Robustness of femtosecond direct written structures in silica glass

Erica Bricchi, Weijia Yang and Peter G. Kazansky

Optoelectronics Research Centre, University of Southampton

Abstract: We demonstrate the extraordinary stability of femtosecond directly written structures to temperature increase. A temperature close to the glass transition is required to degrade birefringence properties and the related nanostructures.

In recent years femtosecond lasers have proved to be a successful tool for micromachining optical transparent materials [1]. The ability of processing the material in one single step as opposed to traditional lithography makes direct writing an alluring fabrication technique. Moreover, since the physical mechanism inducing an index change in the irradiated material is based on nonlinear absorption taking place within the focus of a converging beam, requirements on photosensitivity are greatly reduced, hence allowing complex structures to be directly written in materials traditionally challenging for direct laser processing. Furthermore depending on the laser intensity and the material utilized, different features with either positive or negative index change or voids can be realized thus targeting numerous applications such as waveguides [1], data storage [2], and diffraction optics [3].

In wide-bandgap materials it has been shown that below a certain intensity threshold an isotropic positive index change as high as $3\,10^{-3}$ can be induced, whereas above this threshold the femtosecond written structures are characterized by anisotropic reflection [4], strong birefringence and an average negative index change [5] due to the formation a self-assembled nanostructures [6]. Our observation suggests that free electrons are produced within the focus of a high-power infrared ultrashort pulsed beam. Over a certain intensity threshold the interference between the plasma of electrons and the laser light produces nano-sized gratings with a pitch as small as 150 nm [6]. These structures consist of oxygen deficiency regions (-20 % as compared to pure silica) [6] with a local index change as high as -0.4 [5], making them the strongest laser written nanogratings ever observed.

The results reported here, after investigating the modification with the temperature of the average index change and the birefringence of femtosecond direct written structures proved their extraordinary stability with temperature, furthermore confirming that the birefringence arises from a structural change.

An amplified, mode-locked Ti:Sapphire laser operating at a wavelength of 800 nm, with 170 fs pulse duration and 100 kHz repetition rate, was utilized to fabricate the structures to be tested. The laser light was focused via a 10x (NA = 0.21) objective to a focal spot of ~ 4 μm into a fused silica sample (Herasil) which was mounted upon a computer controlled linear motor translation stage. Five regions of 100x100 μm^2 were scanned at a speed of 80 μm/s within the bulk of silica glass. Each zone was fabricated by writing 100 adjacent lines with a spacing of 1 μm between them and with different average power (P1=214 mW, P2=160 mW, P3=120 mW, P4=80 mW and P5=40 mW).

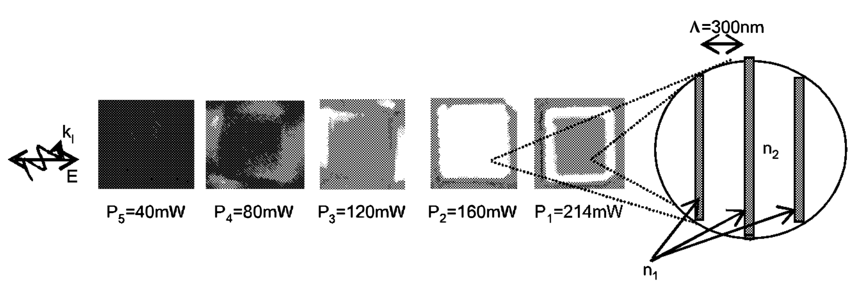

Fig. 1. Color image of the directly written uniform regions embedded in silica. The image was taken with the sample positioned between cross polarizers. The schematic of the self-organized nanostructure formed in the birefringent regions is shown.

Figure 1 shows an image of the sample acquired between cross-polarizers. Both optical inspection, and phase measurements (Fig. 2) confirm that the birefringence onsets at average power of 120 mW (energy per pulse was 1.2 μJ).

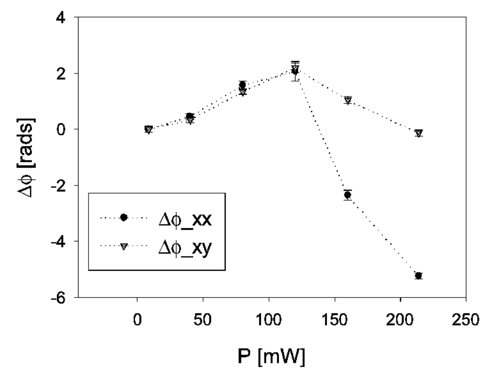

Fig.2. Dependence of the laser induced phase shift for two orthogonal polarizations (xx and xy) versus the average power.

The phase measurements represent the average difference of phase $\Delta\phi$ between the light traveling into the irradiated structures and into pure silica and have been obtained using an interferometric phase-stepping technique [5]. All the measurements were carried out for the two polarizations of the interrogating light parallel to the axes of birefringence; one, *xx*, parallel to the polarization of the light during writing, the other on the perpendicular direction (*xy*). The phase measurements relate to the local indices change according to the following equations:

$$\Delta\phi_{xx} = \frac{2\pi}{\lambda} t \left[\sqrt{\frac{n_1^2 n_2^2}{f n_2^2 + (1-f) n_1^2}} - n_0 \right] \quad (1),$$

$$\Delta\phi_{xy} = \frac{2\pi}{\lambda} t \left[\sqrt{f n_1^2 + (1-f) n_2^2} - n_0 \right]$$

where t is the thickness of the quasi-uniform regions (i.e. the dimension along the direction of the propagation of writing light), λ is the wavelength of the light traveling in the interferometer, n_0 is the index of refraction of silica, n_1 and n_2 are the local indices of refraction of the nanostructure (see inset of Figure 1) and f is the filling factor defined as $f=t_1/\Lambda$, with t_1 width of the regions of local index of refraction n_1 and Λ the period of the nanostructure. For non birefringent structure $n_1=n_2$ and the equations (1) simplify to the well known relation between phase shift and index change.

In order to study the stability of the femtosecond structures with the temperature, the sample was heated at rate of 3°C per minute, kept at 200 °C for one hour and cooled to room temperature at 1 °C per minute. The treatment was then repeated at 500 °C, 800 °C, 1100 °C and 1400 °C and after each annealing step the phase change was measured except for the last case, due to crystallization of the sample.

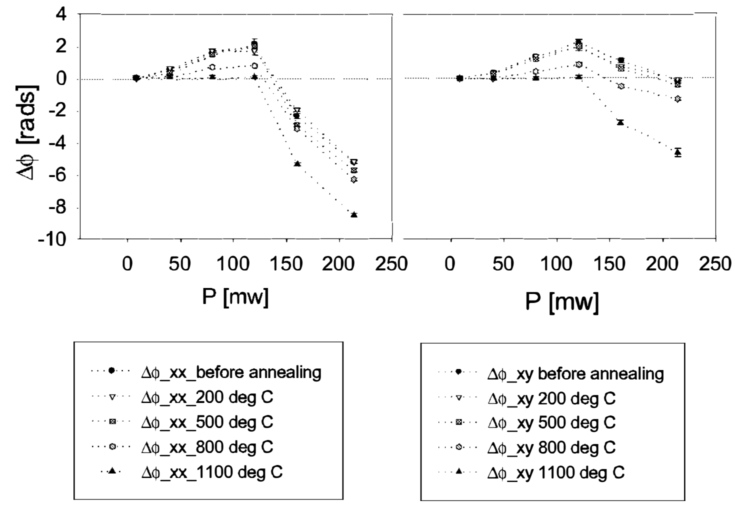

Fig.3. Measurement of the phase shift of the femtosecond direct written uniform regions versus writing power before and after annealing at 200°C, 500°C, 800°C and 1100°C.

The result after annealing show that the birefringent (P_4, and P_5) and the non birefringent structures (P_1, P_2 and P_3) have a different behavior with temperature (Fig. 3).

All the structures are unaltered up to a temperature of 500° C. The index change of the isotropic non birefringent structures decreased of ~60 % after heating at 800° C, and finally disappeared after the annealing at 1100° C. For the birefringent structures, based on the experimental measurements, we estimated a maximum change of the local indices of refraction of the nanostructures of about 32 %. All the birefringent regions were still clearly visible under an optical microscope after annealing at 1100° C.

In conclusion we reported the first experiment related to the annealing of femtosecond directly written structures which showed extraordinary stability with the temperature. Non birefringent regions started degrading at 800° C; whereas the birefringent couldn't be annealed confirming that structural changes are involved in the nanostructures formation.

References:

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