

# Fabrication of 150 nm period grating in fused silica by two-beam interferometric laser induced backside wet etching method

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**Abstract:** Fused silica gratings with periods of 154 nm, 266 nm, and 550 nm have been fabricated by the method of two-beam interferometric laser induced backside wet etching (TWIN-LIBWE). The spatially filtered pulses at 266nm were splitted into two parts and interfered at an incident angle of 60°, 30°, and 14°, respectively, on the backside surface of a fused silica plate contacting with the liquid absorber. The morphology of the etched gratings was characterized by atomic force microscope. According to our knowledge, the produced 154 nm period is the smallest grating constant generated by laser techniques directly in fused silica at present.

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**OCIS codes:** (220.4000) Microstructure fabrication; (230.1950) Diffraction gratings; (350.3390) Laser materials processing

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## 1. Introduction

The laser induced backside wet etching (LIBWE) technique [1] is an intensively studied, promising procedure for micro- and submicromachining of transparent materials. Most UV transparent materials (e.g. fused silica, quartz crystal, pyrex, CaF<sub>2</sub>, BaF<sub>2</sub>, sapphire and other glass types [1-4]) can be processed by this method. LIBWE exhibits several important advantages, which make it more beneficial than the direct laser machining methods, such as low etching threshold fluence, fine etch depth control, debris-free etching, and the relatively large etched area.

The procedure of LIBWE can be briefly described as follows. One side of a transparent target is in contact with a liquid absorber having high absorption coefficient at the wavelength of the irradiating laser. This liquid is illuminated through the transparent material by the laser beam. The contacting liquid layer absorbs the pulse energy, which results in extreme high temperature within a thickness corresponding to the light penetration depth. Due to heat conduction the contacting surface layer of the solid sample is softening or even melts, so that the mechanical removal of this layer becomes very easy [5]. The quickly expanding microjets and high pressure vapor bubbles forming in the hot liquid layer start removing material from the softened target surface [6-9]. Moreover, probably the possible chemical modification of the thin contacting layer of the transparent material (carbon contamination from the liquid) [10] also play an important role in the etching mechanism with the ablation-like material removal.

One of the most important questions from applications' point of view is how small grating period can be produced by this technique into transparent targets. There are only a few studies which reported gratings with micrometer size period produced by LIBWE using mask projection technique [9, 11, 12]. So far, the smallest LIBWE generated grating period was 750 nm [9]. Into fused silica J. Ihlemann et al. produced 830 nm gratings by direct ablation of a mask with an F<sub>2</sub> laser [13]. The resolution of the projection method, which was used for fabrication of fused silica gratings so far, is ultimately limited by the resolution of the imaging system and the laser damage threshold of the mask.

In this paper a new technique, the *two beam interferometric laser induced backside wet etching* (TWIN-LIBWE) is introduced, which is basically a combination of the traditional LIBWE with the two-beam interference method. Then we show that this mask-free procedure makes possible the fabrication of fused silica grating with a period substantially smaller than that previously reported [9]. The shortest achieved grating period in fused silica is 154 nm, which is, according to our knowledge, the smallest grating constant generated by laser etching in fused silica at present.

## 2. Experiments and results

A Q-switched, frequency-doubled Nd:YAG laser ( $\tau_{FWHM}=10$  ns, repetition rate: 10 Hz) was used for implementation of the traditional two-beam interference method. The coherence length of the laser was measured to be 1 cm. In order to produce as uniform structure as possible, the intensity profile of the laser beam was smoothed in two steps. First the second harmonic beam at 532 nm was spatially filtered along with demagnifying the beam size to 4 mm. Then, following an external fourth harmonic generation stage consisted of a CLBO crystal, the ultimate supergaussian-like spatial profile of the 266 nm pulses were formed in a second spatial filter just prior to the beam splitter, while the beam size was further reduced by a factor of two. The *p*-polarized laser beam was splitted into two parts with equal intensities, which were steered at certain incident angle onto the target (Fig. 1).

The delay between the two interfering beams was adjusted to ensure no optical path difference (OPD) at the center of the beams on the target (Fig. 1). Following from the geometry, at the edges of the beam the OPD was not zero but significantly less than the coherence length of the laser. Hence, the visibility of the interference patterns was almost constant and close to unity across the sample.

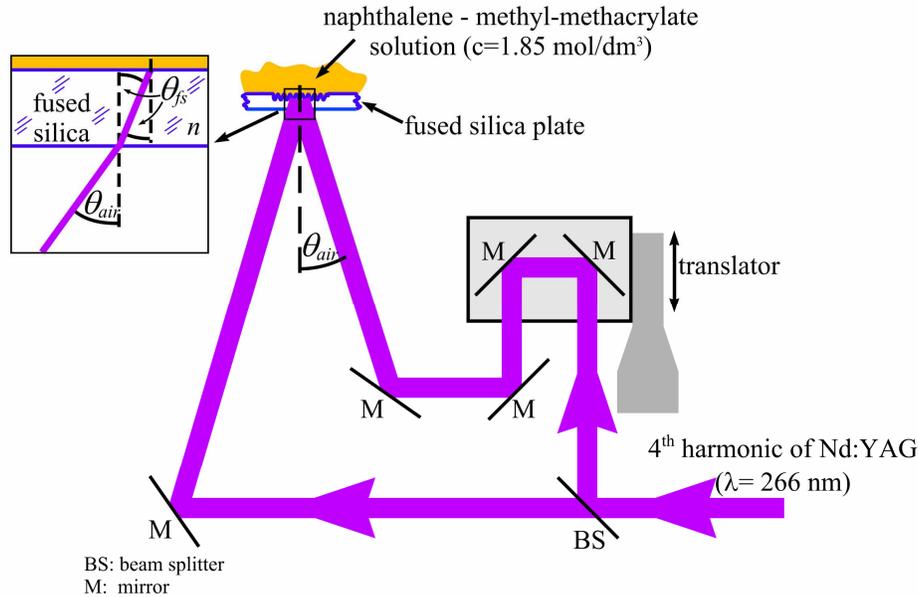


Fig. 1. The scheme of the experimental setup

The interference fringe separation

$$p = \frac{\lambda_{air}}{2 \sin \theta_{air}} \quad (1)$$

determines the period of the grating, where  $\lambda_{air}$  is the wavelength in the laboratory air and  $\theta_{air}$  is the incident angle of the laser beam [14]. In our experiment the incident angles were  $14^\circ$ ,  $30^\circ$ , and  $60^\circ$  corresponding to interference fringe separations of 550 nm, 266 nm, and 154 nm, respectively. The irradiated area was between  $0.53 \text{ mm}^2$  and  $0.68 \text{ mm}^2$ .

Fused silica plates with a thickness of 1 mm (Suprasil II, Heraeus) were applied as target material being transparent at the laser wavelength of 266 nm. Naphthalene and methyl-methacrylate solution with  $c=1.85 \text{ mol/dm}^3$  concentration (saturated solution) was used as liquid absorbent. The morphology of the gratings was studied by a PSIA XE-100 atomic force microscope (AFM) in non contact mode.

Initially the incident angle was chosen to be  $14^\circ$ . The laser fluence was varied within the range of  $285 \text{ mJ/cm}^2$  and  $680 \text{ mJ/cm}^2$ , while the number of laser pulses was changed between 50 and 1800. These parameters were optimized in order to fabricate gratings both with the highest quality and the largest modulation depth, similarly to that was presented in our earlier study [15]. The best quality grating was achieved at  $330 \text{ mJ/cm}^2$  with 50 pulses providing a modulation depth of 120 nm (Fig. 2), while the highest modulation depth of 200 nm was produced by 100 laser pulses at  $420 \text{ mJ/cm}^2$ . In this latter case the quality of the grating was slightly degraded.

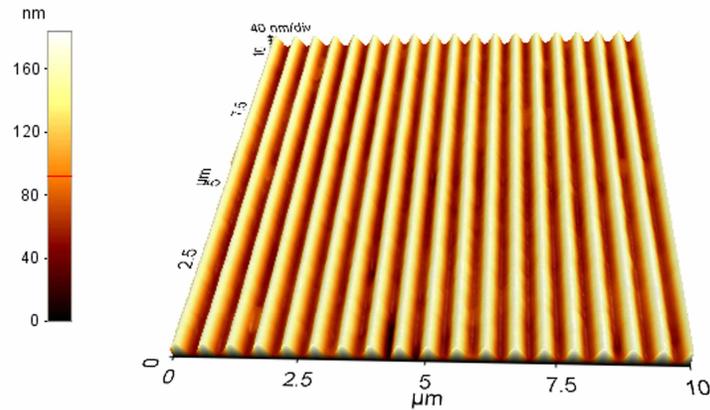


Fig. 2. AFM image of the best quality grating having 550 nm period

Having varied the fluences within a relatively large range, it was experienced that the highest quality gratings were always achieved by laser pulses not substantially more than 50. We think that the possible reason of this optimum value was resulted from two processes. First, as it is known, the etched depth is proportional to the number of laser pulses. Second, the low frequency mechanical vibration of the optical bench as well as the slow air flow in the laboratory made the interference fringes change their spatial position on the sample. The blur of the overlapping fringes is proportional to the time elapsed during the total etching process. Since the repetition rate of the laser is fixed, the blur is eventually proportional to the number of pulses applied. To benefit from this experience, the number of laser pulses was kept constant at 50 for the production of gratings in the following experiments.

As a next step, the incident angle was increased to  $30^\circ$  corresponding to 266 nm interference fringe separation. The highest quality grating (Fig. 3) was produced at a fluence value of  $525 \text{ mJ/cm}^2$ . As it can be seen, the modulation depth decreased and the quality of the grating is slightly deteriorated comparing to the previous case.

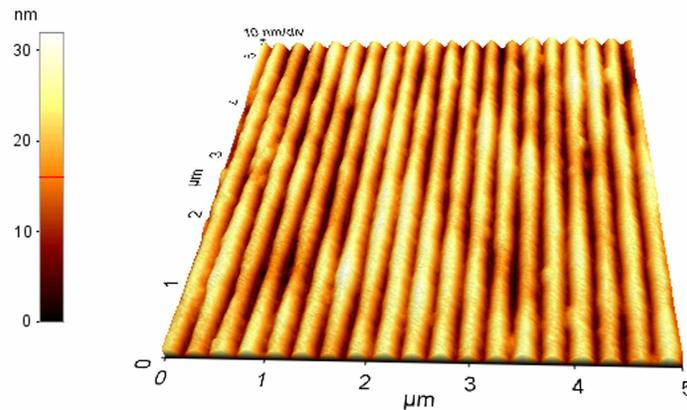


Fig. 3. AFM image about a grating having 266 nm period

The setup was further modified to reach the incident angle of  $60^\circ$ , resulting in a grating period of 154 nm. The optimal quality grating was fabricated at  $500 \text{ mJ/cm}^2$  fluence (Fig. 4).

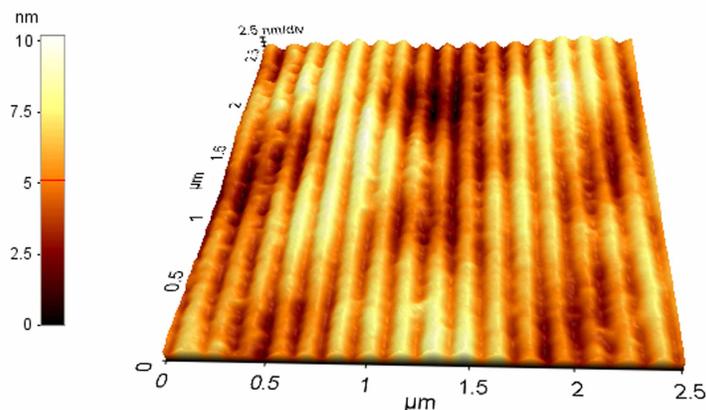


Fig. 4. AFM image about a grating having 154 nm period

The quality of a surface is usually characterized by its surface roughness, which is the average deviation of a surface from a plane. In the case of laser assisted grating fabrication, the modulation of the surface at frequencies lower than the generated grating period is of interest. To establish the surface waviness ( $W_a$ ), which is the usual roughness parameter taken at longer wavelengths, the AFM images were low-pass Fourier-filtered at a cutoff frequency of  $1/2p$ , then  $W_a$  was established. The waviness of the produced gratings (Table 1) was usually higher than the waviness of the unprocessed fused silica target, which was measured to be 0.25 nm. In the optimized cases  $W_a$  had minimum, while the maximum  $W_a$  of the non-optimized procedures was higher than 100 nm.

Table 1. The parameters of the produced gratings

Fabrication parameters			Grating parameters				
Incident angle [°]	Fluence [mJ/cm <sup>2</sup> ]	Number of laser pulses	Grating period [nm]	Groove density [grooves/mm]	Modulation depth [nm]	$W_a$ [nm] ( $\lambda_{\text{cutoff}}=2p$ )	Quality factor (Mod.depth/ $W_a$ )
14	330	50	550	1818	120	2.5	48.0
30	525	50	266	3759	20	1.87	10.7
60	500	50	154	6494	3	1.03	2.9

An equally important parameter of the fabricated gratings is the modulation depth. Since the waviness can be regarded as a low frequency surface noise, it is reasonable to introduce a general quality factor defined by the ratio of the modulation depth and the waviness parameter. Comparing the AFM images of Fig. 2–Fig. 4, the visible quality of the grating deteriorates with decreasing the grating period. Indeed, this quality loss at the two lower period gratings can be seen clearly from the decrease of the quality factor in the last column of the Table 1.

Since the produced 550 nm period structure can be operated as UV transmission gratings, this feature was tested with the 266 nm laser beam. The first and the second order of the diffracted beams were observed at a diffraction angle of approx. 29–30° and 75°, respectively.

### 3. Summary

We introduced a new, so called two-beam interferometric laser induced backside wet etching (TWIN-LIBWE) method to produce fused silica grating with as small period as 154 nm, which is, to our knowledge, the smallest grating constant fabricated by laser techniques directly in fused silica so far. It was also demonstrated that the produced good quality gratings could be operated as UV transmission gratings. These results show that the TWIN-LIBWE technique may be a promising candidate for production of good quality sub-100 nm gratings into fused silica as well as other transparent materials.

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