Applied Surface Science 302 (2014) 42–45

Contents lists available at ScienceDirect

Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

Reducing the incubation effects for rear side laser etching of fused silica

Klaus Zimmer[a,*], Martin Ehrhardt[a], Pierre Lorenz[a], Xi Wang[b], Csaba Vass[c], Tamás Csizmadia[c], Béla Hopp[c]

[a] Leibniz-Institute of Surface Modification, Permoserstr. 15, 04318 Leipzig, Germany
[b] Nanjing University of Science & Technology, Xiaolingwei 200, 210094 Nanjing, China
[c] Department of Optics and Quantum Electronics, University of Szeged, Dóm tér 9, 6720 Szeged, Hungary

ARTICLE INFO

Article history:
Received 17 June 2013
Received in revised form
25 December 2013
Accepted 20 January 2014
Available online 29 January 2014

Keywords:
Laser etching
Incubation effect
LIBWE
LIBDE
Fused silica

ABSTRACT

Laser-induced back side wet and dry etching (LIBWE and LIBDE) were developed for precise etching of transparent materials. However, LIBWE and LIBDE feature characteristics such as incubation effects and etching depth saturation, respectively, that can be unfavourable for applications in ultra-precision machining. Therefore, the techniques of LIBDE and LIBWE were combined in such a manner that the dielectric material was supplied with a thin-film-modified surface before etching by LIBWE.

With this goal fused silica samples were covered with a thin chromium film for surface modification before LIBWE etching in acetone with 25 ns KrF excimer laser pulses. Etching with the first pulse was observed. At laser fluences adequate for hydrocarbon LIBWE (1 J/cm^2) the etching rate for LIBDE is much higher than for LIBWE. In consequence, the etching rate decreases with increasing pulse number up to ten. The surface morphology depends very strong on the laser fluence and the pulse number. Smooth etchings were achieved at high fluences and low pulse numbers. However, uneven, wavy etched surfaces and micron pattern formation were observed for moderate and low laser fluences.

1. Introduction

The rear side etching of dielectric materials with laser radiation by means of additional absorbing materials like liquids, solids, or vapours offers benefits for precise patterning [1,2]. According to the used absorber they are called laser-induced back side wet etching (LIBWE) [3], laser-induced back side dry etching (LIBDE) [4], and laser etching at a surface-adsorbed layer (LESAL) [5]. Each method shows specific characteristics but none of them combines all advantages required for ultra-precision engineering. Such undesired features are incubation effects, etching depth saturation, or remaining surface modifications that make etching of smooth surfaces with a specific depth profile difficult.

Laser-induced back side wet etching (LIBWE) [3] allows the precise patterning of different dielectric materials in consequence of the comparable low etching rate and the observed low surface roughness. Optimized etching conditions enable precise etchings for a particular material up to a predefined depth but needs to consider incubation effects that are linked to the etching mechanism [2,6]. Material modifications due to laser etching with liquid hydrocarbon absorbers are the origin of the incubation at LIBWE [2]. The utilization of liquid metals for LIBWE as well as the application of thin metal films for laser-induced back side dry etching [3], however, results in etching without such pronounced incubation effects. In difference to LIBWE, where the etching depth increases almost linearly with the pulse number after incubation, the etching depth at LIBDE is self-limiting due to the removal of the absorber at etching. Back side etching is feasible for liquid and solid tin within the same material system [7]. However, the etching rates for liquid metals are much higher compared to hydrocarbon absorbers as shown and discussed in Ref. [8].

The mechanisms of back side etching techniques are rather complicated and a number of processes are involved [2,3,6]. The strong absorption near the rear side of the transparent material by the liquid [3] as well as by highly absorbing material modifications of the dielectric material in a shallow near-surface region induces high temperatures near the solid surface [2]. This fast heating above the material's boiling temperatures induces processes like explosive evaporation, high pressures, and material decomposition. However, a number of laser-induced processes due to the rapid heating of the liquid have to be considered for LIBWE, e.g., explosive evaporation, vapour and cavity bubble formation and collapse, and transient pressure generation [9]. For LIBWE regularly absorbing hydrocarbon liquids were used that decompose during laser irradiation and result in chemical surface modifications [10].

---

* Corresponding author. Tel.: +49 341 235 3287; fax: +49 341 235 2584.
E-mail addresses: klaus.zimmer@iom-leipzig.de, zimmer@rz.uni-leipzig.de (K. Zimmer).

0169-4332/$ – see front matter © 2014 Elsevier B.V. All rights reserved.
http://dx.doi.org/10.1016/j.apsusc.2014.01.115

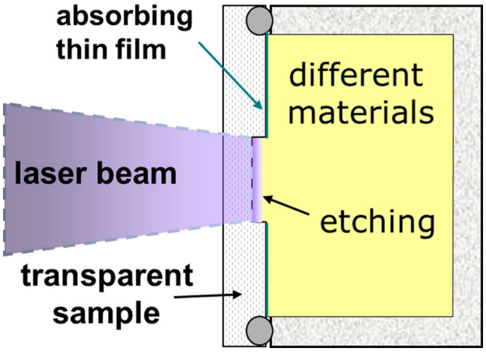

Fig. 1. Sketch of the experimental set-up of the studies for LIBWE-LIBDE combination. In any case a 30 nm chromium-covered fused silica sample was used for back side etching in contact with air, water, and acetone, respectively.

These processes are substantially influenced by the confinement of the laser-matter-interaction at back side etching; for instance LIBDE with non-absorbing liquids was not examined yet.

This study focuses on the combination of LIBWE and LIBDE processes for the further exploration of the involved processes in back side etching and with the aim of reducing the incubation effects but maintaining a constant etching rate. Hence, this combination is called LIBDWE. The intended substantial reduction of incubation effects shall allow a precise predetermination of the etching depth for a given set of etching parameters enabling nanometre depth precision patterning. The known experimental arrangements and laser irradiation configurations for LIBDE and LIBWE [3,11] are combined in this manner that a dielectric sample featuring a surface modification by a sputtered film is used for etching in LIBWE configuration, hence using an absorbing liquid. Here a thin metal film is used for modifying the non-absorbing dielectric surface providing the near-surface laser absorption that is essential for the etching process. Hence, the conditions for laser back side etching are provided for the first pulse by the thin-film-modified surface whereas the etching can be maintained by hydrocarbon liquid applied to the back side, too.

1.1. Experimental and materials

For the experiments double-sided polished fused silica wafers were used without extensive cleaning. The rear side surface was covered with a 30 nm thick chromium film by DC sputtering. The basic experimental set-up for back side etching has already been discussed extensively [2–5]. For all studies the same chamber was used that contains either air, or water, or acetone, see Fig. 1. According to the used substance applied to the rear side of the sample we get the typical configuration of LIBDE, confined LIBDE (cLIBDE), and LIBDWE for air, water, and acetone, respectively. The cLIBDE studies can provide some information on the heat conduction and the confinement of the secondary processes like material evaporation due to the liquid at the sample's back side.

In the experiments a KrF excimer laser beam ($t_p \sim 20$ ns, $\lambda = 248$ nm), that was homogenized to provide an overall energy deviation in the mask plane of below 5% rms, was used for illumination of a square mask with adjustable size (typically 100 μm × 100 μm) that was projected with a reflective objective (×15 demagnification) to the sample rear side. The chamber holding the samples was mounted on top of a stage system that enables computer-controlled positioning and scanning of the laser spot across the sample surface.

The extinction of the 30 nm chromium films on the fused silica samples was measured by UV/VIS spectroscopy. From that the absorption of the thin films was calculated considering the thin-film reflectivity, which can be obtained with the tabulated refractive indices [12], to be 57%. The absorption coefficient of the sputtered films ($\alpha_{Cr}(248\,\text{nm}) = 22.7\,\mu\text{m}^{-1}$) is below the tabulated values but the deviations are related to surface oxidation and film imperfections.

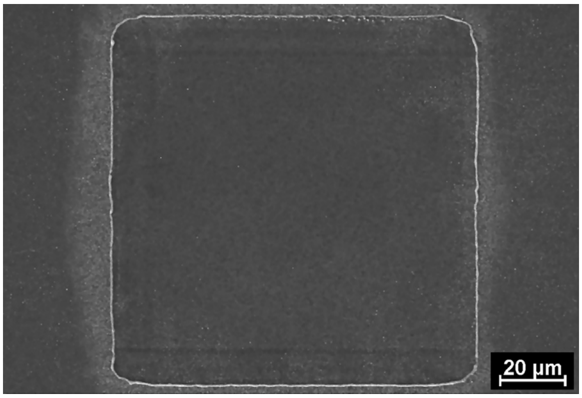

Fig. 2. SEM image of an etching pit achieved with the LIBDWE configuration using a 30 nm chromium film ($F = 3100$ mJ/cm^2, $N = 2$). Some debris and a rim of molten chromium are visible.

After etching the etching depth was measured by with light interference microscopy (WLIM) and the surface morphology was imaged by secondary electron microscopy (SEM) and reflected light optical microscopy (RLOM).

2. Results and discussion

Etching with the first laser pulse was achieved with all three substances (air, water, and acetone) applied to the back side accomplishing the LIBDE, cLIBDE, and the LIBDWE configuration. An etching spot produced by LIBDWE with chromium film is shown in Fig. 2. At these conditions the spot edges are well defined and an almost flat etched surface is found. The metal film was melted at the laser spot edges and forms there a small rim. This was observed for all confining substances. The particulates near the etching pits are debris composed from the chromium film, the etched fused silica substrate, and decomposition products of the hydrocarbon liquid. The most experiments were done within a fluence range of 500–1500 mJ/cm^2 applying pulse numbers of up to 10 due to the final aims of reducing incubation effects, maintaining the etching with high quality and the fact that the optimal laser fluence for high-quality LIBWE is within a narrow fluence range [2,3].

The etching depth was measured in dependence on the laser fluence and the pulse number for different back side substances to study the etching mechanism for the combination of laser-induced back side dry and wet etching. In Fig. 3 the etching depth (d) and the etching rate (Ra) are plotted in dependence on the laser fluence for LIBDE, confined LIBDE, and LIBDWE, respectively.

All three techniques have a different etching behaviour with respect to the threshold fluence and the slopes of the etching rate. The threshold fluences for LIBDE and LIBDWE are near 250 mJ/cm^2 whereas for cLIBDE 500 mJ/cm^2 can be estimated.

The etching rate for LIBDE and cLIBDE is calculated from the depth of the first pulse whereas for LIBDWE the rate was calculated without considering the first (LIBDE) pulse. In this way the etching rates of both processes (LIBDE, LIBWE) were distinguished to find constant rate conditions. Close to the optimal laser fluence near 1000 mJ/cm^2 the etching rate for LIBDE and cLIBDE is much higher compared to the averaged etching rate for LIBDWE. Similar etching rates for LIBDE/LIBWE were found near the etching threshold but in this fluence range regularly rough surfaces were found. Hence, low fluences are not suitable for LIBDWE.