experiments were also performed on UV-irradiated LBG samples.

Results

UV poling

SAXS profiles shown in figures 1and 2 reveal the anisotropy induced by UV-irradiations in LBG glasses. Two profiles have been recorded on the same sample at different positions. The profile is completly isotropic when it is recorded on a non UV-irradiated area, whereas it shows clearly anisotropic shape on a UV-irradiated area.

Thermal poling

A first set of experiments was devoted to scan the samples from one electrode to the other one, whereas the sample was maintained at a steady temperature (300°C) and a constant voltage (10kV). Several samples[1]with different sizes implying a more or less intense electric field were tested. This experiment takes advantage of the small size of

FIG. 1 – SAXS profile recorded on non UV-irradiated area in LBG glass

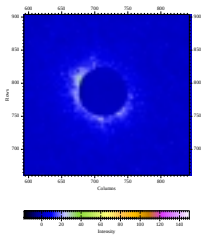
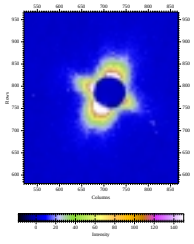


FIG. 2 – SAXS profile recorded on UV-irradiated area in LBG glass



the X-ray beam (300 microns). However our samples were small (around 2mm high) in order to apply a high electric field. We observed then by scanning the sample that we had to be very careful before saying that anisotropy was observed. Actually we observed (see figures 3 and 4 that when the X-ray beam was moved in the neighbourhood of the electrodes, the SAXS profile was distorded by edges diffusion sample.

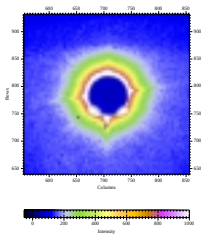


FIG. 3 – SAXS profile recorded in the center of the LBG glass

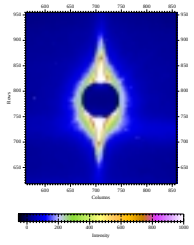


FIG. 4 – SAXS profile recorded near one electrode of the LBG glass

Conclusion

SAXS experiments done on UV-irradiated samples appeared to be a successfull way to put in evidence anisotropy in glasses. We installed the thermal poling experimental setup and observed that we should not jump into conclusions too fast : a distortion of SAXS profiles can be due to anisotropy in the glass but also to edges diffusion of the sample. Aware of this problem, we want to do further experiments on thermal poled glasses.

References

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