Laser-induced backside wet etching of silica glass with ns-pulsed DPSS UV laser at the repetition rate of 40 kHz

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Abstract. Surface micro-structuring of silica glass plates was performed by using laser-induced backside wet etching (LIBWE) upon irradiation with a single-mode laser beam from a diode-pumped solid-state (DPSS) UV laser with 40 kHz repetition rate at 266 nm. We have succeeded in a well-defined micro-pattern formation without debris and microcrack generation for the laser beam. Bubble dynamics after liquid ablation was monitored by impulse pressure detection with a fast-response piezoelectric pressure gauge.

1. Introduction

Laser-induced micro-structuring of various materials has served as an important technique in surface structuring for optics and optoelectronic devices [1]. In particular, significant attention has been given towards the micro-structuring of silica glass in spite of challenges with low optical absorption and brittleness, because of its widespread use in industry. We have demonstrated a novel one-step method, laser-induced backside wet etching (LIBWE), for micro-structuring surfaces of silica glass plates, involving irradiation with nanosecond-pulsed UV lasers [2-29]. The LIBWE method is based on the deposition of laser energy onto a thin layer at a glass-liquid interface that drives ablation from the absorbing liquid substance. Assuming negligible UV absorption by the silica glass, the incident laser beam passes through the glass plate and excitation of the strongly absorbing dye solution. When the dye solution close to the interface is converted into an activated state with sufficient laser fluence, etching on a surface layer of the silica glass is achieved. The depth of the etch pits increases linearly with the number of laser shots. Typical etch rates of the materials were 0.1 - 40 nm/pulse, depending on irradiation conditions such as laser wavelength, laser fluence, and dye concentrations. Micro-structuring of various transparent materials, such as silica glass [2,5-9,11-20,22,23,27,28], quartz [3,18,21,24-26], glasses [19], calcium fluoride [5,18,25], magnesium fluoride [18], barium fluoride [25], fluorocarbon resin [4], and sapphire [10,18,25,29], have been reported. This method can be employed for the production of random-phase-plates [24], Fresnel lens [25], beam homogenizers (plano-convex microlens array) [26], surface relief gratings [19], imprinting templates [13], and microarrays of dye, protein, and polymer-microbeads [11,12].

In this paper, we report on the fabrication of a line grating pattern on silica glass using the LIBWE method. Laser irradiation from a single-mode diode-pumped solid state (DPSS) laser at 266 nm was delivered with a galvanometer-based point scanning system. The laser beam position and velocity was
precisely controlled across the sample surface with the computer-controlled galvanometer system, facilitating flexible beam delivery suitable for rapid prototyping processing according to electronic design data in the computer. Additionally, to monitor the shock wave formation, transient pressure signals caused by laser ablation of toluene liquid were measured with a pressure gauge.

2. Experimental

The fourth harmonic wavelength of an Nd:YVO₄ laser ($\lambda = 266$ nm, full width at half maximum (FWHM) 30 ns, $M^2 < 1.3$) was used as a light source at ambient conditions. The laser was operated at a repetition rate of 40 kHz. The laser beam with ~5 μm diameter laser spot at the target surface was scanned with galvanometer-based point scanning module (GSI Lumonics, HPM10M2), as shown in figure 1. A fused silica glass plate with a thickness of about 0.5 mm was used as a sample. Toluene (Wako Pure Chemical Industries Ltd., S grade) was used without further purification. At the wavelength of 266 nm, the penetration depth, $d$, in pure toluene solution was estimated to be 6.1 μm for single photon absorption [30]. The morphology of etched patterns was analyzed using a confocal scanning laser microscope (Keyence, VK-8500) and scanning electron microscope (Keyence, VE-7800).

Transient pressure was directly measured with a fast-response piezoelectric pressure gauge (Dr. Müller Ingenieurtechnik, Müller-Platte-Gauge, Germany) specifically designed for increased tolerance for use in toluene [16]. The distance between the sensor and glass surface was set at 0.5 mm. The signals were recorded by a digital oscilloscope that allows fast digitizing of long-duration events (LeCroy 9310AL).

![Figure 1. Experimental setup for LIBWE of (a) the galvanometer-based point scanning system and (b) the arrangement for pressure measurement.](image)

3. Results and Discussion

3.1. Micro-structuring of silica glass with galvanometer scanning system

Well-defined etching grooves with about 5 μm in width, which was free of debris and microcracks around the etch area, were fabricated by UV laser irradiation using a single-mode laser beam, are shown in figure 2. The laser irradiation was carried out at laser energy of 1.0 μJ/pulse. The laser beam was scanned on the sample surface at the rate of 100 mm/s. To produce a deep groove structure with a high aspect ratio, multiple scans were repeated up to 150 times across the line position. The etch depth increased with increase in the scan number.

On the other hand, when a tightly focused beam of the single-mode UV laser was incident at a glass-air interface without the liquid, a well-defined etching pit with 10-μm diameter was formed on the silica glass by conventional ablation [31]. However, upon the irradiation of several laser shots accumulated at the same position to make a deep pit structure on the surface, cracks were randomly formed around the etched area, indicating that only single shot irradiation is available for well-defined micro-structuring by this conventional ablation condition. As LIBWE process enables one to accumulate laser pulses at the same position for making a deep hole in the material without crack formation.
formation, the use of LIBWE allows greater flexibility in the types of processing and the geometries of the structures available, such as formation of micro-channels, gratings and deep pits.

**Figure 2.** Cross-sectional SEM images (a,b) and surface optical micrograph (c) of gratings in silica glass etched by LIBWE with 150 scans at 40 kHz and 1.0 μJ/pulse.

3.2. Transient pressure measurement of toluene ablation

According to our previous observation of the transient optical images in the laser ablation of toluene liquid at room temperature monitored by time-resolved shadowgraph technique, shockwave propagation and vapour expansion, which were induced by the explosive vaporization of the liquid ablation, were formed near the interface between silica glass and toluene liquid [9,15,16]. In the present study by DPSS UV laser irradiation at a high repetition rate, the vapour formation might be adversely affected because the period for the expansion and contraction of a vapour bubble was on the order of microsecond.

**Figure 3.** Transient pressure profile of toluene ablation by DPSS UV laser irradiation at repetition rate of 40 kHz with 1.0 μJ/pulse: (a) beginning stage of the ablation, signal impulses marked with “E” are due to vapour expansion and those with “C” are due to bubble collapse; (b) array of pressure signal recordings in 45 ms windows with pulse number increasing left to right and time evolving top to bottom in 25 μs steps. Coloured (darker) spots correspond to an impulse of pressure signals.

Figure 3 shows transient pressure profiles during the DPSS laser irradiation with 1.0 μJ/pulse at 40 kHz. Pair of impulses corresponding to shock wave formation by vapor expansion and bubble collapse was clearly observed in figure 3a [16]. The time duration of the pair is approximately 20 μs. The pairs of the signals were also observed distinctly if the total irradiation period was expanded to 45 ms, in which 1,800 laser shots were involved. All of the pressure signals in 45 ms are illustrated as a three-dimensional mapping in figure 3b, where the Y-axis of time proceeds from top to bottom and X-axis presents a series of laser shots at 40 kHz. The time window for each frame in figure 3b starts at when a laser irradiation pulse strikes the target and evolves for 45 ms. Colored (dark) spots in the figure correspond to an impulse of pressure signals caused by bubble expansion and collapse.
time duration of the pairs in the whole time region is dominantly observed in the range of 15-25 μs. The observation suggests that LIBWE is possible by the DPSS UV laser irradiation at a high repetition rate of several tens of kHz.

4. Summary
We have demonstrated that well-defined micropatterns on a silica glass plate can be fabricated by LIBWE method using galvanometer-based point scanning of DPSS UV laser. The point scanning system at a high repetition rate is suitable for a flexible rapid prototyping as a mask-less exposure system. The system could be easily utilized in mass production. Although transient shockwaves and micro-bubbles induced by laser ablation of the liquid expanded to the surroundings in the nano- and micro-second timescale upon the laser irradiation at a repetition rate of 40 kHz, deep micro-trenches were successfully generated on the glass surfaces without any crack or debris formation. These results indicate that highly energetic states or species that can etch a silica glass surface possess a short lifetime, with minimal influence outside the etched areas.

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