Precision glass microstructure fabrication using ultrafast laser induced chemical etching

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Abstract

Femtosecond laser irradiation followed by chemical etching (FLICE) is a novel micro-fabrication technique that allows versatile production of 3D hollow structures, however the use of the most common etchant, hydrofluoric acid (HF), is problematic since it is extremely hazardous. We have utilized an alternative technique that employs a less hazardous etchant, potassium hydroxide (KOH), and have examined the laser processing parameters for etched high-aspect microfluidic tunnels, micro- and macro- surface structures in-bulk of fused silica substrates. The impact of etching temperature is investigated suggesting an applicability of an effective and HF-free FLICE process fulfilling the need for etch rate or etch selectivity.

1. Introduction

Irradiation of a tightly focused femtosecond laser beam inside fused silica causes material modification that may boost its etch ability [1]. Based on this phenomenon, femtosecond laser irradiation followed by chemical etching (FLICE) had been established to enable versatile 3D fabrication of microfluidic structures via selective etching [2]. Hydrofluoric (HF) acid etching of silicate glass is widely used but problematic: etching selectivity is not ideal as raw glass is etched at several μm/h [4]; HF depletes as a tunnel elongates up to several mms [4]; HF is a highly hazardous substance. As a replacement, potassium hydroxide (KOH) offers a higher etching selectivity, an almost constant etch rate regardless of tunnel elongation with reduced toxicity [3]. This paper presents the FLICE method using KOH aqueous solution to fabricate high-aspect microfluidic tunnels on fused silica substrates. Moreover, for the first time to our knowledge, we propose a method to fabricate micro- and macro-surface structures using KOH under a higher etching temperature.

2. Experimental

Two lasers were used to process UV fused silica plates with their parameters listed in Table 1. Beams were circularly polarised and focused by an objective (f=12.7 mm) 150 µm below the top surface. Single straight lines were written with independently controlled pulse energy, repetition rate and translation speed to obtain different exposure levels. Samples were then ground at the edges to expose the irradiated zones, and etched in 10M KOH aqueous solution.

Table 1: Parameters of laser systems

Laser model	Wavelength	Duration	Repetition rate	Beam ϕ	M^2
Spectra-physics Hurricane *i*	800 nm	130 fs	1-5 kHz	5 mm	2
Amplitude Systèmes Satsuma	1030 nm	280 fs	1-2000 kHz	2.2 mm	1.1

3. Results and discussion

Using the Hurricane *i* laser, a laser processing window was characterized with pulse energy from 1.08 to 3.60 µJ (or pulse peak fluence of 10 to 34 J/cm^2) and linear translation speed from 0.1 to 1.5 mm/s at a fixed repetition rate of 5 kHz. The etching temperature was set to 80 °C and the sample was rinsed, dried and measured hourly. Figure 1 (a) and (b) illustrate lengths and image of tunnels after etching for 4 hours. Among the pulse energy and speed settings, (2.52 µJ, 0.75 mm/s) was considered optimal as it balances etch rate, etch repeatability and avoidance of undesired damage. The enhanced etch rate of photo-modified SiO_2 under this condition was 200 µm/h, guaranteeing a selectivity of 450:1 given that the etch rate of raw SiO_2 is 0.44 µm/h [5]. The conical shape that comes along with HF etching is hence significantly reduced by using KOH. A tunnel aspect ratio of 75:1 was achieved.

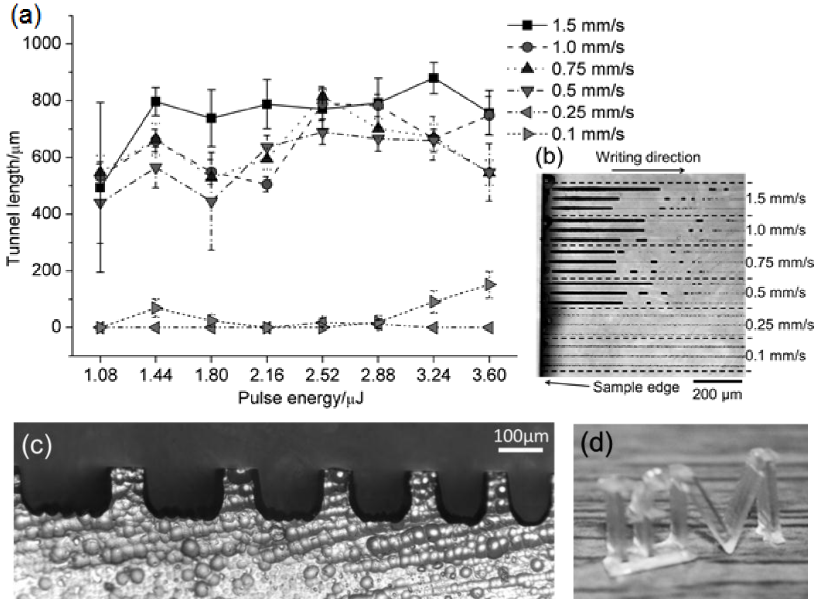

Figure 1: (a) Tunnel lengths subject to pulse energy and processing speed. (b) An overhead view of tunnels in groups of three processed with 2.52 µJ pulses. Transparent zones along tunnels contained residual rinsing water. (c) A side view of the channels' profile after etching. (d) A glass logo of Institute for Manufacturing (9.8×4.8×1.8 mm^3) fabricated with FLICE.

Using the Satsuma laser, six groups of lines were written with 3.4 µJ and 1.0 mm/s at 125 kHz. Each group consisted of 5 lines separated by 5-30 µm. Surface channels (Figure 1 (c)) about 100 µm deep across the 10 mm long glass plate were etched with KOH solution at 120 °C for 220 min. It was observed that the plate thinned by 70 µm, suggesting an etch rate of raw glass of 9.5 µm/h which is comparable to that of HF. An average bottom surface roughness Ra= (5.68 ± 0.59) µm was obtained using a white light interferometer. As a demonstration an Institute for Manufacturing logo (Figure 1 (d)) was also made by firstly layering contours at a step of 90 µm, then etched for 2 hours under 120 °C to expose the pattern, plus another 40 hours under 80 °C to avoid undesired etching of raw material.

From these results we conclude that the SiO_2 processed with Hurricane *i* obtained a considerably selective etchability to permit the formation of tunnels via etching with

KOH solution at 80 °C. Meanwhile, the SiO_2 processed with Satsuma was not sufficiently active to allow etching in horizontal plane. The etching selectivity in this case was merely several to one but still sufficient to allow the formation of channels. The etching of photo-modified volume happened in the first place in vertical direction after the raw SiO_2 above it was rapidly etched away using 120 °C KOH solution.

4. Conclusions

We have utilised an alternative FLICE technique that employs KOH as etchant, and have examined the experimental procedure to fabricate straight in-bulk tunnels, surface channels and macroscopic features on fused silica substrates under different etching conditions. The requirement for etch selectivity (450:1) or raw glass removal rate (9.5 μm/h) may be fulfilled by different laser parameters and controlling the etching temperature at 80 and 120 °C, suggesting the feasibility of an effective and HF-free FLICE process. We hope this technique could offer an advantageous and environmental friendly fabrication process for microfluidic lab-on-chip devices.

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