ESRF	**Experiment title:** X-ray Poling in silica glasses	**Experiment number**: ME-1300
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Shifts:	**Local contact(s)**: Nathalie Boudet	*Received at ESRF:*

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Report: We got several runs on ESRF in 2005 for studying X-ray poling. The problem was not obvious : it was to store an electric field in an insulator using X-rays by monitoring the energy of the X-rays and controlling the charge movement applying an external electric field.

We performed all the analyses planned in the project excepted that the beam energy was so difficult to change that we finally kept the same. This was very annoying as we did not know if the chosen energy was good for our problem (the future showed that in fact it was not the case). We spent a lot of time for understanding the leakage currents in the experiment as typically we were measuring a few picoamps. But we failed to find the answer in the time reserved. We restarted the study on Elettra later and we found the answer : there is current of the same magnitude arising from scattered X-rays on the gas molecules. In addition, at Elettra we were able to move the monochromator and understood the X-ray penetration effect. There was a compromise to find minimising the X-ray penetration and maximising the photoinduced conductivity.

So, we are depressed to say that finally, we were not able to achieve a publication or a communication at a conference with results obtained at the ESRF but at Elettra.

The enclosed paper has been accepted in the conference Silica 2006, 25-28 June Palermo Sicilia, at CSC2006 3-7 July 2006 Tours, France, and also at EOS topical meeting on Non-linear Optics 16-19 October 2006 Paris.

Optimization of the X-ray poling process, R. Blum[*], B. Poumellec[*], R. Cortes[**], S. Gree[*], M. Lancry[*]

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1. INTRODUCTION

Over the past decade, the large increase in worldwide needs for communication and the growth of fiber optics based network, have created a need for silica-based active optical components. Since the discovery of thermal poling by Myers *et al*., tailoring second harmonic generation (SHG) within silica glasses, this subject became a vast field of studies. Beside thermal poling, different poling processes such as UV assisted poling or e-beam poling, have then been demonstrated. Poling efficiency differs from one process to another, but the SHG is defined always by a non-linear coefficient d_{33} which is in the order of 1 pm.V^{1}. This coefficient is proportional to intrinsic third order optical non linear coefficient (always existing) and a poling induced electric field (the poling field).

A year ago, we were the first to report a novel method of poling by mean of conjugate action of x-ray irradiation and high external electric field. A former study, mentioned X-ray irradiation as a pretreatment method in order to enhance thermal poling efficiency but it was the first time to our knowledge that poling was achieved by such mean. This process, later referred to as X-ray poling, had many interesting features but needed optimization.

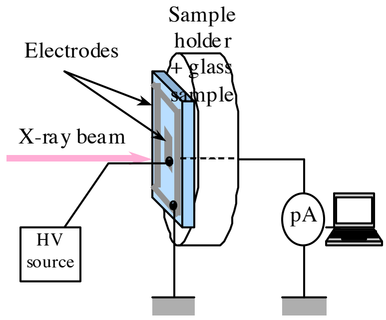

Figure 1 : Description of electrical connections on sample

2. EXPERIMENTAL DETAILS

Poling was performed on 1mm thick spectrosil pure silica sample. Three thin silver electrodes were deposited on the surface of the samples by ion sputtering: one central electrodes on each side of the sample for application of the 2 kV external electric field and the third as a guard electrode. Electrical connectivity was ensured by conductive carbon tape. Electric field (2 kV) was applied using a Hamamatsu voltage source. Leakage current was continuously measured during poling using a Keithley 487 picoammeter. X-ray irradiation was performed on the XRD beamline of Elettra synchrotron facility. Electric field was applied on sample side facing the x-ray beam, and current was measured on the back electrode to ensure a better isolation of the picoammeter from air ionization under x-rays. The guard electrode was grounded. (See figure 1 for a scheme of the set-up). Different sets of irradiations were performed using energies from 6 to 11 keV. All irradiation parameters were tested: x-ray energy, x-ray dose, fluence and poling time.

SHG efficiency was determined using a standard Maker fringe set-up equipped with a 1064 nm YAG laser (1kHz, 170 ns pulse duration, 520 mW mean power).

3. RESULTS

1. Current measurements

Prior to poling treatment, we tested our material using a standard poling/depoling cycle. During poling, both High Voltage (HV) and x-rays (RX) are applied, while during depoling HV is shut off but sample is still irradiated. Current measurements for two successive test cycles at different x-ray flux, are shown in 2.

Many interesting points can be noted on such current measurements:

-during poling, when RX are turned on, we can observe an induction peak followed by a plateau which is directly related to the induced photoconductivity.

-The level of the plateau is related to Xray flux. That is quite normal, a higher flux implies a higher photoconductivity and thus a higher current.

-Surface under depoling peak seems to be proportional to surface under poling peak.

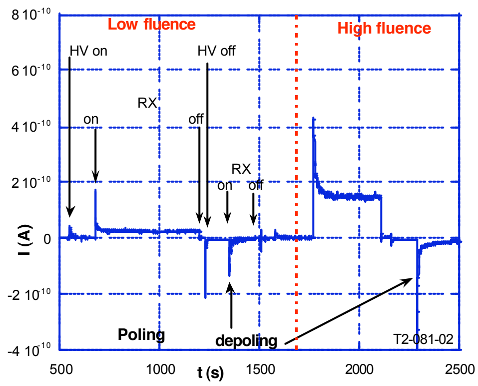

Figure 2: Current measurements during two successive test cycles with varying x-ray flux.

Sample #	E_{RX} (keV)	poling t (s)	flux (ph/s)	dose (ph)	$P_{2\_}$ (nW)
1	6	200	$1.47\ 10^{12}$	$2.93\ 10^{14}$	1.45
2	6	17850	$7.05\ 10^{8}$	$1.26\ 10^{13}$	0.52
3	8	1800	$6.12\ 10^{10}$	$1.1\ 10^{14}$	7.96
4	8	150	$6.12\ 10^{10}$	$9.18\ 10^{12}$	breakdown
5	8	45	$6.56\ 10^{10}$	$2.95\ 10^{12}$	breakdown
6	8	5	$6.56\ 10^{10}$	$3.28\ 10^{11}$	weak
7	9	2700	$5.75\ 10^{11}$	$1.55\ 10^{15}$	5.23
8	8.7	700	$1.02\ 10^{11}$	$7.14\ 10^{13}$	3.3
9	8.7	75	$9.04\ 10^{10}$	$6.78\ 10^{12}$	No signal
10	8.7	30	$9.04\ 10^{10}$	$2.71\ 10^{12}$	No data
11	8.7	5	$9.04\ 10^{10}$	$4.52\ 10^{11}$	1.4
12	8.7	1	$9.82\ 10^{10}$	$9.82\ 10^{10}$	1.03
13	11	1100	$1.14\ 10^{11}$	$1.25\ 10^{14}$	0.31
14	11	380	$1.24\ 10^{11}$	$4.7\ 10^{13}$	0.58

2. Optical Nonlinearity

Table 1: Poling conditions and induced Non-Linearity

Comparison of the samples can be achieved using the value of the SH peak power. Table 1 summarizes poling conditions and measured optical properties for all samples. For all poling energies, a general trend can be established: the higher the Xray dose, the higher the SHG. The signal seems to be optimal for an Xray energy of 8 keV.

3. Interpretation

Interaction of X-ray with silica generates electron/hole pairs. These species migrate in opposite direction under the influence of the high external electric field. Once trapped, they generate a space charge responsible for a high electric field that induces SHG. The X-ray photon energy is the key parameter. X-ray penetration is fitted to the material in fixing the energy in order to obtain a strong attenuation of the X-rays for minimizing the transport current (photo-conductivity effect) but nevertheless allowing reaching the steady state (kinetics). The distribution of electrons and holes in the thickness of the sample is thus dependent on the X-ray energy. We are currently working on a model of charge migration that tends to proof that 8 keV is the appropriate value for inducing the highest electric field and thus the highest SHG.

4. CONCLUSION

Results shows that it is possible to obtain poling efficiency of the same order than with other processes, but there are some advantages in favour of this method.

- X-ray energy can be chosen according to the sample thickness for a strong enhancement of the built in electric field.
- the steady state is reached within an hour with a synchrotron beam for a mm thick sample. Due to energy band width, the fluence ratio with a laboratory generator is of the order of 3 orders of magnitude. These ratio can be overcome considering thin films 2 orders of magnitude thinner and taking into account that the built-in field can be achieved by storing in this case and not by screening.

Several studies are needed to complete the work :

- enhancement of $\chi^{(3)}$ by appropriate irradiation,
- dynamic simulation of the process.