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Microstructuring of fused silica by laser-induced backside wet etching using picosecond laser pulses

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ABSTRACT

The laser-induced backside wet etching (LIBWE) is an advanced laser processing method used for structuring transparent materials. LIBWE with nanosecond laser pulses has been successfully demonstrated for various materials, e.g. oxides (fused silica, sapphire) or fluorides (CaF_2, MgF_2), and applied for the fabrication of microstructures. In the present study, LIBWE of fused silica with mode-locked picosecond ($t_p = 10$ ps) lasers at UV wavelengths ($\lambda_1 = 355$ nm and $\lambda_2 = 266$ nm) using a (pyrene) toluene solution was demonstrated for the first time. The influence of the experimental parameters, such as laser fluence, pulse number, and absorbing liquid, on the etch rate and the resulting surface morphology were investigated. The etch rate grew linearly with the laser fluence in the low and in the high fluence range with different slopes. Incubation at low pulse numbers as well as a nearly constant etch rate after a specific pulse number for example were observed. Additionally, the etch rate depended on the absorbing liquid used; whereas the higher absorption of the admixture of pyrene in the used toluene enhances the etch rate and decreases the threshold fluence. With a $\lambda_1 = 266$ nm laser set-up, an exceptionally smooth surface in the etch pits was achieved. For both wavelengths ($\lambda_1 = 266$ nm and $\lambda_2 = 355$ nm), LIPSS (laser-induced periodic surface structures) formation was observed, especially at laser fluences near the thresholds of 170 and 120 mJ/cm^2, respectively.

1. Introduction

The laser-induced backside wet etching is an innovative etching technique used for microstructuring of fused silica, sapphire, CaF_2, BaF_2, and other transparent dielectrics [1–3]. The method was initially investigated by Wang et al. [4,5] and is characterized by producing low threshold fluence and low roughness of the etched surface. Usually, transparent dielectrics were are patterned by using laser ablation and by exploiting the laser radiation of a short wavelength, for example a F_2 laser, whose photon energy is higher than the band gap of these materials. Ultrashort pulse lasers were also used in dielectric patterning. Another structuring approach for such materials is dry etching, but this requires photolithographic masking. LIBWE is the only laser processing method which provides the flexibility as well as the low threshold fluence and the nanometre-scaled depth control which were necessary for the production of micro-optical elements such as Fresnel-lenses or diffractive gratings.

LIBWE with nanosecond lasers has been investigated in a number of studies [2,3,5–8] and the influence of various experimental parameters on the etch rate and surface morphology was examined for a variety materials. However, the etch mechanism of LIBWE with nanosecond lasers is still in discussion. Commonly, the etching has been explained with the following thermal etching approach [2,9,10]: the backside of the sample is in contact with a highly absorbent liquid. The laser beam then penetrates the transparent sample and is absorbed in a small liquid volume close to the sample's backside surface. Consequently, a thin layer of the sample surface drastically warms up through heat diffused from the laser-heated liquid to the surface, causing the layer to reach temperatures up to the melting or softening point of the sample material. A number of laser-induced secondary processes in the liquid, for example, bubble formation and collapse, have also to be considered. Such mechanical processes generate pressure and stress onto the softened surface of the sample and finally cause the removal of the material. However, the principle background

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processes as well as the mechanism as a whole are not yet fully understood.

To improve the understanding of these LIBWE processes investigations with picosecond (ps) and femtosecond (fs) laser pulses have been performed by Böhme et al. [11] and Pissadakis et al. [12]. It should be noted that a simple thermal etching mechanism was excluded due to the short heat diffusion length in a femtosecond pulse duration time scale. Contrary to the thermal dominated etching mechanism for ns LIBWE, an etching mechanism based on the generation of a defect-enriched surface layer, in which the laser beam can be absorbed, was suggested for fs LIBWE [11]. Additionally, the short pulse lengths as well as the high intensity of the laser pulse affect the interaction of the photons with the sample material and with the liquid. Hence, new processes with ultrashort laser pulses in comparison to nanosecond laser pulses have been considered, especially nonlinear processes such as multiphotonic absorption or laser-induced defect generation and accumulation.

This study focuses on LIBWE of fused silica in the picosecond time scale ($t_\mathrm{p} = 10$ ps). The influence of several process parameters, e.g. wavelength, pulse number, absorbing liquid, and laser fluence on the etch rate were investigated. The effect of these parameters on the morphology of the etch pits is shown. The present results were compared and discussed on the basis of previous studies which had investigated LIBWE with nanosecond [2–5,10,13] and ultrashort laser pulses [11,12].

2. Experimental

The basic experimental set-up for laser-induced backside wet etching is described elsewhere [6]. In this study, two different laser systems were used for the investigations on etching with UV laser radiation.

The first experimental system was based on the 4th harmonic of a Nd:YVO$_4$-laser (EKSPLA PL10100 with external harmonic module) which provided a wavelength of $\lambda = 266$ nm and a pulse length of approximately 10 ps. The pulses were selected at a repetition rate of $R_\mathrm{p} = 1$ kHz and the UV beam was focused by a lens with a focal length of 50 mm onto the backside of the fused silica sample. The sample was moved by a computer-controlled *x*–*y* stage, relative to the fixed laser beam.

The second experimental set-up used the 3rd harmonic of a Nd:YVO$_4$-laser (EKSPLA PL10100/TH) with a wavelength of $\lambda = 355$ nm and a pulse length of approximately 10 ps. The pulses were selected at a repetition rate of R_p 50 kHz. The laser system was used in conjunction with a laser scanning system with the telecentric *f*–Θ lens having a focal length of 103 mm. For both laser systems, an external Pockels cell was used for the selection of the pulse numbers and pulse energy control.

The fused silica samples cut from double sided polished wafers with a thickness of about 380 μm and a surface roughness of less then 0.3 nm (rms) were used "as received", without any additional cleaning. As the absorbing liquid, either pure toluene or a saturated pyrene–toluene solution was used. After etching, the samples were ultrasonically cleaned with an acetone-ethanol solution. The etch depth was measured with a white light interference microscope (Micromap 512) and the topography of the surface was studied by a secondary electron microscope (SEM) after coating the samples with a thin Cr film.

The laser fluence *F* was calculated using the pulse energy E_pulse and the Gaussian beam radius w_0 which was measured by the D^2 method [14,15]:

$$F = \frac{2^* E_\mathrm{pulse}}{\pi^* w_0^2}. \qquad (1)$$

Using the laser scanning system, the pulse number *N* per etched area was approximated with

$$N = \frac{w_0^* 2^* R_\mathrm{p}}{v_\mathrm{scan}}, \qquad (2)$$

where R_p is the pulse repetition rate and v_scan is the scan speed of the laser beam.

The etch rate was calculated from the final etching depth and the applied pulse number and, therefore, corresponds to the averaged etch rate.

3. Results and discussion

The first etching results with ps laser pulses at a wavelength of $\lambda = 266$ nm are presented. The etch rate of fused silica in dependence on the laser fluence for pure toluene as the absorbing liquid is shown in Fig. 1. The etch rate devolution can be separated into two fluence ranges with linear slopes. In the low fluence range up to 600 mJ/cm^2, the etch rate grows up to 45 nm/pulse with a slope of approximately 110 (nm/pulse)/(J/cm^2). At high fluences, the slope increases to 400 (nm/pulse)/(J/cm^2) and an etch rate of ∼100 nm/pulse is achieved at a fluence of 700 mJ/cm^2. A division into fluence ranges with different etch rate slopes is also occurred at nanosecond LIBWE with 248 nm [1,3,8,9]. However, the etch rate values at nanosecond LIBWE are roughly one order of magnitude less [8]. The threshold fluence F_th of ps LIBWE with $\lambda = 266$ nm was evaluated to be approximately 170 mJ/cm^2 by extrapolating the linear regression of the etch rate in Fig. 1. This approach to define the threshold fluence is not exact because very close to the threshold fluence it is possible that the etch rate does not dependence linear on the laser fluence. However, this approach to define the threshold by extrapolating the linear regression of the etch rate is common and allowed the comparison of the found experimental data with data from references. In comparison to LIBWE with nanosecond pulses (F_th ∼350 mJ/cm^2), this threshold value is lower by a factor of 2 [8]. The threshold of the sub-picosecond UV LIBWE done by Böhme et al. [11] was approximated to be in the same fluence range as ps LIBWE with a $\lambda = 266$ nm wavelength.

The etch rate in dependence on the applied pulse number is shown in Fig. 2 for two different laser fluences. The linear fits of the etch rate at the fluences of 190 mJ/cm^2 and 310 mJ/cm^2 are shown.

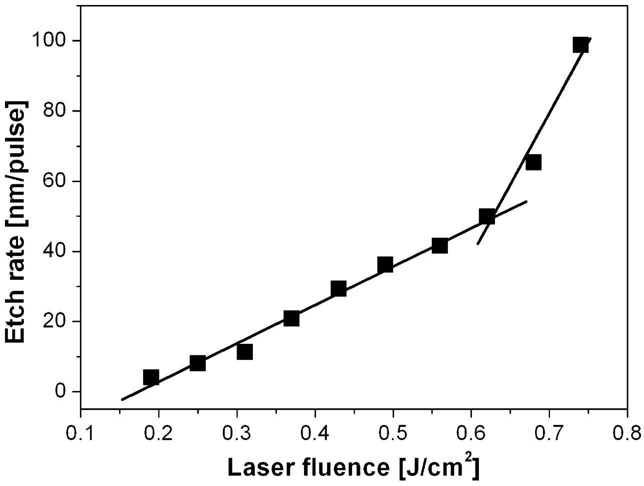

Fig. 1. Etch rate of fused silica in dependence on the laser fluence for etching with a 10 ps pulse duration and $\lambda = 266$ nm wavelength laser set-up. The absorbing liquid was pure toluene and the etch rate was calculated from 25 laser pulses. In addition to the measured data (full rectangle), a linear fit of the measured data in the low and the high fluence ranges is shown (lines).