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Indirect laser etching of fused silica: Towards high etching rate processing

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Abstract

The indirect laser processing approach (LIBWE) laser-induced backside wet etching allows defined microstructuring of transparent materials at low laser fluences with high quality. The optical and the thermal properties of the solid/liquid interface determine the temperatures and therefore the etching mechanism in conjunction with the dynamic processes at the interface due to the fast heating/cooling rates. The exploration of organic liquid solvents and solutions such as 0.5 M pyrene/toluene results in low etch rates (~20 nm/pulse). By means of liquid metals as absorber here, demonstrated for gallium (Ga), etch rates up to 600 nm/pulse can be achieved. Regardless of the high etch rates a still smooth surface similar to etching with organic liquid solutions can be observed. A comparative study of the two kinds of absorbing liquids, organic and metallic, investigates the etch rates regarding the fluence and pulse quantity. Thereby, the effect of incubation processes as result of surface modification on the etching is discussed. In contrast to pyrene/toluene solution the metallic absorber cannot decompose and consequently no decomposition products can alter the solid/liquid interface to enhance the absorption for the laser radiation. Hence, incubation can be neglected in the case of the silica/gallium interface so that this system is a suitable model to investigate the primary processes of LIBWE. To prove the proposed thermal etch mechanism an analytical temperature model based on a solution of the heat equation is derived for laser absorption at the silica/gallium interface.

Keywords: Laser; Fused silica; LIBWE; Etching; Transparent material; Gallium; Organic liquid; Etch mechanism; Surface modification; Solid/liquid interface

1. Introduction

During the past decade many investigations have been carried out of indirect or hybrid laser processing techniques for micromachining transparent materials [1–5]. Some methods make use of interaction processes of laser irradiated absorbing additives with the backside surface. One representative of hybrid processing is (LIBWE) laser-induced backside wet etching [3,6] which makes use of organic liquids as absorbing additives. The method is characterized by low threshold fluences, etch rates in the nanometer range, and minimal, nearly optical roughness at optimized experimental conditions. These attributes make LIBWE to a promising candidate for high-quality laser processing in microsystem technology.

The etch rates in dependence on the processing parameters and the properties of the etched surface are studied extensively [3,6–11]. Furthermore, the application potential of LIBWE is very high as various studies show that demonstrate high quality patterning of surfaces in the submicron range, etching of refractive and diffractive patterns, scribing deep trenches, and patterning for live science applications [9,12–17].

The etch mechanism actually under discussion is suggested as a sequence of surface heating by the hot laser-heated liquid, an increasing of the solid temperature up to a critical value (e.g. the softening or melting point) followed by mechanical removing of the softened or melted surface layer by the heated liquid together with shock waves on high pressure [3,8,9,11]. The etching is accompanied regularly by incubation effects [10,18] similar to laser ablation on air [19] with the results of instabilities of the etching at low fluences and pulse numbers, the increase of the etch rate during prolonged pulsed laser irradiation, and the reduction of the threshold fluence for etching with the number of applied laser pulses. The incubation processes are accomplished by alterations of the surface, e.g., defect generation and chemical surface modification that influence the laser energy deposition in the near surface region. At LIBWE with hydrocarbon solutions one origin for the surface modification and defect generation and consequently the incubation effect is the decomposition of the organic molecules. Due to the high laser power initialized pressure-, temperature-, and photon-induced fragmentation processes [20–22] the formation of a carbon modified layer [6,7,9,23–25]

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is caused that changes the physical/optical/chemical properties of the solid/liquid interface.

In this work the LIBWE process by means of metallic liquid gallium is compared with that using organic solution 0.5 M pyrene/toluene. The etch rate behavior in dependence on the laser fluence and pulse number as well as the surface quality of etch pits as criteria is discussed. A temperature model based on an analytical solution of the heat equation is derived from the silica/gallium interface and is proved to the experiment.

2. Experimental

The experimental set-up for laser-induced backside wet etching is described elsewhere [3,6]. A KrF excimer laser ($\lambda = 248$ nm, 10 Hz, 20 ns) incorporated into a laser workstation [6] was used for the experiments. The laser-processing chamber was attached to a computer controlled *x–y–z* stage. A reflective objective (Schwarzschild, 15× demagnification) with an optical resolution of 1.5 μm was used for projecting a square aperture with a size of 100 μm × 100 μm onto the sample backside. The samples were processed with a fixed set of laser fluences and pulse quantities. In all investigations fused silica samples cut from double side polished wafer with a thickness of about 380 μm and low surface roughness (<0.3 nm rms) were used as received without additional cleaning. A solution of pyrene with a concentration of 0.5 M (mol/l) solved in toluene as well as pure gallium (Ga) were used as liquid absorber in comparison. After the etching process the samples from experiments with liquid metal were cleaned from the gallium first mechanically with a soft tissue and after that by selective etching the gallium residues with diluted hydrochloric acid. Finally all the samples were cleaned ultrasonically with distilled water and acetone. The depths of the etched pits were measured with a white light interference microscope (50× magnification). Secondary electron microscopy (SEM) and electron probe microanalysis (EPMA) were performed after deposition of a thin gold film to the samples surface to study the topography and the chemical composition of the near surface region.

3. Results

The laser fluence has the most important effect on the etch rate and the etched surface quality using either organic solution or liquid metal as absorbing liquid. In Fig. 1 the etch rate over a wide and similar fluence range for a 0.5 M solution of pyrene/toluene (a) and gallium (b) is presented in comparison. The etch rate per laser pulse was determined from the measured final etch depth of etch pits.

The determined etch rate behavior with different fluence regions as depicted in the graph in Fig. 1a is characteristic for LIBWE with organic solutions [6,9,10]. The experimental threshold fluence – the lowest fluence at which a measurable etching occurs – after applying 300 pulses is determined to be at 0.34 J/cm^2 which is more than one magnitude less than for direct laser ablation of fused silica on air being at more than 10 J/cm^2 [26]. The separation into the fluence regions was carried out by concerning the slopes of the etch rate and different surface roughness [9,10] and comprises (i) low fluence range (<0.55 J/cm^2, see inset in Fig. 1a), (ii) middle fluence range (0.5–1.3 J/cm^2), and (iii) high fluence range (>1.3 J/cm^2). Strong incubation effects characterize region (i) causing nonlinear etch rate growth and an enhanced surface roughness (see SEM image in Fig. 2a) while in region (ii) the etch rate increases almost linearly from 7 to 30 nm/pulse representing a slope of 25 nm/(J cm^2) and a high surface quality (Fig. 2b). For fluences in region (iii) the etching is characterized by high rates (up to 400 nm/pulse), a steeper etch rate slope of 60 nm/(J cm^2), and a jump in roughness due to the occurrence of surface features related to rapid resolidification after melting (Fig. 2c) that will be an indication for a changed etch mechanism compared with the gentle-etch behavior in the middle fluence range.

The etch rate behavior by means of liquid gallium is depicted in the graph in Fig. 1b. The experimental threshold fluence of about 1.3 J/cm^2 is comparatively high in comparison to LIBWE with hydrocarbon solution (Fig. 1b) but significantly lower than for laser ablation on air. The reflectivity of about 80% and the 300-time higher thermal conductivity of the metallic gallium compared to toluene are probably the main

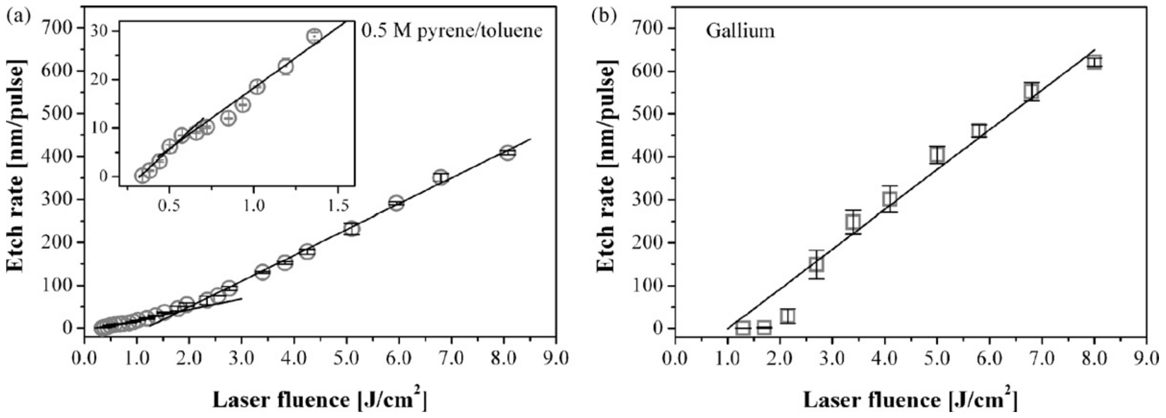

Fig. 1. Etch rate in dependence on the laser fluence in a wide fluence range by means of (a) 0.5 M pyrene/toluene solution and (b) gallium as absorbing organic and metallic liquid, respectively. Additionally, the respective linear fits are included concerning different fluence ranges.