Extraordinary stability of femtosecond direct written structures

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Abstract: We report novel results on the stability of femtosecond direct written structures: a silica sample previously irradiated with ultrashort pulses was annealed at increasing temperatures till 1400° C where it crystallized. Our results show that the birefringent direct written structures are stable till a temperature close to the glass transition of silica. After annealing at a temperature as high as 1100° C the form birefringence of the structures is still present, proving that the nature of the laser induced anisotropy is related to a structural change.

The use of lasers to directly pattern optoelectronic devices primarily utilizes direct irradiation by UV light. Nevertheless in recent years femtosecond lasers have proved to be an interesting alternative route for micromachining within the bulk of transparent materials [1].

Using nonlinear absorption taking place within the focus of a converging beam, thus not requiring photosensitivity, complex structures can be directly written [2].

In wide-badgap materials our observations suggest free electrons are produced within the focus of a high-power infrared ultrashort pulsed beam. The interaction between this plasma of electrons and the laser produces micron-sized gratings of a 150nm and 300nm pitch which are the result of the interference between electron plasma waves and the laser field [3]. It has been shown that the lines forming these periodical structures have an oxygen deficiency of 20% as compared to pure silica [3] and a local index change as high as -0.4 [4], making them the strongest laser written nanogratings ever observed.

The arising of this self-organized nanostructure, whose period is smaller than the wavelength of light, causes the direct written structures to be birefringent [4]. This explanation, supported by experimental and theoretical results [3,4] can also justify the observation of anisotropic reflection that we previously published [5].

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 tunable between 800nm and 850nm, with 150fs pulse duration and 100kHz repetition rate, was utilized to fabricate the samples. The laser light was focused via either a 10x (NA=0.21) or 50x (NA=0.55) objective to a focal spot of ~4μm or ~1.5μm respectively into a fused silica sample (Herasil) which was mounted upon a computer controlled linear motor translation stage. Using this setup with the laser tuned at 800nm and focused via the 50x objective, an array of 5 quasi uniform regions of 100x100 μm each was directly-written within the bulk of silica glass (sample A). Each processed zone was realized by writing 100 adjacent lines with a spacing of 1μm between them and with different average power (P1a=268mW, P2a=200mW, P3a=150mW, P4a=100mW and P5a=50mW). A second sample was realized with the same procedure, but focusing the light through the 10x objective. In this case (sample B) the average powers used were P1b=214mW, P2b=160mW, P3b=120mW, P4b=80mW and P5b=40mW. Both samples were written scanning the laser at a speed of 80μm/s (the number of pulses on sample A was 5000, 1900 for sample B).

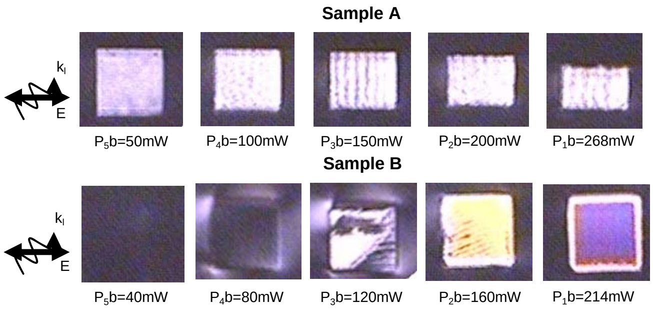

Fig.1. Picture of the structures of sample A and B taken between cross polarizers.

Figure 1 shows an image of the two samples acquired between cross-polarizers. While sample A (written in a tighter focusing regime with the 50x objective) is birefringent for any of the power levels used, in sample B birefringence can be clearly observed above a threshold of average power of 120 mW (energy per pulse 1.2 μJ) indicating that the phenomenon is fluence-dependent. Following a definition previously used, the non birefringent structures (the first two structures of sample B) will be referred type 1 and the birefringent ones (sample A and the last two structures of sample B) as type 2.

We have previously proved that the form birefrincence is given by the arising of a self-organized nanograting, which can result in a negative average index change [4]. In order to enable the measurement of the refractive index change of each zone with respect to the unprocessed bulk, an interferometric phase-stepping technique was utilized (more experimental details on the interferometric set-up can be found in [4]). The results of those measurements represent the difference of phase $\Delta\phi$ between the light traveling into the irradiated structures and into pure silica, which is related to the average index change through the following equation:

$$\Delta n = [\lambda/(t2\pi)]\Delta\phi, \qquad (1)$$

where Δn is the average index change, t the thickness of the structure, and λ the wavelength of light.
The samples were cut in order to measure the thickness t (Fig.2) of each structure (this explains the irregular shape of some of the structures in Fig. 1) and the index change measurements were carried out for the two polarizations of the interrogating light lying along the axes of birefringence; one, conventionally called *xx*, parallel to the polarization of the field which was used to write the structures, the other perpendicular to it (*xy*). The results are shown in Fig.3.

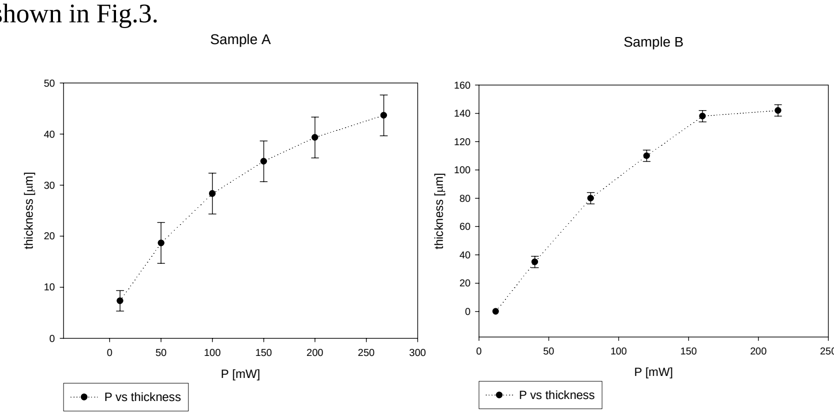

Fig.2. Experimental measurements of the thickness of the samples A and B.

As expected from the theoretical model developed in [4], all the birefringent regions showed a negative index change respect to the unprocessed silica, whereas for intensity below the threshold of birefringence, the regions were characterized by a positive index change. Both samples were then annealed to study how femtosecond written structures behaved with temperature and to verify if there was any difference between structures of type 1 and 2.

The samples were 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 experiment was then repeated at 500°C, 800°C, 1100°C and 1400°C and after each annealing process the index change was measured except for the last case, being sample crystallized.

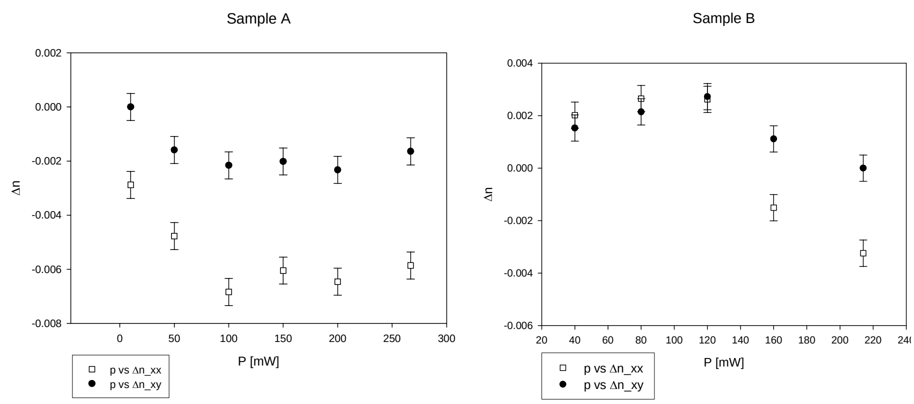

Fig.3. Measurement of the average index changes of the femtosecond direct written uniform regions of sample A (left) and sample B (right) along the axes of birefringence xx and xy.

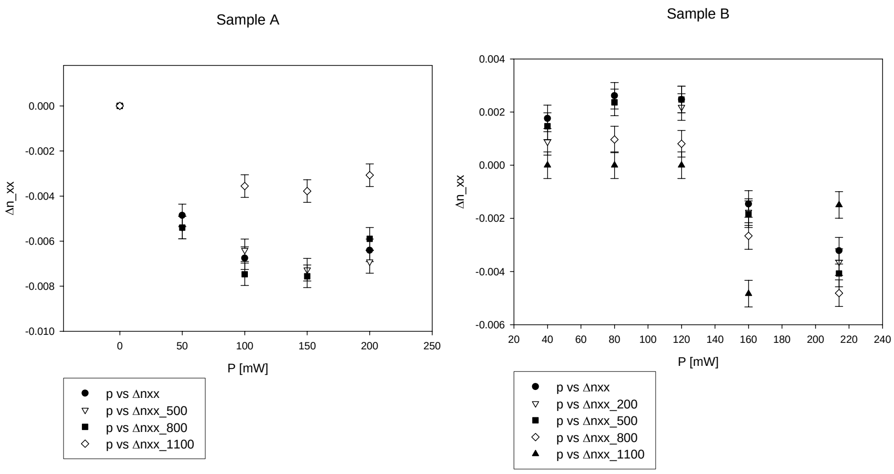

Fig.4. Measurement of the average index changes of the femtosecond direct written uniform regions of sample A (left) and sample B (right) along the strongest birefringent axis before and after annealing at 200°C, 500°C, 800°C and 1100°C.

The results of the annealing experiment presented in Fig. 4 show that the birefringent structures (all the points of sample A and the last two points in power on the graph of sample B) and the non birefringent ones have a different behavior with temperature.

The index change of the structures of type 1 decreased of ~70% after heating at 800°C and disappeared after the annealing process at 1100°C (first three points in the picture of the right of Fig.4). The index change of the regions belonging to the type 2 was instead within the error bars of the measurement done before the annealing up to a temperature of 800°C, and started being affected only at 1100°C. In particular, after the annealing process at 1100°C, the index change of sample A reduced of ~47%; the last two structures of sample B instead did not seem to follow this trend. This anomaly could be explained considering that 1100°C is around the glass transition temperature of pure silica when the glass starts its transition towards liquid state and becomes unstable.

Nevertheless all the birefringent regions were visible under an optical microscope and they were still birefringent (Fig.5 shows one of the region after the annealing process at 1100°C as reference).

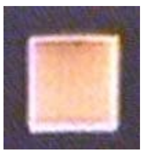

Fig. 5 .Picture of one structure of sample B (written at P1b=214 mW) taken between cross polarizers after the annealing process at 1100°C.

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 disappeared only at a temperature of 1100°C; the birefringent ones couldn't be annealed and kept their anisotropy at a temperature as high as 1100°C which is a result in good agreement with the measurement of highest index change ever reported for a laser induced nanograting.

1. K. Miura, J. Qiu, H. Inouye, T. Mitsuyu, and K. Hirao. "Photowritten optical waveguides in various glasses with ultashort pulse laser" Appl. Phys. Lett. **71**, 3329-3331 (1997).
2. E. Bricchi, J. D. Mills, P. G. Kazansky, B. G. Klappauf and J. J. Baumberg. "Birefringent Fresnel Zone Plates in Silica by Femtosecond Laser Machining''Opt. Lett. **27**, 2200-2202 (2002).
3. Y. Shimotsuma, P. G. Kazansky, J. Qiu and K. Hirao. "Self-organized nanogratings in glass irradiated by ultrashort light pulses" Phys. Rev. Lett. **24**, 2474051-2474054 (2003).
4. E. Bricchi, B. G. Klappauf and P. G. Kazansky. "Form birefringence and negative index change created by femtosecond direct writing in transparent material''Opt. Lett. **29**, 119-121 (2004).
5. J. D. Mills, P. G. Kazansky, E. Bricchi and J. J. Baumberg. "Embedded anisotropic microreflectors by femtosecond-laser nanomachining''Appl. Phys. Lett. **81**, 196-198 (2002).