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VACUUM POLING: AN IMPROVED TECHNIQUE FOR EFFECTIVE THERMAL POLING OF SILICA GLASS AND GERMANOSILICATE OPTICAL FIBRES

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Abstract

The use of thermal poling *in vacuum* to eliminate the spreading out of the poled regions beyond the boundaries of the positive electrode is proposed and experimentally demonstrated. A substantial improvement in reproducibility and quality of the induced second-order susceptibility is achieved.

The report of Myers et al [1], that a second-order nonlinearity of the order of 1 pm/V can be induced in fused silica by thermal poling has opened up the prospect of linear electro-optic modulators and frequency converters monolithically integrated into optical fibres or planar glass wavegudes. During thermal poling in fused silica, a 1 mm thick sample is heated to $\sim$250-300°C at an applied voltage 3-5 kV. After cooling and removal of the applied field, the second-order nonlinearity is observed only near the anodic surface. This can be explained by the appearance of a high electrostatic field (of order of 10^7 V/cm) in a thin depletion region near the anodic surface [1]. We have suggested that this frozen-in electrostatic field arises between two layers of space charge near the anodic surface: A negatively charged layer depleted with cations, and a positively charged layer created by ionization in the high field between the depleted layer and the anode [2]. But some aspects of this phenomenon, such as substantial lateral spreading of the second-order nonlinearity beyond the boundaries of the positive electrode, are not fully understood. We have suggested that this spreading may be caused by the surface conductivity of glass sample [3]. Whatever its cause, the elimination of this spreading is extremely important for creation of quasi-phase-matched $\chi^{(2)}$ gratings (in glass and probably in ferroelectric crystals such as $LiNbO_3$ or $LiTaO_3$) and, as will be clear from below, for thermal poling of optical fibres [4]. In this Letter a method for elimination of spreading out of poled regions is proposed and experimentally tested. A significant improvement in the reproducibility of thermally poling in silica fibres is achieved.

First of all, we tested the influence of surface conductivity. For this we poled two silica glass samples with different values of surface conductivity. One sample was treated before poling for about 30 min at 75°C in hexamethyldisilazane (HMDS). As a result of this, the silica surface is nearly completely covered with a monolayer of CH_3 - groups, which makes any adsorption of polar H_2O - groups very unlikely (the factor of reduction of surface conductivity is about 10^3). The other sample was not treated. After a standard thermal poling procedure (2 mm thick samples at 4.3 kV applied voltage, anode width ~2.5 mm, 280°C for 15 min), their surfaces were scanned by a focused laser beam for evidence of second harmonic generation in different regions of the samples. A mode-locked and Q-switched Nd:YAG laser operating at 1064nm was used as pump source. No difference in the spreading effect was observed between treated and untreated samples. The width of poled regions was about 1.8 times wider then the width of electrode in the both samples (Figure 1). The result of this test shows that surface conductivity is not responsible for the spreading effect.

But another kind of conductivity exists near the anodic surface, one which may cause the spreading effect. This is conductivity due to electrical breakdown of the air. Simple estimates show that the electric field near the anode will exceed the breakdown field of air (~30 kV/cm), assuming a high ionic conductivity of silica glass at 280°C. In other words, air subjected to high fields near the anode acts as a very good conductor, increasing the real width of the anode. This electrical breakdown problem could of course be eliminated by poling in vacuum or possibly in some other gas or oil with a higher breakdown threshold.

We tested our suggestion experimentally. Thermal poling was carried out in an evacuated chamber at about 1.2×10^{-5} mbar. The temperature of the glass sample was raised using a radiant heater, and the poling parameters were the same as in the poling experiments in air. Second harmonic testing of the vacuum poled samples showed no evidence of spreading out of the poled regions. This proves that the spreading effect is caused by electrical breakdown of the air near the anode.

We have also observed that the elimination of air leads to significant improvements in the thermally poling of optical fibres (Figure 2). In our fibre poling experiments [4] we noticed that the reproducibility of the second-order nonlinearity is rather low - only of about 3 % of poled fibres had a $\chi^{(2)}$ of ~ 0.2 pm/V - most had a nonlinearity an order of magnitude lower. Such poor reproducibility can be explained by electrical breakdown in the air. Indeed, we discovered that the supporting silica substrate (underneath the fibre) was also poled (Figure 2), despite there being no direct electrical contact. To understand this, let us consider a diagram of the fibre poling arrangement, and its corresponding equivalent circuit (Figure 2). R_{d1} and R_1 are the resistances of the depletion region and the remaining fibre, R_{d2} and R_2 the resistances of the depletion region and supporting substrate and R_i is the air resistance.

In the case of electrical breakdown in the air:

$$R_i \ll R_{d1}, R_1, R_{d2}; \qquad R_{d2} \gg R_{d1}, R_1, R_2 \tag{1}$$

leading to:

$$\frac{V_{d1}}{V_{app}} \simeq \frac{R_i}{(R_{d2}+R_2)} \ll 1; \quad \frac{V_{d2}}{V_{app}} \simeq \frac{R_{d2}}{(R_{d2}+R_2)} \simeq 1$$

which shows that only the supporting silica substrate will be poled. In the absence of air breakdown:

$$R_i \gg R_{d1}, R_1, R_{d2}, R_2; \qquad R_{d1} \gg R_{d2}, R_1, R_2 \tag{3}$$

leading to:

$$\frac{V_{d1}}{V_{app}} \simeq \frac{R_{d1}}{(R_1+R_{d1}+R_2+R_{d2})} \simeq 1; \quad \frac{V_{d2}}{V_{app}} \simeq \frac{R_{d2}}{(R_1+R_{d1}+R_2+R_{d2})} \ll 1$$

which shows that the poling will take place only in the fibre.

To test this predictions, we poled fibre in an evacuated chamber. The fibre core was Ge-doped (diameter 16 μm, numerical aperture 0.09, fibre outer diameter 125 μm). The OH concentrations were 80 ppm and 150 ppm respectively in the core and in the cladding (formed from Herasil-1). Regions $\sim$8 mm long were side-polished to within 1 μm of the core using a simple wheel polishing technique. The side-polished fibres were placed on top of 2 mm thick silica substrates, manufactured by the same method as the starting tubes (Herasil-1), and the final assembly was sandwiched between two electrodes with the anode on top of the polished fibre surface (Figure 2). Thermal poling and vacuum parameters were the same as for bulk glass substrates.

Second harmonic tests of the vacuum poled fibres showed almost 100% reproducibility of the value of effective second-order susceptibility $\chi^{(2)}_{eff}$ of about 0.2 pm/V ($\chi^{(2)}_{eff} = \chi^{(2)}\eta$, where η is the overlap factor between fibre mode and the nonlinearity).

In conclusion, the spreading-out effect in poled silica samples is caused by electrical breakdown of the air. It is effectively eliminated by poling in vacuum. Significant improvements in the thermally poling of optical fibres are also achieved if vacuum poling is used.

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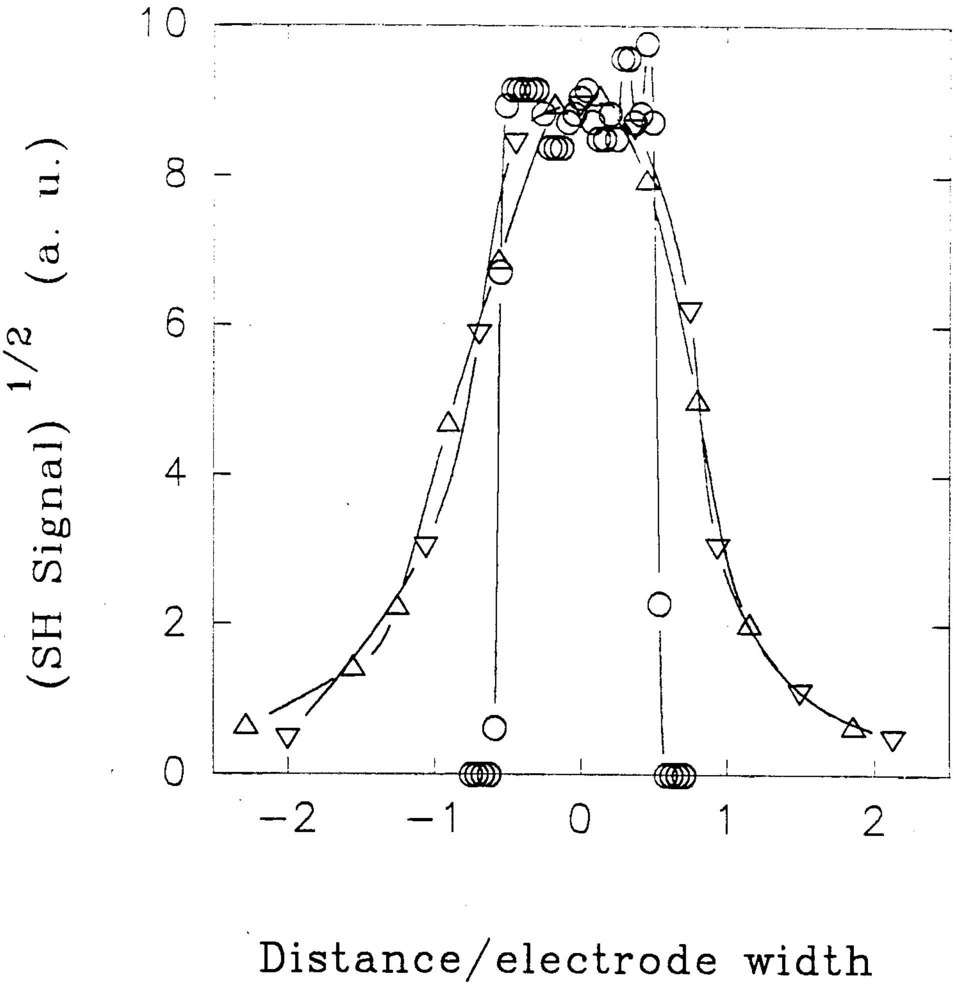
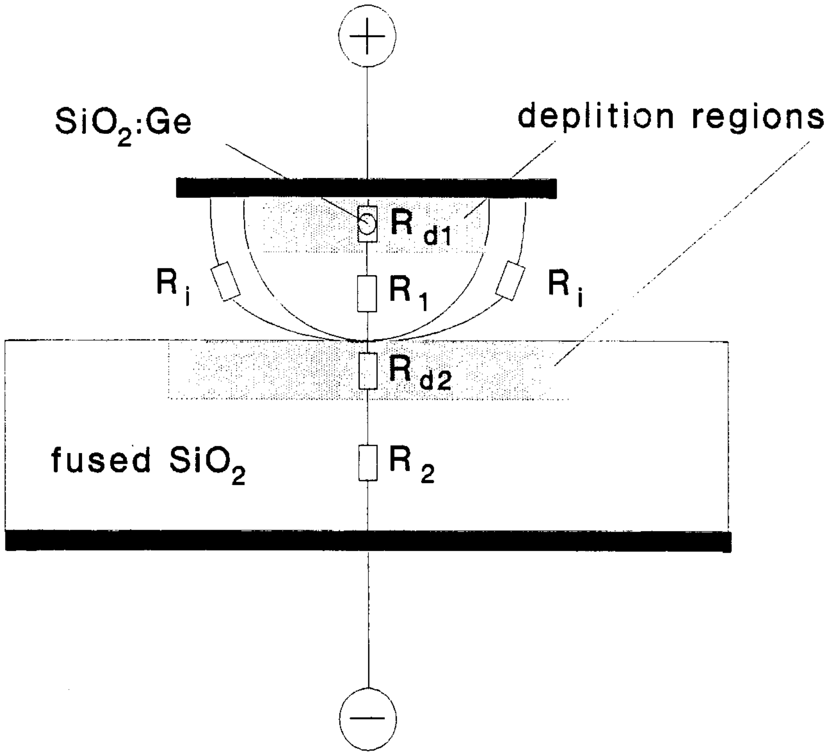
FIGURE CAPTIONS

1. Square root of the second harmonic signal versus the distance from the centre of the positive electrode for HMDS treated silica sample (▽), control sample without any treatment (△) and vacuum poled silica sample (○).

2. Diagram of the fibre thermal poling arrangement and corresponding equivalent circuit.


10
8
6
4
2
0
−2
−1
0
1
2
(SH Signal) $^{1/2}$ (a. u.)
Distance/electrode width


SiO$_2$:Ge
deplition regions
R$_{d1}$
R$_i$
R$_1$
R$_i$
R$_{d2}$
fused SiO$_2$
R$_2$
+
−