| ESRF | **Experiment title:** In situ thermal poling in glasses: voltage, temperature and position dependencies | **Experiment number**: 02 01 726 |
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## Report:

### Introduction

The last experiment 02-01-690 demonstrated the ability of BM02 facilities to probe anisotropy in Lanthanum-Boron-Germanium (LBG) glassy system which was irradiated before with an UV nanosecond laser. This technique induces very localized birefringence which is observed with a polarizing microscope. Another technique called "thermal poling" induces birefringence in the bulk of the sample. It 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. The anisotropy, which is observed in the whole bulk for LBG glass, is supposed to concern the orientation of fragments of the amorphous matrix or nano-crystals dispersed in the glass. Stillwellite like ferroelectric nano-crystals are assumed to be responsible for the birefringence induced by thermal poling. 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[1,2] and tellure glasses.

### Experimental

For our experiment 02-01-690, we built a new set up allowing us to perform *in situ* thermal poling experiments. For the experiment 02-01-726, we improved our set up, taking into account the difficulties during the previous experiment: a DC high voltage supply was delivering until 10kV in a furnace smaller than before allowing to reach rapidly a regulated temperature from 20°C to 650°C. The kind of electrodes was changed: stainless steel electrodes were replaced by silicon electrodes covered with a thin layer of silica in order to prevent injections of charges into the sample and to provide a better contact between sample and electrodes.

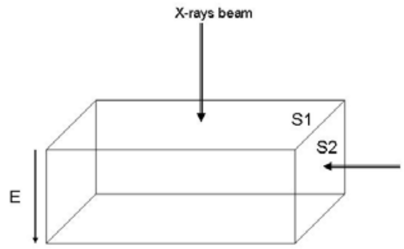
First one LBG sample which was *ex situ* thermally poled (300°C and 8kV/mm) along one direction was probed. Then *in situ* thermal poling was performed in vacuum, for different temperatures and voltages, on two kinds of glassy systems: LBG ($La_2O_3$-$B_2O_3$-$GeO_2$) and Tellure (70 $TeO_2$-30 ZnO with $Na^+$ ions) systems.

Plates were polished before to a thickness of 250 µm because these glasses contain heavy atoms (especially lanthanum atoms) which absorb X-rays in a large amount. The energy of the X-rays beam was 19 keV.

## Results

### *Ex situ thermal poling*

SAXS profiles of thermally poled LBG sample are shown below. X-rays are first focused on the surface S1 perpendicular to the poling field direction (a) and then on the surface S2 in both orientations related to the electric field (b1) and (b2).




The SAXS profiles are shown in figure 1. These different geometries of the sample versus X-rays beam show that SAXS instruments are able to detect anisotropy (on figures (b1) and (b2)), even if the effect is very light. A preferential direction along the electric field is evidenced by the two last spectra.

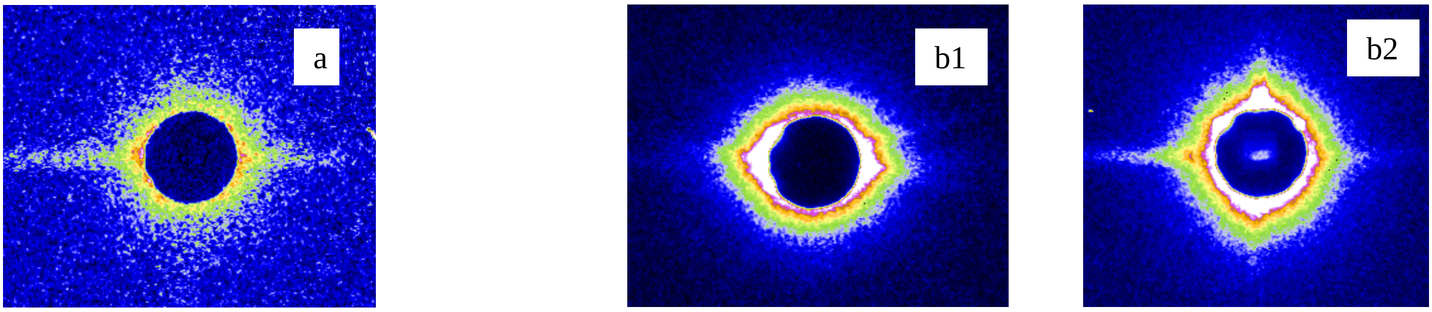



***Fig. 1***: SAXS profiles recorded in different geometries: (a) X-rays are focused on the S1 surface, (b1) and (b2) X-rays are focused on the S2 surface, the electric field direction is either horizontal either vertical.

### *In situ thermal poling*

When submitted to high voltage and high temperature, the very thin samples of LBG or tellurite glass were supposed to break their isotropy. In order to discriminate the mechanisms which may occur during poling, it is relevant to scan the sample between both electrodes. Near one electrode, the recorded spetrum remains isotropic (not shown here) and no edges diffusion of the sample was observed. The physical contact between electrodes and sample was therefore improved.

One LBG sample heated until 500°C and submitted to 10 kV shows an isotropic SAXS profile excepted at the end of the experiment, at ambient temperature. It showed there an anisotropic SAXS profile (see figure 2), only near the electrode, which lead to make the assumption that a surface cristallisation may have occured[3]. This was checked with diffraction experiment but no sign of crystals was observed. Excepted this phenomenon, no anisotropy was detected in LBG or tellure glasses.

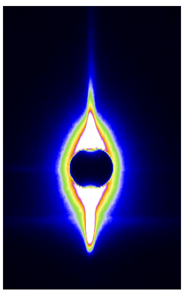


***Fig. 2***: SAXS profile recorded in LBG glass near one electrode after "poling" at ambient conditions

## Conclusion

We improved our set up to perform *in situ* thermal poling, especially concerning the physical contact between glass and electrodes. However the expected results were missing for mainly two reasons: because of a lack of control on voltage and current in the sample and because the samples were too thin to show a visible anisotropy. A light anisotropic signal related to the orientation of the sample was nevertheless detected at the beginning of the experiment on a thicker sample poled *ex situ*.

## Bibliography

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