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MONOLITHIC THREE-DIMENSIONAL INTEGRATION OF MICRO-FLUIDIC CHANNELS AND OPTICAL WAVEGUIDES IN FUSED SILICA

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ABSTRACT

This paper presents dramatic improvements in the micro-fabrication of three-dimensional microfluidic channels and high-aspect ratio tunnels within the bulk of a fused silica substrate. We also report the fabrication of optical waveguides within the same substrate, which is a major step towards the integration of sensing capabilities within microfluidic networks.

This integrated device, which combines both fluidic channels and optical waveguides, opens new opportunities in bio- and chemical sensing. The flexibility of the improved manufacturing process offers substantial new design capabilities, especially for single channel probing and massively parallel processing and sensing.

INTRODUCTION

For the past decade, Lab-on-a-Chips and Biochips have gained increasing attention as a way to obtain faster, more reliable and more efficient chemical analysis. Fluidic channels are an essential component of these systems, playing a role equivalent to that of data-buses in computers.

In this paper, we propose a new approach to manufacture high-aspect ratio microfluidic channels and tunnels in fused silica. The microfabrication process is carried out in two steps: (i) a predefined pattern is written with femtosecond laser pulses inside the volume (bulk) of fused silica substrate glass. (ii) This is followed by an etching step in a low-concentration (typ. 5%) aqueous solution of HF acid. Kondo *et al.* [1] have first proposed a related process for photo-etchable glass. However this material processing is more complex, requiring an additional heat treatment to precipitate crystallites before the etching step. Marcinkevičius *et al.* [2] described a similar process to manufacture micro-holes in fused silica. In these pioneering works, the microfluidic tunnel cross-section geometry is equivalent to the laser beam shape. As a result, only very small structures (the typical cross-section was a few microns) have been obtained. This is often undesirable for micro-fluidics applications.

Our approach solves this problem. We use a scanning process (referred as “painting” [3]) to create fluidics channels and tunnels of virtually any shape. We have also explored the opportunity of combining optical waveguides and fluidic channels with the same manufacturing process.

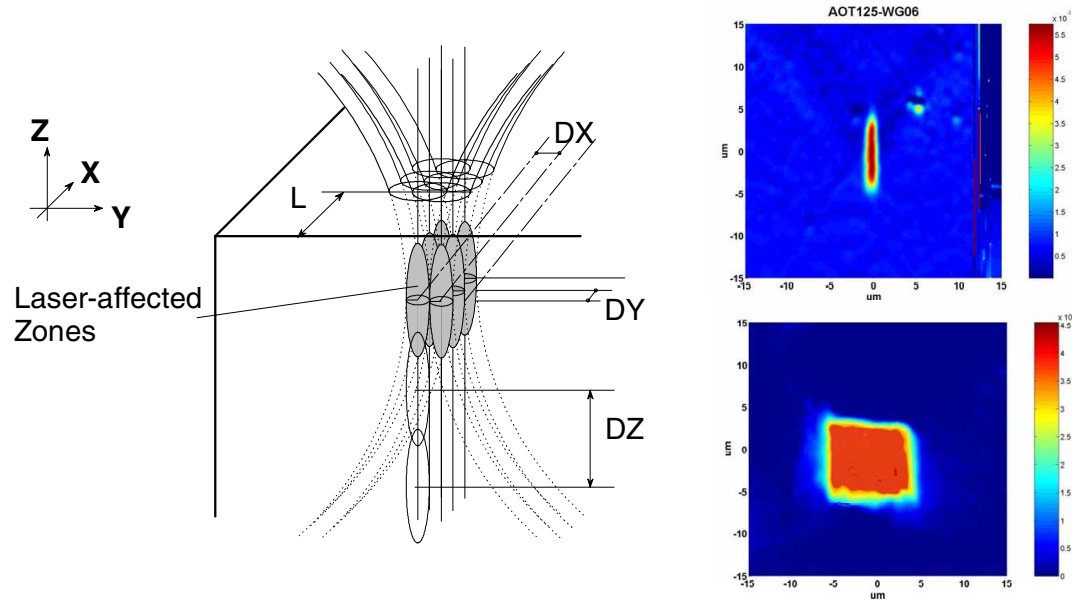


Figure 1. Illustration of the painting process [5]: by moving the substrate in three directions, one can connect laser-affected zones, thus forming large volumetric patterns. The pictures on the right show two optical refractive index maps: the top is produced with a single exposure while the bottom one is produced by “painting” multiple lines enough close to each other such that the resulting affected zone has a nearly continuous structure.

EXPERIMENTAL METHOD

When fused silica is exposed to femtosecond laser pulses, the index of refraction is locally modified [4] along an ellipsoid whose volume is defined by the optical system and by the laser parameters (beam size, energy). To obtain a more symmetrical cross-section, a process called “painting” was developed. It consists of juxtaposing multiple laser-affected zones. This approach can be used to generate complex shapes, as shown on Figure 1. We have used this process to define tunnels or channels. A two-step procedure was applied:

1. **Laser exposure:** the desired pattern of a tunnel or channel is applied to the glass by painting numerous adjacent lines. To do so, the substrate is mounted on translation stages with a typical repeatability of 300 nm and a resolution of 100 nm. The sample is exposed to femtosecond laser pulses emitted from a Ti:Sapphire laser operating at 800 nm. The pulse width is 100-fs at a repetition rate of 250 kHz.
2. **Etching:** After laser illumination, the glass is immersed in a low-concentration aqueous solution of HF acid for three hours. For these experiments, the solution is kept at ambient temperature. The volume of the solution is typically 40 mL (or at least ten times the volume of the glass specimen). After etching, specimens are rinsed in de-ionized water and cleaned in an ultrasonic bath made of isopropanol-3 solution for about 5 min. This is followed by a final rinsing and drying.

High purity fused silica substrates were used. Fused silica exhibits good optical properties from the UV to the IR, as well as excellent chemical stability. These attributes make it a very attractive material for Lab-on-a-Chip applications.

A Nikon (Measuring Microscope MM4, Quadra-check 2000) optical microscope equipped with high precision motorized stages was used to visually inspect and measure the profile of tunnels and surface channels. Etched zones are clearly visible and have a characteristic opaque to dark color in bright field and are shiny in dark field. An SEM (JEOL 840) was also used to inspect specimens at higher magnification.

RESULTS

Tunnels

Fused silica is transparent at the optical wavelength of the laser. The femtosecond laser-matter interaction occurs through a multi-photon process at the focal point. Tunnels were obtained by painting lines deep below the glass surface. When immersed in HF aqueous solution, it was observed that the etching rate is several times faster in the region where the material has been laser exposed than in non-illuminated regions. Table I reports two illustrative examples of parameters that were used to create tunnels. These tunnels were made in the same piece of glass (about 25 x 12 x 12 mm). Each tunnel has seen exactly the same procedure in term of sample preparation and chemical etching.

Table I. Two sets of parameters used to fabricate microtunnels.

Tunnel		A (Fig. 2)	B (Fig. 3)
Painting parameters	Distance below surface (μm)	75	420
	Width (μm)	30	30
	Laser Power (mW)	50	73
	Length (mm)	5	5

Figure 2 shows the result obtained for the tunnel A (see Table I). After three hours of etching, the tunnel is about 70 microns wide and 1.2 mm long. The laser painted cross-section consists of 10 lines with a spacing of 3 microns. The writing speed was 500 microns/second. Remarkably, the lines are clearly visible under the optical microscope. This suggests that the volume obtained by painting is not homogeneous regarding etching rate. The optical microscope picture in Figure 2 also suggests that the material in between lines is less easily etched than the one that was in the middle of the spot.

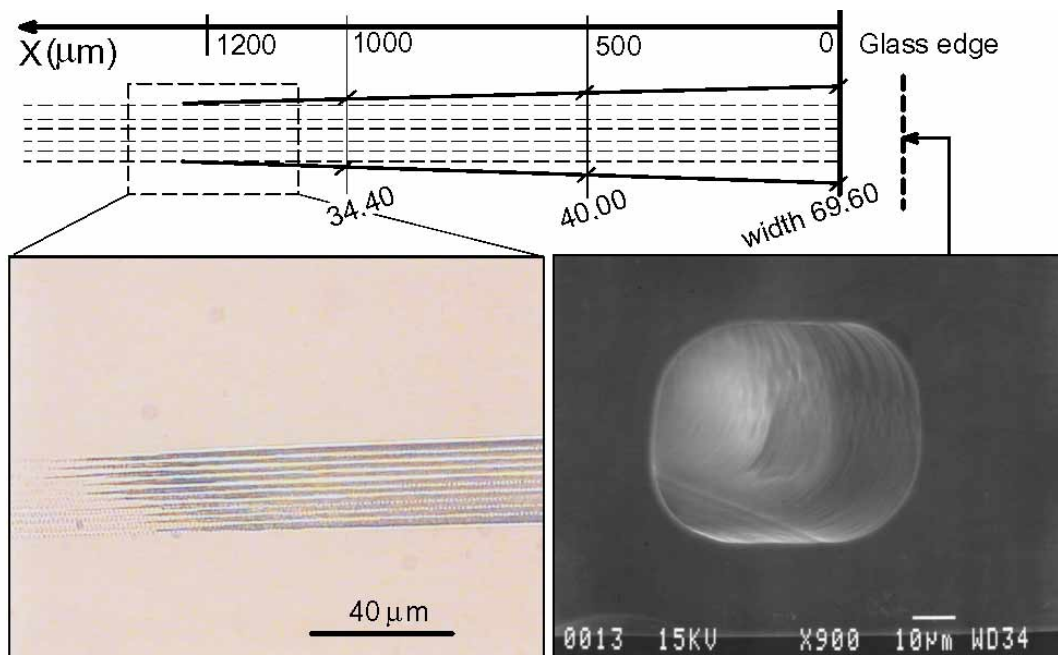


Figure 2. Tunnel A.: optical and scanning electron microscopes observations.

The profile of the tunnel was measured at three different points (0, 0.5 and 1 mm). The data is reported in Figure 2. The tunnel has a conical shape. However, despite a long etching time (3h), the angle is typically 2.7 deg between 0 and 0.5 mm and 1.8 deg between 0.5 to 1 mm (these values are an average from 24 tunnels). This rather small angle suggests that at this concentration, the etching rate along the laser-affected zone is significantly higher than the normal etching rate of the glass.

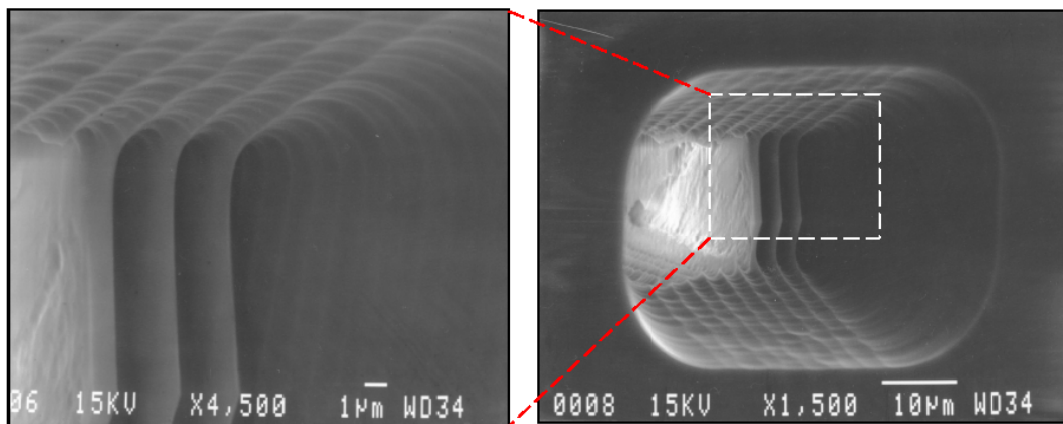


Figure 3. Tunnel B. SEM pictures of the tunnel entrance. The tunnel is 0.8 mm long and about 70 microns wide at the largest cross-section.

Figure 3 shows tunnel B. It was written deeper below the glass surface than tunnel A (420 microns vs 75 microns). The laser power was adapted to take into account the effect of the glass thickness and the associated optical aberrations. This channel was incompletely etched.

Dramatic structures can be seen:

- There is a conical shape at the entrance of the tunnel. We suspect this is a result of a clipping of the writing laser beam when it reaches the edge of the substrate. This “edge effect” is a function of the depth of the tunnel.
- The painting lines are clearly visible and an interesting structure is revealed on the right side of the SEM picture. This structure tends to confirm the non-homogeneity of transformation of the material induced by the laser writing process. The region corresponding to the center (laser waist) seems to be etched faster than the material found in between lines.
- Noticeable, periodic oscillations in the structure of the glass (typically 1 micron) are also visible in the longitudinal direction of the channel.

Surface channels

Channels were manufactured with a similar specimen and according to the same procedure as the tunnels, with the exception that the laser pattern was on the surface of the fused silica.

Figure 4 (left) shows the time-dependent etching profile of a surface channel under which an optical waveguide was written. The etching profile was obtained by image processing and contour extraction of optical microscope images. The purpose of this test was to demonstrate that it was possible to form optical waveguides and microfluidic channels on a common substrate and that the two features could be processed at the same time. As shown on Figure 4, after 3 hours, the microfluidic channel is fully etched. Normally we would stop the etching process at this point, but for demonstration purposes, the etching process was allowed to continue. After 6 hours, a depression starts to be seen at the bottom of the channel. It is further revealed after 9 hours. This small secondary channel formed at the bottom of the main channel is the optical waveguide that has been etched. This experiment shows that a waveguide can be placed in the immediate vicinity of a microfluidic channel, and that its physical integrity will be preserved with extended time (here for 6 hours).

The right side of Figure 4 shows the etching rate along lateral and transverse directions - as measured on three different channels sharing the same substrate. The channel is almost completely etched after three hours. Etching rate in the longitudinal direction seems to be significantly faster. As shown on Figure 2, 1.2 mm-long tunnels were obtained after three hours of etching, which suggests that the etching rate in the longitudinal direction is in the range of 300 to 400 microns per hour.

CONCLUSION

Femtosecond laser and chemical etching combined with an appropriate patterning process (painting) can be effectively used to micro-manufacture channels as well as tunnels in fused silica. In addition, it is possible to integrate with the microfluidic channels, optical waveguides. These two functionalities are manufactured with the same tool and at the same time. This approach provides the required accuracy necessary for optical sensing. These optical waveguides can be used to probe the fluid flowing inside

the channels or the tunnels. This will pave the road to the development of fully integrated platform that combines fluidics and optical sensing capabilities.

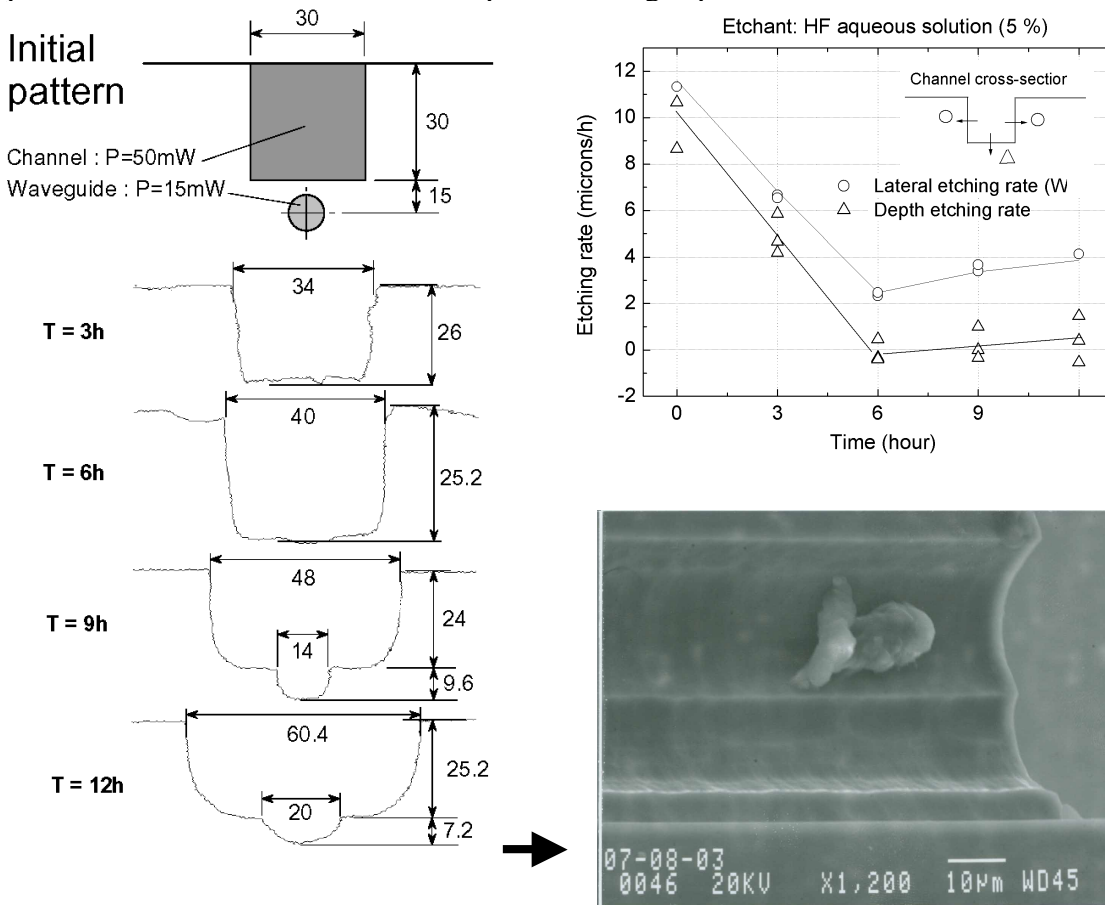


Figure 4. Micro-channels. Left – etched profile progression versus etching time. Right – Time-dependant etching rate in lateral and transverse directions.

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