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Fabrication of micro-optical elements in quartz by laser induced backside wet etching

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Abstract

Micro-optical elements in quartz with continuous profiles were fabricated by laser induced backside wet etching, where the quartz plate is in contact with an organic solution, that strongly absorbs UV laser light. The absorption of the laser light by the organic molecules generates a temperature and pressure jump at the quartz–liquid interface, which results in etching of the quartz. This method allows microstructuring of quartz with an XeCl or KrF excimer laser at laser fluences well below the damage threshold of quartz for these wavelengths. The obtained structures in quartz vary from binary structures, as part of random phase plates, to complex three-dimensional structures, e.g., Fresnel lenses. The Fresnel lenses were created by applying diffractive grey tone phase masks fabricated by e-beam lithography. The roughness of the obtained structures varies from 50 to 500 nm, depending on the laser fluence and the applied substrate.
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1. Introduction

The miniaturization of optical elements is one of the key techniques in modern optics. Arrays of microoptical elements, especially in quartz, are applied, for example, as beam homogenizers for high-power excimer and Nd^{3+}:YAG lasers. Micro-optical elements with continuous profiles, e.g., Fresnel lenses, are usually prepared by photolithography and subsequent transfer of the resist profile into the substrate surface by reactive ion etching (see, for example, Ref. [1]). This technique requires an excellent control of the exposure dose, the resist characteristics, and the proportional etching process.

A simpler method for the fabrication of micro Fresnel lenses in polymers has been reported by

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David et al. [2]. Here, a diffractive grey tone phase mask consisting of phase grating structures designed to scatter a defined fraction of the incoming radiation out of the imaging aperture was applied to transfer the mask image into the polymer by direct laser ablation. However, quartz glass is transparent to the common laser wavelength and can only be structured by vacuum UV (VUV) (157 nm) lasers [3,4] or by ultrafast (fs or ps) pulsed lasers [5–7]. The VUV lasers are complicated to use, as they require vacuum or at least a transparent inert gas for the beam pass and standard quartz optics have a strong absorption. On the other hand, today's ultrafast lasers have a low power, so they can only be used with a small focused beam. As a consequence, complex patterns have to be generated by slow, sequential scanning.

Niino and co-workers investigated the etching of quartz using various lasers (i.e., excimer and Nd:YAG) and an organic (e.g., pyrene in acetone) solution, which is in contact with the quartz plate [8–10]. The organic solution is irradiated *through* the quartz and the UV photons are strongly absorbed in a thin liquid layer in contact with the quartz. Excitation and relaxation of the pyrene generates a temperature jump at the quartz–liquid interface, which results in etching of the quartz. This technique was named laser induced backside wet etching (LIBWE). Up to now mainly proof of principle experiments, i.e., hole and line structures prepared in quartz by using LIBWE and applying a slit mask [8–10] or interference grating projection were reported [11].

The fabrication of complex patterns with continuous profiles, i.e., Fresnel lenses, in quartz by LIBWE and diffractive grey tone phase masks is presented in this paper.

2. Experimental

A quartz plate with a thickness of 0.5 mm was used as sample. As irradiation sources a KrF (248 nm, FWHM 30 ns, Lambda Physics) and an XeCl (308 nm, FWHM 30 ns, Lambda Physics) excimer laser were applied. The intensity of the lasers was controlled by an attenuator, consisting of a dielectrical plate. A solution of pyrene in acetone with a concentration of 0.4 M/l was utilized as ''etchant''. One side of the quartz plate is in contact with the solution, while irradiation is applied through the other side as shown in Fig. 1. Diffractive grey tone phase masks, fabricated by e-beam lithography [2], are utilized to modulate the laser intensity. The mask image is projected onto the quartz by a lens ($f = 10$ cm) creating structures with a typical size of 0.5×0.5 mm^2. The etching is carried out with laser fluences from 0.7 to 1.1 J/cm^2 and a variable number of pulses, typically around

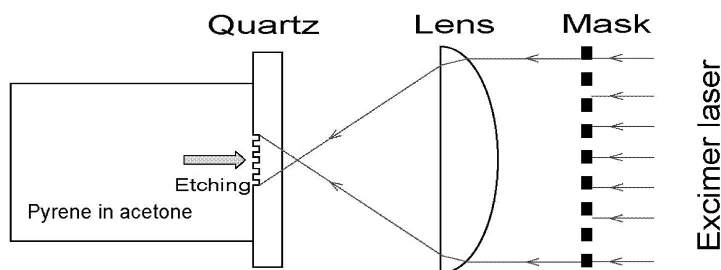

Fig. 1. Scheme of the experimental setup for laser induced backside wet etching.

800. The depth profiles of the structures are analyzed with a profilometer and by scanning electron microscopy (SEM).

3. Results and discussion

The etching of quartz by LIBWE revealed a behaviour similar to the ablation of polymer, where a threshold fluence of ablation exists. Only with laser fluences above this threshold can ablation be accomplished. The threshold for quartz using LIBWE was determined experimentally by measuring the etch rates at various laser fluences (shown in Fig. 2A). The threshold fluence for irradiation at 308 nm is 0.5 J/cm^2 and for 248 nm irradiation is 0.3 J/cm^2. The obtained threshold values are well below the damage threshold of quartz, which ranges from 10 to 20 J/cm^2 for 248 nm and 308 nm irradiation [4]. Above the threshold two linear regimes are observed, as shown in Fig. 2A. At low fluences (below 500 mJ/cm^2 for 248 nm and 700 mJ/cm^2 for 308 nm) the etch rate only increases slowly with fluence, while above these values the slopes of the curves are about 2.5-times higher (in absolute values). At all fluences the etch rate of quartz is larger for 248 nm irradiation than for 308 nm irradiation. This is due to the different absorption mechanisms in the pyrene molecule at the different irradiation wavelengths [12,13]. A typical roughness of the etched areas of 50 nm was obtained. The roughness depends on the quality of material and on the applied laser fluences.

The efficient etching of quartz with etch rates of several nm per pulse is due to the deposition of the laser energy in a thin pyrene–acetone layer above the quartz surface. The linear absorption coefficient of pyrene in dilute solution was calculated from absorption spectra, and an optical penetration depth of 0.7 μm was determined for a pyrene acetone solution (0.4 M/l). This suggests, that the laser energy is deposited in a layer of less than 1 μm thickness. The laser induced temperature jumps at the quartz–liquid interface for various laser fluences (shown in Fig. 2B) and irradiation wavelengths were calculated by a modified model for nonradiative relaxation of organic molecules developed for laser

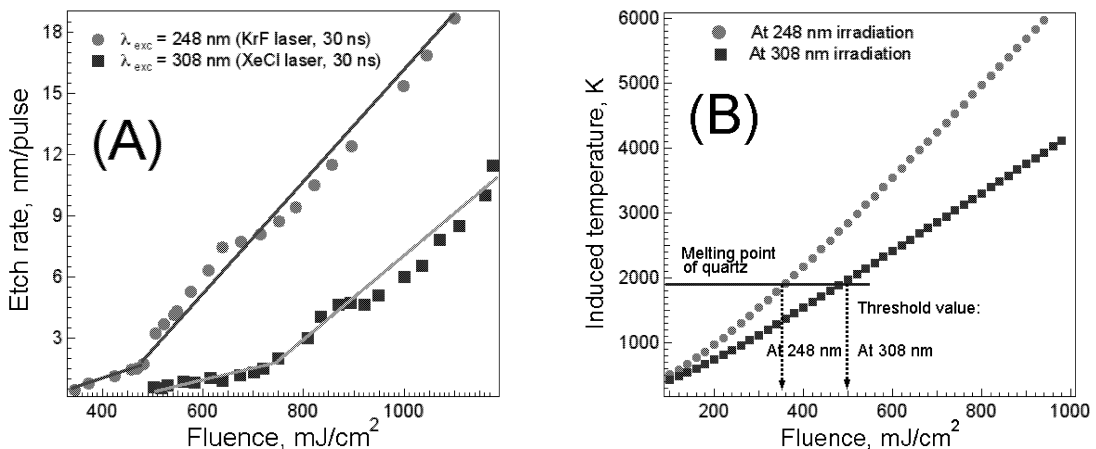

Fig. 2. (A) Etch rates of quartz versus laser fluence: (○) KrF laser (248 nm), (□) XeCl laser (308 nm). (B) Calculated temperature at the quartz liquid interface, as a function of the laser fluence: (○) KrF laser (248 nm), (□) XeCl laser (308 nm). The laser energy is absorbed in a layer with a thickness of 0.7 μm.

ablation of polymers [12,13]. The laser induced temperature of the quartz surface in contact with the liquid is around 1900 K for both threshold fluences (248 and 308 nm). This temperature corresponds quite well to the melting point of quartz (1880 K). The reason why melting of quartz coincides with etching is not clear, as ''liquid'' quartz is not soluble in acetone. One possible explanation can be derived from the critical temperature T_C of acetone which is about 500 K. When the laser induced temperature in the pyrene–acetone solution exceeds T_C, the solution vaporizes and forms bubbles (cavitations). The fast expanding or collapsing bubbles create a high pressure which interacts with the surface of the melted quartz and removes the quartz by mechanical forces. This mechanism can explain the experimental etching results in the low fluence range, while for the high fluence range another mechanism may be dominant. A likely process for removing the ''softened'' quartz are acoustic waves, which may be dominant in the high fluence range. The detailed analysis of the LIBWE process and the different mechanisms are an ongoing project.

To investigate the spatial resolution of the LIBWE process, a Siemens star in quartz was fabricated by KrF laser irradiation (248 nm) with a fluence of 0.7 J/cm^2 and 800 pulses (shown in Fig. 3A and B). The SEM pictures of the Siemens star reveal a clean etching without any pronounced cracking of the quartz. We were able to resolve structures sizes down to 2 μm by analysing the SEM pictures (Fig. 3). Whether this represents an inherent limitation of the LIBWE process is not clear, as the used projection optics was not optimised in terms of resolution.

Microstructures in quartz have, as discussed above, important applications in optics. Various three-dimensional structures such as Fresnel lenses with continuous structure profiles were prepared to test the potential of LIBWE as a fabrication method. A Fresnel lens etched into quartz with 308 nm radiation and a fluence of 1.1 J/cm^2 is shown in Fig. 4. The line scan of the Fresnel lens shown in Fig. 4b reveals a plateau in the centre, which is not etched at all. This is probably due to the fact, that the fluence through the diffractive grey tone phase mask is below the threshold fluence for the ablation of quartz in this region. This effect could be avoided by considering the threshold in the design of the mask. The depth variations of the left and right sides are probably due to the inhomogeneous laser beam profile. Fig. 5 shows the resulting structure when using the same mask for 248 nm irradiation (0.7 J/cm^2 and 800 pulses). The SEM picture of the Fresnel lens reveals sharp edges between the

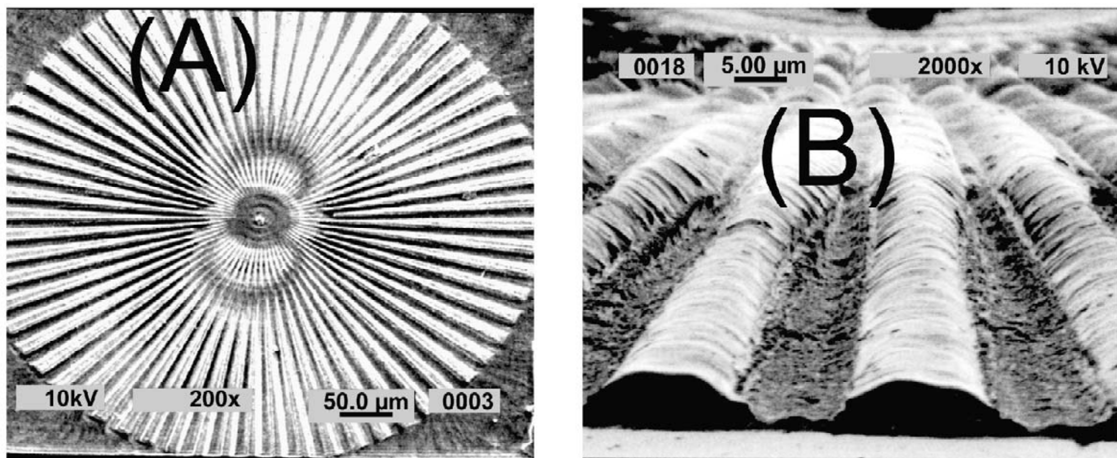

Fig. 3. SEM picture of a Siemens Star etched in quartz by LIBWE using a KrF laser.

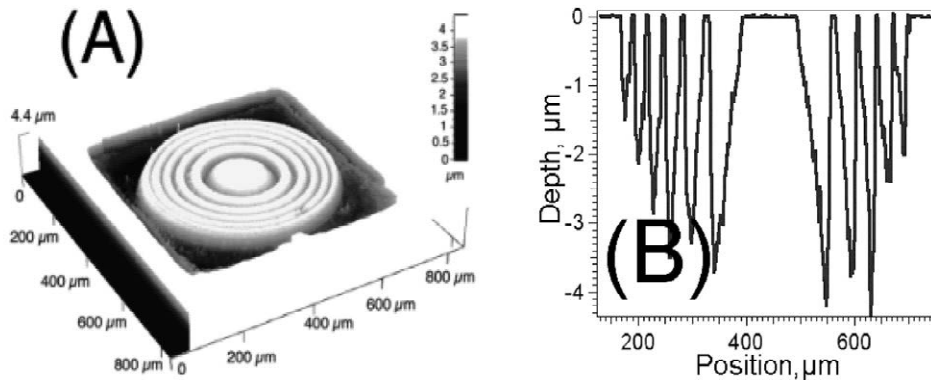

Fig. 4. (A) 3D profilometer scan of a Fresnel lens etched in quartz by LIBWE using an XeCl (308 nm) laser. (B) Line scan of the etch profile, corresponding to the Fresnel lens in (A).

concentric rings. The line scan (shown in Fig. 5B) presents the typical features of a Fresnel lens, i.e., a parabolic depth profile in the center and triangular structure on the sides (see, for example, Ref. [14]). No effects of the materials threshold are observed. The reason for this is, that the depth of the diffractive mask structures was optimised for 308 nm, which results in a considerable transmission of the ‘‘dark’’ areas of the mask.

Another example for a micro optical device is a random phase plate (RPP) (shown in Fig. 6). The RPP can be used as a ‘‘beam homogeniser’’ and is a possible alternative to the common microlens arrays, which are very expensive. A random phase plate [15] is a binary diffractive optic, which enlarges a focal spot while imposing a smooth spatial profile. The surface of the RPP was randomly tessellated in a square basic elemental shape. Half of the elements produce a zero phase shift while the other half produce a π phase shift. The design of the RPP starts with computer generation of the positions of the basic elements in the substrate, which should be etched to a depth of $d = \lambda/[2 \cdot (n-1)]$, where n is the refractive index of the substrate at the operating wavelength λ. The optimization of the size of the basic patterns corresponds to the effective homogenizing of the

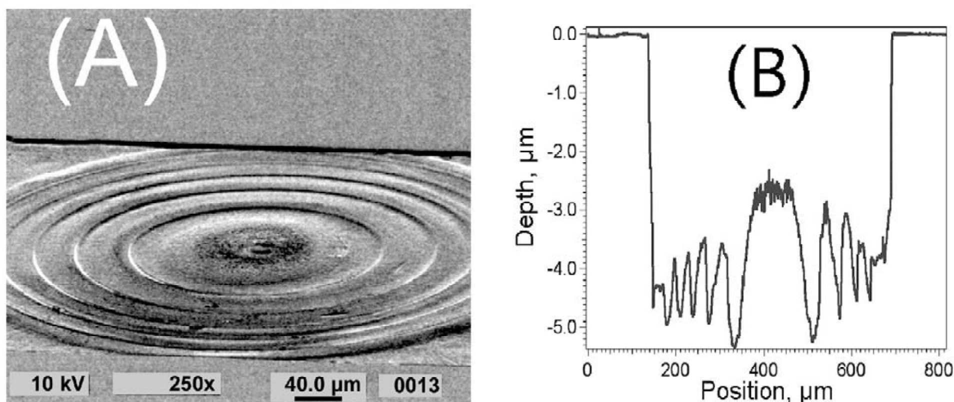

Fig. 5. (A) SEM picture of a Fresnel lens etched in quartz by LIBWE using a KrF (248 nm) laser. (B) Line scan of the etch profile, corresponding to the Fresnel lens in (A).

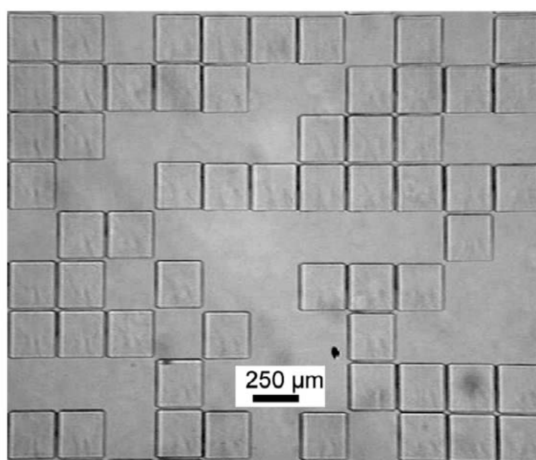

Fig. 6. Section of a random phase plate in quartz prepared by LIBWE using an XeCl laser.

incoming beam intensity. Each basic element acts as a diffractive aperture and the resulting focal profile is the superposition of the diffracted light from all other apertures.

An RPP with a pattern size of 250 μm was prepared in quartz by LIBWE. The etch depth of the RPP was designed for testing the RPP with the fourth harmonic of the Nd^{3+}:YAG laser at 266 nm. The spatial energy distributions within the focal spot without and with RPP was examined by a CCD system. The results are shown in Fig. 7A and B. Analysis of the experimental results suggests that the energy distribution close to the focal plane without the RPP has a nearly Gaussian profile, and a spot size of a 200 μm. The beam size close to the focal plane increases with the RPP in front of the collecting lens, as shown in Fig. 7B. The flat top spatial energy profile, observed with the RPP shows clearly a better, more homogenous energy distribution.

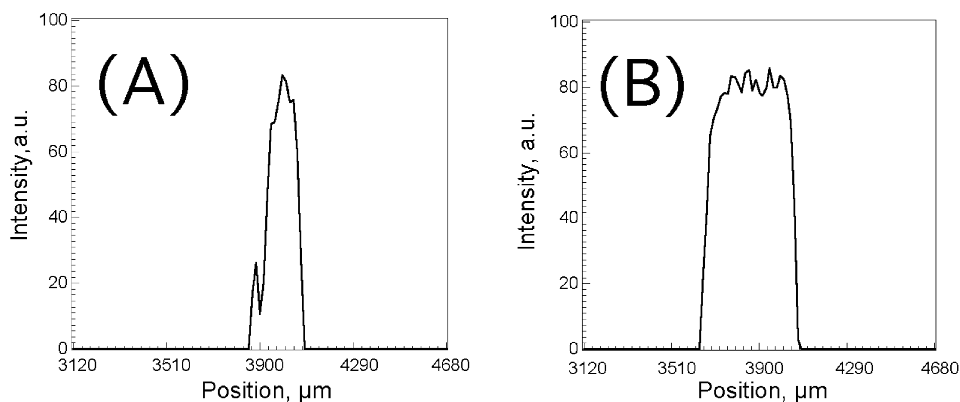

Fig. 7. Beam profile of a Nd:YAG laser (fourth harmonic, 266 nm) close to the focal plane without an RPP (A) and with an RPP (B).

4. Conclusions and outlook

We have shown that laser induced backside wet etching is a suitable technique for the fast fabrication of microstructures in quartz using a relatively simple setup. Our first results indicate, that the application of diffractive grey tone masks allows the creation of complex three-dimensional patterns, e.g., Fresnel lenses.

Another optical element that has been created by LIBWE is a random phase plate suited as a beam homogenizer for high power excimer and Nd^{3+}:YAG lasers.

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