Laser Induced Backside Wet Etching: Mechanisms and Fabrication of Micro-Optical Elements

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Abstract. A novel method for the fabrication of complex 3 dimensional structures, such as plano-convex microlens arrays in quartz, is presented. This technique is applied for precise micromachining of UV transparent materials, e.g. quartz, by using a conventional XeCl excimer laser and an organic solution which is in contact with the substrate. Strong absorption of the intense laser light results in the formation of the high pressure and temperature jumps at the quartz-liquid interface, which are the key elements in this process. A combination of laser assisted wet etching with the projection of a Diffractive Gray Tone Phase Mask is applied to fabricate diffractive and refractive micro-lens arrays in quartz. A quartz micro-lens array, which consists of 10 × 10 plano-convex microlenses is tested as a beam homogenizer for the quadrupled Nd:YAG laser.

1. Introduction

Quartz is one of the leading materials in optics and optoelectronics due to its high transparency over a wide wavelength range and its strong resistance to mechanical and chemical damage. Therefore, micro-optical components, such as quartz micro-lenses, are used in many different applications including optical interconnections in telecommunication, optical imaging systems for UV photolithography and in UV laser beam shaping and homogenizing [1]. However, fabrication of micro-lenses in quartz is both technologically challenging and expensive due to the properties of the material.

A conventional technique for the fabrication of the quartz micro-lenses is the combination of photolithography, resist melt-reflow and Reactive Ion Etching (RIE) [1, 2]. This approach requires high vacuum conditions, and precise control of the proportional etching process. Direct structuring of quartz can be also achieved by femtosecond lasers or vacuum ultraviolet lasers [3-6] due to the effective multi-photon ionization or absorption processes involved. However, at present femtosecond lasers have both cost and high volume processing limitations (e.g. small beam diameter) and the fabrication of quartz micro lenses can be a tedious and slow scanning process. For VUV lasers, expensive optical components and a high vacuum environment (or at least optically transparent gasses) are required for the beam path. Thus, it is of interest to consider alternative approaches for micromachining of quartz.

A novel approach for the fabrication of microstructures in quartz and in other UV transparent materials, such as CaF_2, BaF_2, sapphire has been developed by Niino et al [7-14] This method is known as Laser Induced Backside Wet Etching (LIBWE) and has been applied for the fabrication of square, round and line microstructures in fused silica [7, 8, 15, 16], CaF_2 [17, 18], BaF_2 [18], sapphire

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[18-20], fluorinated resin [9]. The major difference between LIBWE and other methods which are used for structuring of UV transparent materials is the application of conventional KrF or XeCl excimer lasers and an organic liquid which strongly absorbs the laser radiation. The principle of LIBWE is based on the generation of intense temperature and pressure increases at the thin interface between the material and liquid due to the strong absorption of the laser light. Various organic solutions such as pyrene in acetone, THF, toluene and cyclohexane [7-9, 15, 18, 19, 21, 22], aqueous pyranine or naphatelene [23], naphthalene in methyl methacrylate[24] and pure toluene [12, 14, 25] can be applied as etching media. A XeCl (308 nm, 30 ns), KrF (248 nm, 20 ns), ArF (193 nm, 20 ns) or XeF (351 nm, 20 ns) laser can be applied as the irradiation source.

The additional combination of LIBWE with an interference setup allows fabrication of submicron quartz gratings [21]. We developed and optimized a new technique for the fabrication of complex structures such as diffractive and refractive micro-lens arrays [18, 22, 26, 27]. This approach is a combination of LIBWE and projection of a Diffractive Gray Tone Phase Mask (DGTPM). The DGTPM is a periodical binary structure fabricated in quartz by e-beam lithography and RIE [28, 29]. The DGTPM is used to modulate the incoming laser beam intensity projected onto the backside of the quartz substrate, where the etching takes place. This method can be used to fabricate a micro-lens array with the diameter of each micro-lens ranging from 0.25 up to 0.5 mm. In order to fabricate larger optical elements we would need to use a more energetic laser pulse, e.g. XeCl (308 nm, 200 ns, 20 J/pulse, Sopra). However, such lasers have much longer laser pulse durations and therefore the etch rate behavior may be very different compared to etching with a conventional XeCl excimer laser (308 nm, 30 ns, 0.6 J/pulse, LambdaPhysics). In this paper we present new results on the etching of quartz with a XeCl excimer laser which has a longer (200 ns) pulse length. In addition, an array of quartz micro-lenses with a diameter of each micro-lens of 1 mm was fabricated. This micro-lens array was tested as a beam homogenizer for a high power Nd:YAG laser beam.

2. Experimental Setup

The irradiated sample consisted of a quartz plate with a thickness of 2.3 mm and a RMS roughness of 5 nm. Two XeCl excimer lasers (λ=308 nm) with different pulse lengths (30ns and 200 ns) were used to irradiate the quartz and 1.4 M pyrene in the tetrahydrofuran (THF) etching media. The etch rate of quartz was measured using the experimental setup described elsewhere [22]. The laser fluence was varied between 0.5 J/cm^2 and 2.5 J/cm^2 by using a dielectric attenuator plate, while the laser pulse number was varied between 10 and 800, depending on the laser fluence. The in-plane size of the etched features was 1.2 × 1.2 mm. The depth profile of the etched features was obtained with a Dektak 8000 surface contact profilometer. The surface of the etched features was examined by confocal Raman microscopy using a HeNe laser (632 nm, 30 mW) excitation source.

The experimental setup for the fabrication of quartz microlens arrays is shown in Figure 1. The laser beam passes through the circular aperture and Diffractive Gray Tone Phase Mask. The DGTPM is used to modulate the incoming laser beam intensity, which is projected by an imaging lens (f=10 cm) onto the backside of the sample, where the etching occurs.

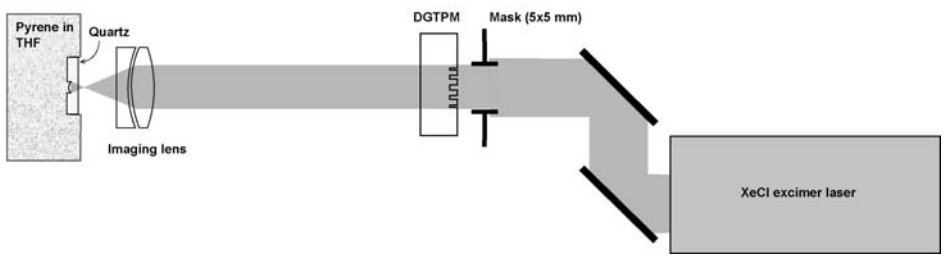

Figure. 1 Experimental setup for the fabrication of the quartz micro-lens array.

3. Results and Discussion

The efficient etching of quartz by LIBWE with an etch rate up to 35 nm/pulse (shown in Figure 2A) is related to the strong absorption of the laser light at the thin layer between the quartz and liquid (light penetration depth is less than 1 micron [8]). Rapid relaxation of the excited dye molecules generates a fast increase of the substrate-liquid interface temperature resulting in softening, melting or even boiling of the quartz surface. Subsequently, a shockwave is formed and material is removed from the molten surface.

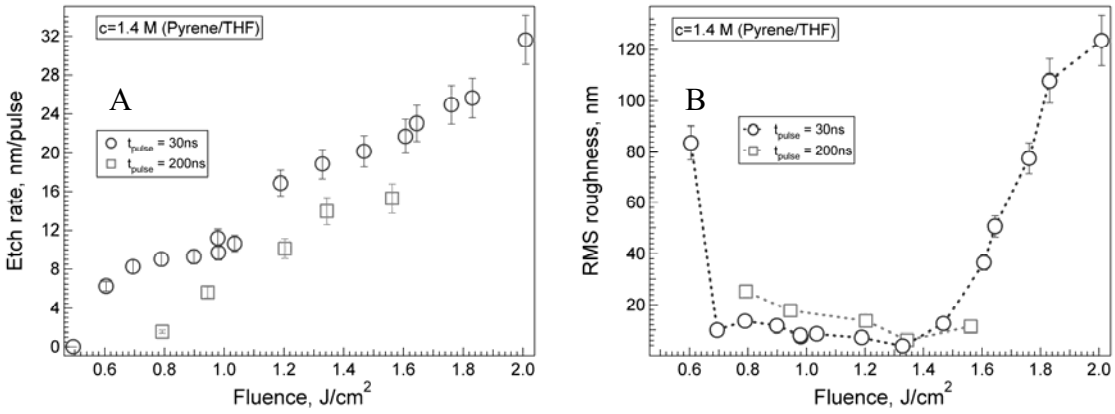

Figure 2. The etch rate (A) and the etch roughness (B) of quartz by LIBWE.

An important characteristic of the LIBWE process is the threshold fluence, which is defined as the lowest fluence above which significant etching starts. The experimentally obtained threshold fluence for ablating quartz using 30 ns and 200 ns XeCl excimer laser pulses is 0.5 J/cm^2 and 0.8 J/cm^2, respectively. The different threshold values observed for different pulse length can be related to the difference in the temperature jump at the quartz-liquid interface, which will be explained in detail later. The etch rate and etch roughness (shown in Figure 2B) obtained for the shorter laser pulse reveals a complex behavior where several etching mechanisms take place. This behavior was also observed using different solvents, containing pyrene [18, 26, 27] and it was pointed out [27] that at the low laser fluence the temperature jump maybe not high enough to melt the material [26, 27]. Etching is only observed after a certain number of pulses and carbon deposits are observed on the quartz surface prior and after etching. This indicates that the temperature jump was not high enough to melt the quartz but is sufficient to decompose the pyrene/solvent to form the carbon deposits. The formation of this carbon layer alters the absorption properties of the area under irradiation and causes an increase of the temperature jump which can now reach the melting point of quartz. Furthermore, molten quartz can be removed mechanically by the pressure jump generated from the shock wave or rapid growth and collapse of micro-bubbles [14, 22, 30].

The low laser fluence where strong incubation is observed also results in an increase of the etch roughness, which is clearly detectable in the case of low concentrated solutions [26, 27]. However, in the case of a highly concentrated solution, e.g. 1.4 M, an increase of the etch roughness is observed only slightly above the threshold fluence (shown in Figure 2B), although the incubation (not shown) and formation of carbon deposits is still detectable at the higher laser fluences (*less than* 0.9 J/cm^2).
The higher values of surface roughness observed with increasing laser fluence are probably related to the enhanced etching capability of the plasma formed in solution. Plasma formation becomes significant as the laser fluence-and correspondingly the solution temperature-is increased, and a drastic increase of the etch roughness of quartz was also observed in our earlier experiments [26]. However, an increase of the temperature results in a corresponding increase in the etch rate, which is not observable for 1.4 M pyrene in THF solution.

The etch rate of quartz using the longer laser pulse is clearly lower compared to the etching with the shorter laser pulse, which can be related to the lower temperature increase at the quartz-liquid

interface. The temperature jump calculated by the theoretical model developed by Fukumura et al. [31] suggests that a decrease in the laser pulse duration causes an increase of the laser induced temperature rise, i.e. the temperature at the quartz-liquid interface evaluated for 30 ns laser pulse at 1 J/cm^2 laser fluence is slightly above the melting point of quartz (~1600 C: [24]), while the T-jump calculated for 200 ns pulse is 500 K lower and does not exceed the melting point of quartz even at 1.2 J/cm^2. The etching of quartz using long laser pulses may also be influenced by the formation of carbon deposits, resulting from the decomposition of the solvent. This etching mechanism is similar to that obtained for the 30 ns laser pulse at low fluence. At the high laser fluences the etching mechanism for 200 ns laser pulses may change further, however further investigation is required to confirm this.

The surface of the features etched using 30 ns and 200 ns laser pulses and laser fluence of 1.6 J/cm^2 were examined by the confocal Raman microscopy (shown in Figure 3). The G and D bands of the amorphous carbon [32] are clearly revealed in the Raman spectra of quartz structured with 200 ns laser pulses), while no carbon deposits are detected for 30 ns laser pulse, indicating that the etching mechanism is clearly influenced by the formation of carbon when using the longer pulse.

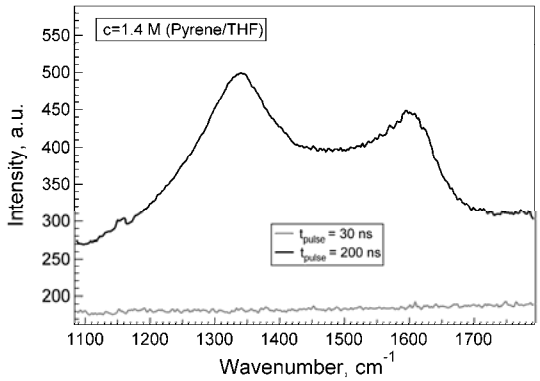

Figure 3. Raman spectra of quartz etched by LIBWE for 30 ns and 200 ns laser pulse (F=1.6 J/cm^2, 20 pulse) illustrating the carbon-associated D and G peaks at 1340 cm^{-1} and 1580 cm^{-1} [32] respectively.

The fabrication of quartz micro-lenses begins with the development of the Diffractive Gray Tone Phase Mask. This mask is produced in the quartz plate using e-beam lithography and Reactive Ion Etching (RIE) [28] and encodes the designed plano-convex lens shape structure. The mask is used for converting the flat top laser beam profile into the radial symmetry shape profile, which is imaged onto the backside of the sample, as shown in Figure 1. The threshold fluence of quartz (0.5 J/cm^2) and the lowest laser fluence above which no detectible carbon deposits on etched quartz areas is detected (0.85 J/cm^2) are taking into account while designing DGTPM. The laser fluences which are used for the fabrication of micro-lens lies in the range between 0.85 J/cm^2 to 1.5 J/cm^2 and fulfill the requirements of lowest etch roughness and lowest number of incubation. An array of the refractive micro-lenses is fabricated by using a step and repeat method.

The combination of LIBWE and projection of DGTPM is a very promising approach for the rapid prototyping of diffractive and refractive quartz micro-lenses, which can be applied as beam homogenizers for high power UV lasers. A digital image of the hexagonally packed quartz micro-lens array fabricated by LIBWE and projection of Diffractive Gray Tone Phase Mask is shown in Figure 4.

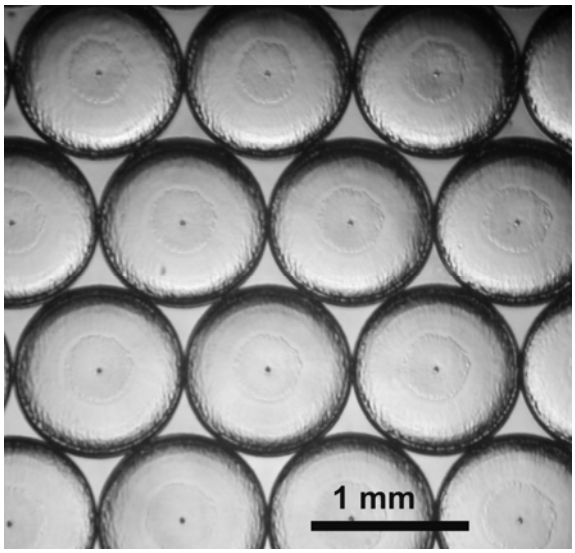

Figure 4. Image of the quartz microlens array fabricated by LIBWE and projection of a DGTPM

Each micro-lens in this array has a parabolic shape, which is in good agreement with the theoretical fit described in [26]. The micro-lens diameter is 1 mm, while the focal length of each lens is 60 mm.
The losses of the laser light through the micro-lens array is around 13 % and is related to the reflection losses (≈ 8%) and absorption/scattering losses (≈ 5%). The latest can be due to the above discussed thin carbon film formation on the top of the lenses, which can be removed by an additional cleaning process using RIE.

Homogenization of a Nd:YAG laser beam profile was tested using 10 × 10 micro-lens array and an experimental setup, described elsewhere [27]. The principle of the beam homogenizer is based on the splitting of the incoming laser beam into many beamlets that are superimposed on the focal plane of the collecting lens, placed close to the micro-lens array. Since the homogenized beam size is proportional to focal length of the collecting lens and the diameter and focal length of the micro-lens [33], the array can be optimized for specific structuring applications. The Nd:YAG laser beam profile with and without the beam homogenizer is shown in Figure 5. A clear improvement of the Nd:YAG beam profile is obtained using beam homogenizer which consists of the micro-lens array and the collecting lens (f=500 mm).

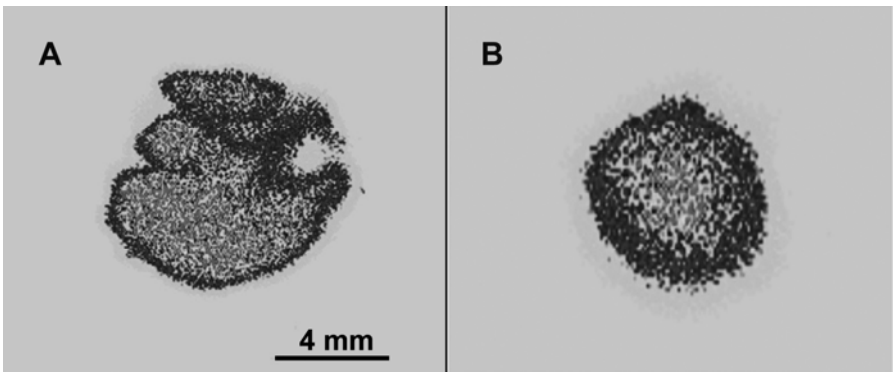

Figure 5. Scanned image of the spatial profile of a frequency quadrupled Nd:YAG laser beam patterned onto photosensitive paper. (A) The original beam profile with poor homogeneity due to a damaged region in one of the harmonic crystals. (B) Beam profile after modification using the laser-machined quartz micro-lens array.

4. Conclusion and Outlook

The combination of laser assisted wet etching and projection of diffractive gray tone masks is an alternative *one step* technique for the fabrication of refractive and diffractive micro-optical elements in quartz. In the present study we used a conventional XeCl excimer laser (308 nm, 30 ns) and an organic solution in contact with the substrate to build the micro-optical element. The laser fluence required for the fabrication of micro-optical elements is in the range (0.9-2) J/cm^2, which is well below the ablation damage threshold of quartz without solution [34]. The roughness of etched features in quartz varied between 10 to 200 nm and strongly depends on the laser fluence and laser pulse duration. The etching mechanism is influenced by the formation of carbon deposits at the low laser fluences for short laser pulses, while at the high laser fluences (above 1 J/cm^2) the etching increases approximately linearly with laser fluence (~ 20 nm per pulse). For longer laser pulses (200 ns) the etching mechanism did not change over the applied fluence range (0.8-1.6 J/cm^2) and clearly involves the formation of a thin carbon film at the hot quartz surface. Furthermore, the etch rate is lower when using the longer laser pulses indicating that etching with longer laser pulses (~100 ns) is more suitable for the fabrication of optical components in UV transparent materials, particularly since the high energy provided by these lasers can etch large optical component in a single shot. Finally, we have demonstrated that this approach for quartz micro-lens fabrication by LIBWE is an effective method of developing homogenizers which can dramatically improved the spatial homogeneity of a laser beam.

Acknowledgment

The authors would like to acknowledge discussions with Dr. Donagh O'Mahony and his help in preparing the manuscript.

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