# UV femtosecond laser inscribes a 300-nm-period nanostructure in a pure fused silica

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**Abstract**

We report on the first recording of a ~150-nm-pitch periodical structure in a permanently moving sample of a pure fused silica using the tightly-focused, 82 nJ, 267 nm, 300 fs, 1 kHz laser pulses.

**Keywords:** femtosecond UV laser, microfabrication, fused silica, nanostructure, DIC microscopy

## 1. Introduction

Very recently, using tightly-focused femtosecond near-IR pulses, periodical sub-micron structures have been recorded [1–3]. Such microfabrication utilizes the multi-photon approach [4], which allows the inscription inside various non-photosensitive optical materials. The combination of multi-photon excitation with the point-by-point technique [5] offers the great potential of creating non-uniform chirped gratings by controlling the rate of femtosecond pulses or the sample translation speed.

It is well known that the propagation of a femtosecond pulse inside a bulk dielectric (e.g. fused silica glass) with a peak power exceeding the threshold (critical power) results in its self-focusing. Such a regime is characterized by essential reduction of the spatial dimensions of the inscribed modifications below the diffraction limit [6]. To date, the minimal period of nanostructure achieved by the use of a femtosecond near-IR beam is 535 nm [2, 3]. Shifting the wavelength of the inscribing Ti:sapphire laser radiation to the UV range (e.g. to 267 nm by third-

harmonic generation [7]) allows for overcoming this limitation and recording structures with even smaller periods. Such a development is very important, for example, for the point-by-point fabrication of first-order Bragg gratings at the Yb-doped fibre laser wavelength. Though femtosecond laser pulses at 264 (or 267) nm wavelength were widely used before for long-period and Bragg grating inscription in standard telecom [8, 9, 10] and pure-fused-silica photonic crystal [11, 12] fibres, this approach has not previously been employed for microfabrication purposes.

In this paper, the application of UV femtosecond pulses to structure fabrication in a pure fused silica is described for the first time. Use of a 267 nm femtosecond light source has led to decrease of the achievable nanostructure period down to 300 nm which, to the best of our knowledge, has never been reported previously.

## 2. Experimental set-up

Femtosecond pulses at 800 nm were produced by a Ti:sapphire chirped pulse amplification laser system consisting of „Tsunami“ oscillator and „Spitfire“ amplifier (both from Spectra-Physics). This laser system delivered 0.8 mJ pulses with 150 fs duration and 1 kHz repetition rate. The IR beam diameter after amplifier was 2.5 mm at FWHM. The set-up for third-harmonic generation (THG) was similar to that described earlier [13]. The UV pulse energy was monitored by the PD10 photodiode head (Ophir Optronics). The measured energy of third harmonic pulses reached 120 μJ with pump energy at 800 nm of 760 μJ. By varying the optical delay and measuring the cross-correlation function, we estimated the 267 nm pulse width to be about 300 fs.

Fused silica samples of 50×20 mm size and 1 mm thickness (Schott Glas) were used in the experiments. They were moved in the horizontal plane in two perpendicular directions by the air-bearing translation stage ABL-1000 (Aerotech). The translation speed was varied in the range of

0.1–1.0 mm/s. The absolute and relative micropositioning accuracies were both better than 50 nm.

The UV beam was directed in a strictly perpendicular direction onto the surface of the fused silica sample from the top. The UV laser beam could be focused to any selected depth below the surface with accuracy of 1 μm. The focusing depth inside the sample was varied from 0 to 300 μm. For the inscription process, we utilised both π and σ polarizations of the 267 nm beam (parallel and perpendicular to the translation direction, respectively). For focusing we used a reflective micro-objective with numerical aperture of 0.65 (Ealing), manipulated by a 3D-micropositioning manual translation stage 17 MAX 303 (Melles Griot). The inscription energy values were varied between 80 and 3400 nJ. To estimate the beam-waist diameter in focus, we can use the well-known expression for diffraction-limited focusing $w_0 = 1.22\lambda / NA$, where λ is the inscription wavelength and NA is the numerical aperture of the micro-objective. Substituting λ = 267 nm and NA = 0.65 gives the beam-waist diameter $w_0$ of 500 nm and corresponding lower limit for the irradiation intensity in focus of around $(1–3) \times 10^{14}$ W/cm$^2$.

For annealing of the recorded structures the CWF11/23 oven (Carbolite) was used.

## 3. **Characterisation of inscribed structures**

Characterisation of the irradiated samples was performed using an optical microscope, Axioscope-2 MOT plus (Zeiss), which was equipped for both transmitted light and differential interference contrast (DIC) measurements. The resolution of a conventional optical microscope is considered to be of the order of the illumination wavelength: in our experiment, even with the use of a blue filter it was rather difficult to distinguish the 600-nm-period perturbations induced in the bulk of the material. However, the use of DIC technique enabled us to monitor structures with periods down to 300 nm. In the experiments, the combination of a Plan-Apochromat oil immersed objective (×100/1.40/DIC) and an Achromatic-Aplanatic condenser (1.4H/PH/DIC) with numerical aperture of 0.6 (or higher) was applied. The DIC-prism (III/1.4) and DIC-slider

(×100/1.40III) were also used; such combination seems to be the best one commercially available from Zeiss. The refractive index variation is expected to be of the order of $10^{-4}$, which corresponds to a few times more than the sensitivity level of this DIC microscope, based on the comparison experiments conducted with the known samples.

**4. Results and discussion**

We started our inscription experiments with the optimization of the focal position below the sample surface. For a pulse energy of 3400 nJ and translation speed of 1.0 mm/s, inscription was possible only at the depth of 180 μm. For 1380 nJ pulses, inscription at the same speed started only at the depth of 225 μm. At the depth of 300 μm, inscription was possible with much lower pulse energies (down to 82 nJ) and at different translation speed values (down to 0.3 mm/s).

Figures 1 (a) and (b) demonstrate the periodic structures obtained inside the fused silica sample at the 300 μm depth using 82 nJ, 267 nm, 300 fs, 1 kHz pulses with π and σ polarizations, respectively. From these pictures it is clearly seen that, at the translation speed of 1 mm/s, the length of 10 μm bar corresponds *exactly* to 10 periods of the recorded nanostructure; therefore, this structure possesses a period of 1000 nm. As our repetition rate is of 1000 pps, each circular spot on the 1 mm/s microphotographs is inscribed by a *single laser pulse*. The decrease of the sample translation speed from 1 mm/s to 0.6, 0.5 and 0.4 mm/s allowed us to create nanostructures with periods of 600, 500 and 400 nm [for both polarizations, Figs. 1 (a), (b)]. Finally, for the σ polarization of the inscribing beam and a sample translation speed of 0.3 mm/s [Fig. 1 (b)], we managed to record a structure, which contains exactly 33.3 periods on the 10 μm length. This corresponds to the period as short as 300 nm, which, to the best of our knowledge, has never been observed before.

It is important to note that DIC microscopy does not present the real image; rather, the resulting picture contains the information of both the intensity distribution and the derivative of the optical phase between two orthogonally polarised beams, spatially separated by a distance

*smaller* than the resolution of the ×100, NA = 1.4 microscope objective. Thus, periods of about few hundred nanometres can be detected.

The minimal pulse energy value of 82 nJ used for the inscription can be advantageously compared with the typical pulse energy value of 600 nJ used earlier in 800 nm nanostructure fabrication [2, 3]. Such a decrease in the inscription energy is in line with the decrease of the order of the absorption process, from five-photon to two-photon [4]. It should be emphasized that the laser pulse peak power values used in our experiments (270–670 kW) were well above the critical power value for self-focusing, which at 267 nm is estimated to be ~150 kW (compared with 2300 kW at 800 nm [3]). The importance of the 300-μm-long glass layer for the substantial decrease of the focal beam spot could be related to nonlinear effects and/or spherical aberration compensation provided by the design of the microscope objective.

Annealing at 850 °C for 26 hours did not reveal any visible degradation of the recorded structures, supporting the involvement of irreversible damage in our inscription process.

In conclusion, using tightly-focused UV femtosecond laser pulses, we have succeeded in recording a 300-nm-period nanostructure inside a pure fused silica sample. In order to fabricate a first-order Bragg grating in Yb-doped fibre, one should be able to inscribe a ~360-nm-period structure inside the fibre core, and our work provides a first demonstration of this possibility with important application in the fibre laser area.

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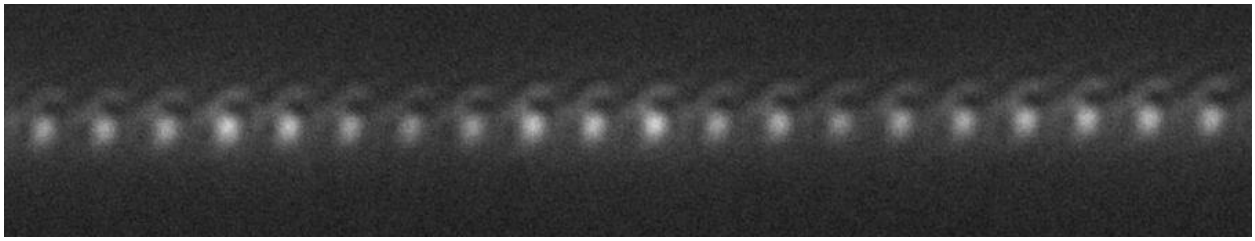
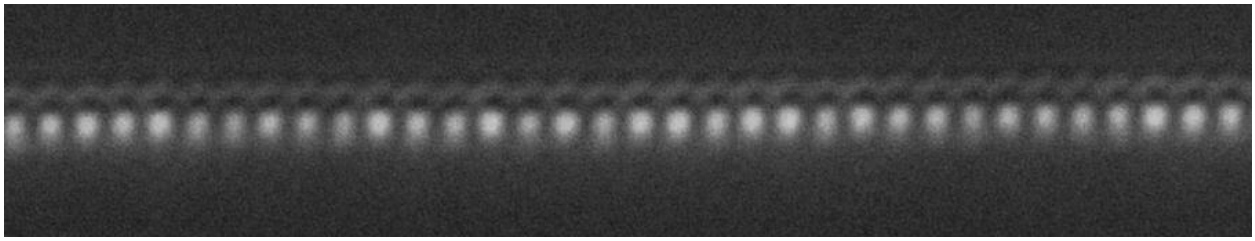
**Figures captions.**

Fig. 1. Microphotographs of the structures inscribed at different sample translation speeds. The energy of the inscribing pulses was 82 nJ, with polarization either (a) parallel to the translation direction ($\pi$ polarization) or (b) perpendicular to the translation direction ($\sigma$ polarization). The size of the bars corresponds to 10 $\mu$m.

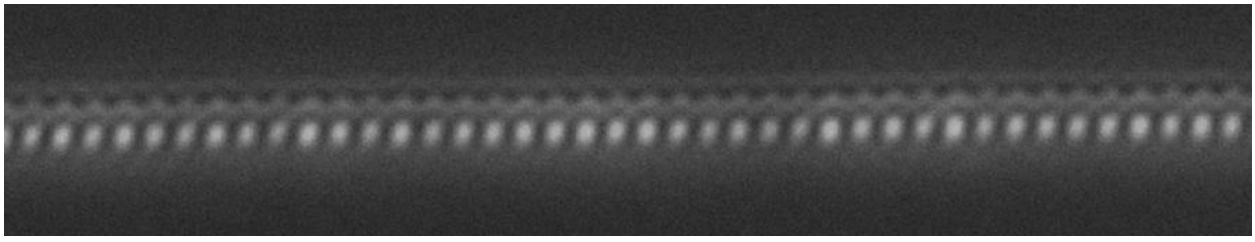
**1.0 mm/s**



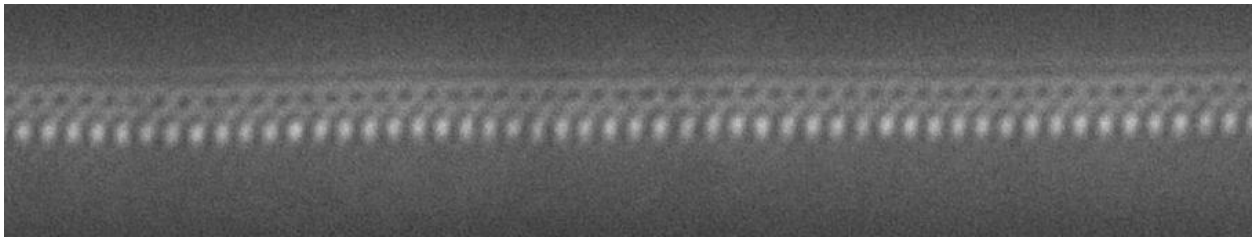
**0.6 mm/s**



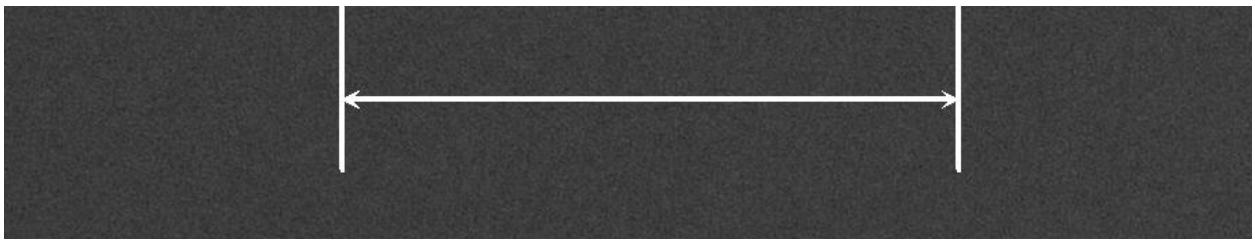
**0.5 mm/s**



**0.4 mm/s**

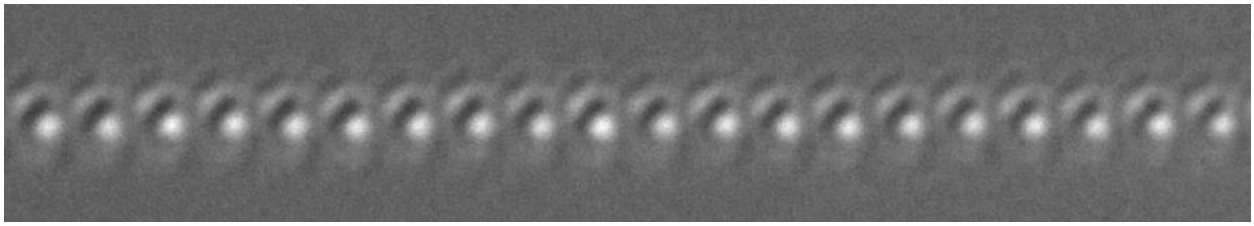


**10 mm**

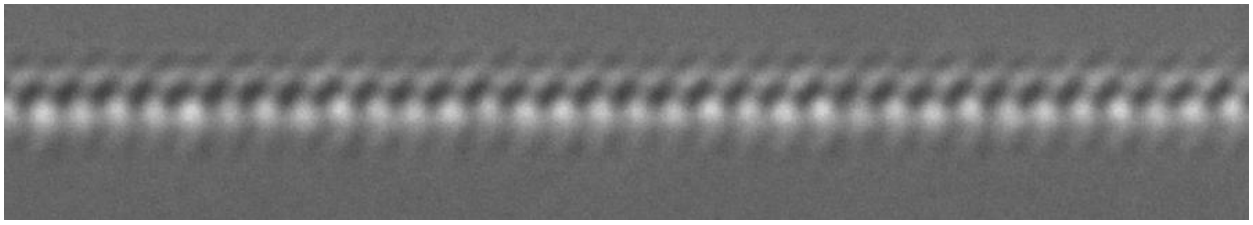


**(a)**

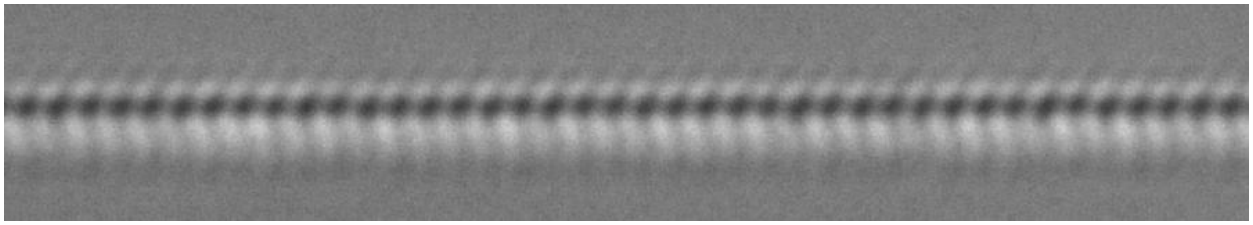
**1.0 mm/s**



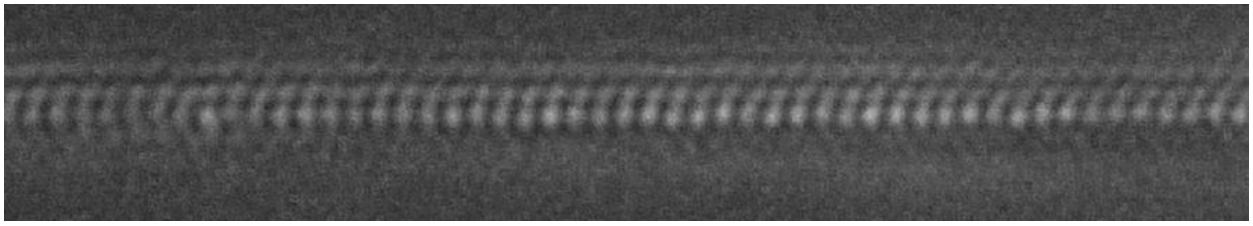
**0.6 mm/s**



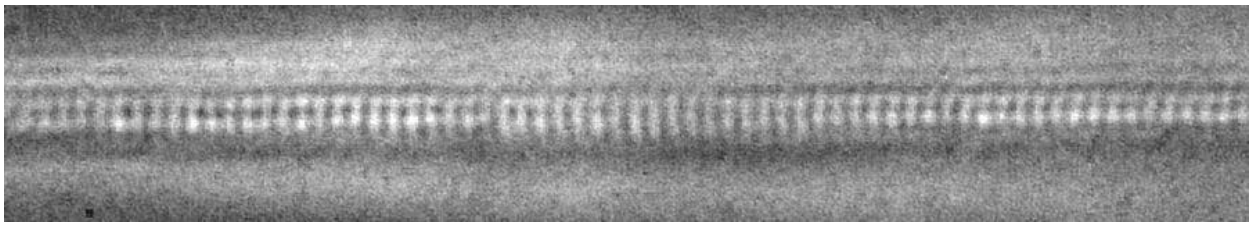
**0.5 mm/s**



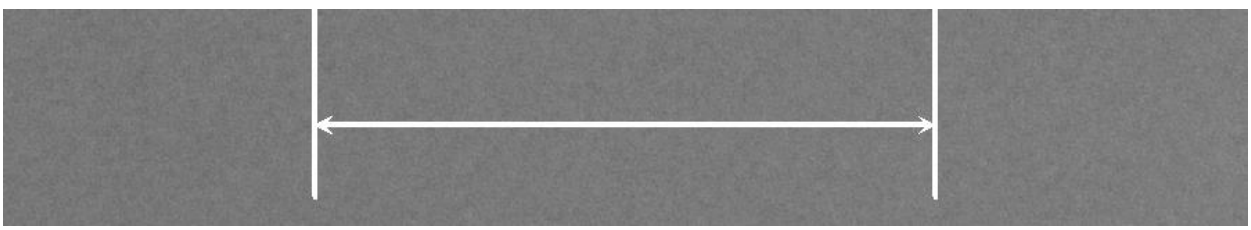
**0.4 mm/s**



**0.3 mm/s**



**10 mm**



**(b)**