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Characterization of Nanoscale Structures Observed in Femtosecond Laser Micromachining

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We present a quantitative analysis of self-arranged sub-wavelength structures created by femtosecond laser micromachining in silica. From the spatial correlation we calculate the periods of the structures versus laser energy and wavelength, and translation speed.

Thanks to its unique properties, the femtosecond laser direct writing technique offers the potential for realizing three-dimensional photonic devices fabricated in a single process and in a variety of transparent materials. Moreover, depending on the laser intensity and the sample material, 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]. It has been shown that structures written in silica glass above a certain intensity threshold exhibit nano-sized periodic substructures within the irradiated volume [4], ensuing in anisotropic reflection [5], strong birefringence and an average negative index change [6]. The inspection of these sub-wavelength structures revealed the presence of distinct periodicities whose values and orientations strongly suggest that they originate from optical/plasma wave interference [7]. Embedded microreflectors and retardation plates based on these nanogratings have been reported [5,6], however their optimization requires a systematic characterization of the parameters of the periodic structures versus the laser parameters. Recently, the changes in the nanogratings due to the variation of the pulse duration have been qualitatively investigated [8].

Here we report a quantitative study of the dependence of the periodic structures on the laser energy, wavelength, and the writing speed. In addition, we measure the modification of the grating periodicity along the length of the structures.

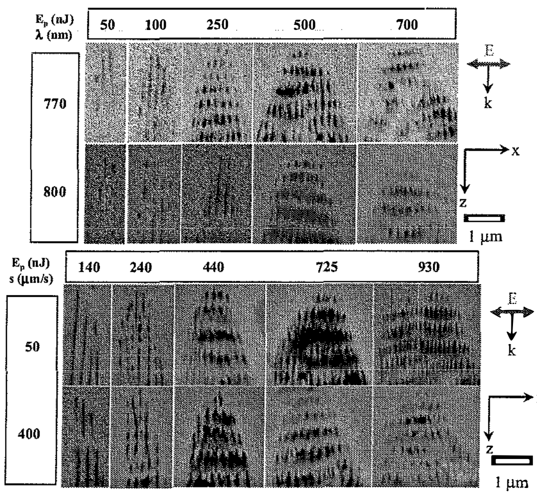

Fig. 1. SEM images of nanogratings formed by femtosecond writing in silica glass.
k: laser wave vector, E: electric field, E_p: pulse energy, and s: writing speed.

In our experiment the radiation from a regeneratively amplified mode-locked Ti: Sapphire laser (150 fs pulse duration, 250 kHz repetition rate), operating at a wavelength tunable between 750 nm and 850 nm, was focused via 50x (NA=0.55) microscope objective into silica samples (Herasil 1), which were mounted on a 3D linear-motor translation stage. The speed of writing were ranged from 50 µm/s to 500 µm/s and the pulse energies were 0.1 - 0.9 µJ. A series of lines was written in the samples about 200 µm below the surface, while the samples were translated along the y direction, perpendicular to the beam (z). The laser was linearly polarized in x, the direction perpendicular to the translation of the samples. After irradiation each sample was polished down to the structures on the plane xz perpendicular to the translation direction and subsequently analyzed with an SEM in backscattering mode. The resulting images are shown for a wide range of writing parameters in Figure 1.

Confirming previous observations, Figure 2 shows that besides the main period (Λ) in the direction of the polarization of the laser (x), a transverse periodicity ($\Lambda_\perp$) is observed in the direction of the laser propagation. Utilizing an algorithm based on the evaluation of the correlation coefficients between different points in the image versus their distance in one direction (x or z), we calculated the average value of the periods from the first maximum of the correlation functions (Fig.2).

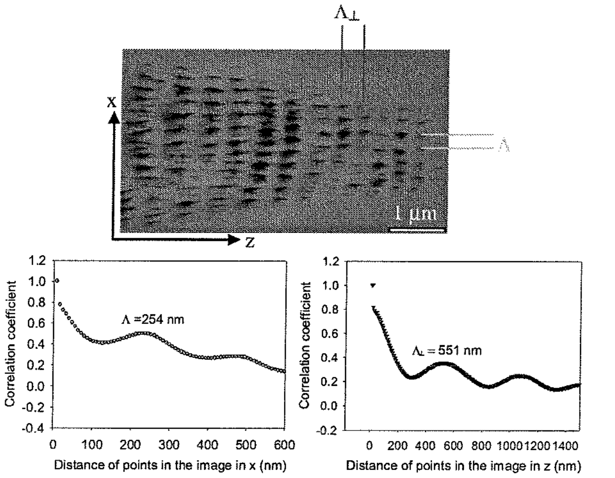

Fig. 2. SEM image of part of the crossection of a written line (λ=800 nm, s=100 µm/s, E_p=0.5 µJ) and the corresponding correlation coefficients calculated along x and z.

Our preliminary analysis of the numerous acquired images reveals that, over the considered ranges, the translation speed does not affect the periodicity Λ, while increasing the laser energy by almost a factor 10, Λ decreases by ~20%. The preliminary results on the wavelength dependence suggest that shorter wavelengths also yield significantly smaller structures (Fig. 3 (a)). Finally we show that the transverse periodicity $\Lambda_\perp$ increases along the direction of the beam propagation (Fig. 3 (b)). These experimental results are interpreted in terms of the theoretical model based on optical/plasma wave interference.

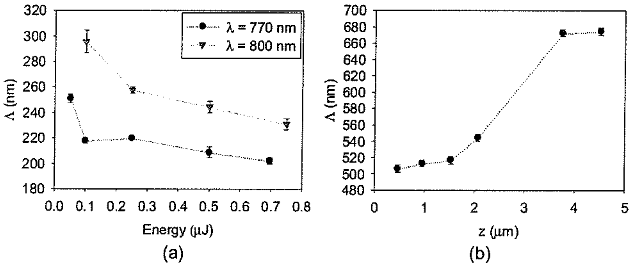

Fig.3. (a) Main period (Λ) versus energy calculated for two different values of laser wavelength. Writing speed s=100 µm/s (b) Variability of the secondary period $\Lambda_\perp$ versus the laser propagation direction (z). Writing parameters: λ=800 nm, s=400 µm/s, E_p=0.9 µJ.

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