FABRICATION OF NANO CHANNEL SYSTEMS IN QUARTZ BY LASER-INDUCED SPLITTING

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Abstract

This paper reports a novel laser processing technique for making channels in the nano regime. A Nd:YAG laser has been used to dry fabricate micro channels (25µm-100µm diameter) in a 1cm^3 fused silica substrate by thermal-induced processing. By controlling the locations of these initiating micro channels on a silica cube, a 1D-controllable self-connecting nano fracture can be formed as a rectangular channel. This nano channel is smooth with extremely high aspect ratio (~10^4 depth to width ratio). The length is controlled by the separation of the initiating micro channels. This laser-based nano channel fabrication technique is fast, and inexpensive with potential applications in capillary electrophoresis, and electro-osmosis driven nano-filtration.

1. Introduction

Capillary electrophoresis (CE) has been refined over the past decade and is replacing the conventional gel electrophoresis as a more versatile technology in analyzing proteins and DNA's [1]. Fused silica micro channels (typically 10µm-100µm diameter) are used as capillaries in CE. By further decreasing the dimensions of the capillary, the negative effects of joule heating in electrophoresis can be reduced since the temperature distribution in the channel is more uniform, and heat is better dissipated [2]. The result of this is that more data can be analyzed in shorter time. When a rectangular channel with a high aspect ratio (depth/width) is used, the optical on-column detection sensitivity improves significantly since the surface to volume ratio is larger. Potentially 2D separation can also be realized in the rectangular channels.

Conventional microfabrication methods such as masking and substrate etching have been reported by Matsumoto *et al.* in [3] to make micro-to-nano channels. While highly precise and reliable, the use of e-beam lithography and RIE etching can be expensive and time consuming. Qin and Li have shown a novel technique in creating complex micro channels using a Nd:YAG laser in a dry process [4]. This paper reports the use of this technique to create nano *fractures* as fluidic channels. We believe that it is the first ever report on initiating and controlling nano scale fracture for constructive applications. Potential applications for these nano-sized rectangular channels include capillary electrophoresis (CE) and nano-filtration.

2. Fabrication of Micro-channels

As reported by Qin and Li [4], Nd:YAG lasers can be used to create complex 3D micro channel systems bounded by true solid 3D quartz substrates of any shape by means of *plasma-induced* or *thermal-induced* method. In laser micromachining, the energy of the laser need to be absorbed and transformed into heat by the material under processing. However, quartz is a very optically transparent material and its average absorptivity for the spectrum from visible to near infrared is less than 5%. Hence, it is very difficult to machine quartz using a mid-power Nd:YAG laser with wavelength of 1064 nm by front surface absorption of laser energy. Nevertheless, we have demonstrated that by using a higher electric field to allow an electric breakdown at the exit surface of a transparent material, laser-micromachined channel systems in optically transparent quartz cubes can be fabricated (see Figure 1). Moreover, we have found that if the laser-micromachined micro channels on a quartz substrate are strategically placed, nano-sized channels can be induced.

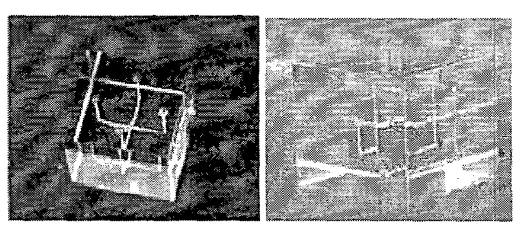


Figure 1. Complex micro channels in a 3-D substrate fabricated by laser micromachining (Qin and Li [4]).

2.1 Thermal-induced Fabrication

A Q-switched Nd:YAG laser, 40 W of maximum power, (Electrox Scriba II D40, UK), was used in our work. The frequency of the laser pulse was set at 2 kHz and the pulse width of the laser was 100-300 nanoseconds. The laser beam was focused by a scanning lens (f·θ lens) with a focal length of 100mm onto the sample surface. The Gaussian spot size of the beam was determined to be about 400 μm^2 at 1064 nm (radius of ~ 11.3 μm at $1/e^2$ intensity). All channels were formed in optical grade fused quartz cubes (bubble free synthetic silica, Almaz Optics, Inc., USA). The drilling processes were carried out in air with atmospheric pressure with the laser beam directly focused on the substrates (maskless).

A microscope photo of a channel fabricated by thermal-induced processing is shown in Figure 2. By knowing the laser pulse frequency, pulse width, focal spot, and measured average power, the laser fluence at the rear surface of a quartz substrate was estimated to be about 160 ~ 370 J/cm^2 (0.65 ~ 1.5 mJ pulse energy) for the thermal-induced process. The average laser power was measured using a Power MAX5200 laser power meter (Molectron, Detector Inc.). Quartz is a fragile material, so the channel shape depends strongly on the state of stress of the quartz during the laser interaction. As shown, there are many thermal cracks around the channel. Also, residual stresses in the quartz may cause secondary fracturing. This effect affects the channel cross-section and reduces the straightness of the channel. The channel cross-sectional geometry cannot be well controlled, as shown in Figure 2.

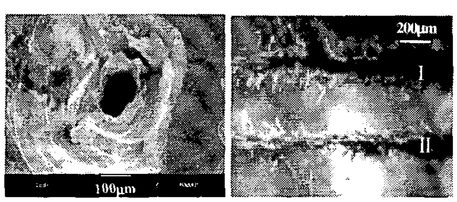


Figure 2. Thermal-induced laser-fabricated channels.

2.2 Plasma-induced Fabrication

Plasma of most elements can be produced by a high-intensity pulsed laser irradiation. When a series of laser pulses strike a solid target and are absorbed by the target, a characteristic sequence of energy conversion processes leads to the production of hot and dense plasma consisting of matter in an extreme state of high-energy concentration. The structure of the solid target plays a key role during the laser-target interaction. A flat target may just absorb incoming irradiation once, while a cavity may store irradiation for a much longer time by reflecting the irradiation on the interior wall of the cavity. Therefore, there is much more opportunity for a cavern target to absorb irradiation energy and plasma may be more easily induced in a cavity.

A quartz substrate was first pre-damaged at a site by thermal-induced processing, and the following laser pulses could be easily absorbed by this locally damaged site. Then the laser beam was focused on this initiated site on the quartz surface. Once the damaged site began to absorb enough laser energy, hot and dense plasma of quartz was produced around this site. The plasma attacked and melted the neighboring quartz and then a hollow was formed. The hollow functioned as a cavity for the following laser pulses. The laser-induced plasma of quartz was produced in the cavity on each subsequent pulse to drill the hollow further downward, producing a micro channel. The channels fabricated by this process are of high quality with a smooth kerf surface and no thermal cracks are observed, as shown in Figure 3. This process can also be used to smooth-out the thermal-induced channels, hence, complex channel systems with smooth interior wall can be fabricated inside a quartz substrate by the combination of the two processes.

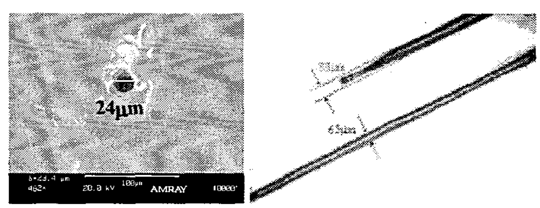


Figure 3. Thermal-induced laser-fabricated channels.

3. Fabrication of Nano Channels

By drilling thermally-induced initiating micro channels (60μm-100μm diameter) in the succession as illustrated in Figure 4, a consistent nano "fracture" propagates along the aligned axis to form the nano and micro rectangular channels. We have found that 3 initiating micro channels are necessary to induce a nano channel. The reason for this phenomenon is under investigation, and is most likely due to induced stress gradient on the quartz substrate caused by laser-processed cavities.

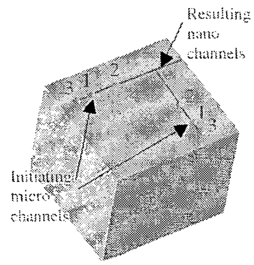


Figure 4. On a $1cm^3$ substrate, the initiating circular micro channels are fabricated in the sequence indicated by the numbers, and the resulting nano channels are shown.

The widths of the induced channels range from ~500nm to 1.5µm as shown in Figure 5. These nano channels extend through the substrate (1cm). By aligning three or more initiating micro channels in an axis normal to the original nano channel, a new nano channel will intersect the original to form a 2-D system as shown in Figure 6. These channels are formed and controlled by strategic placement of initiating and terminating micro channels. The nano channels can be created in both fused silica and crystalline quartz.

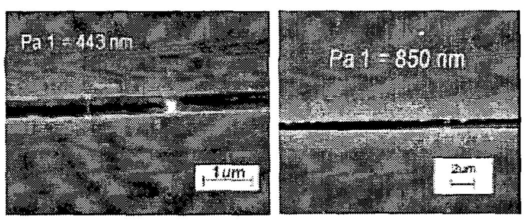
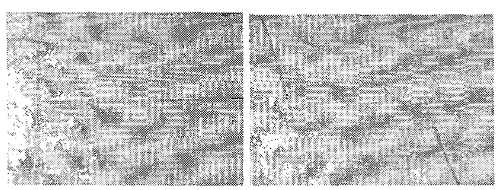
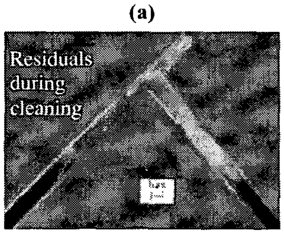


Figure 5. SEM of the nano channels with different channel widths.



(a)



(b)

Figure 6. Intersecting nano channels. (a) Microscope photos of nano channels intersecting. (b) SEM showing the geometry of intersection.

3.1 Test for Connectivity of the Nano Channels

Since the nano channels initiate from the micro channels, the micro channels can be used as the sources and drains of cellular samples suspended in a fluid medium and the nano channels can be used as the workspace. We have verified that these interconnected channels are continuous by injecting $FeCl_3$ conductive dye into an inlet micro channel. The channels were proved to be connected visually, as shown in Figure 7. Connectivity was also tested by measuring the resistance between an inlet and an outlet micro channel connected by a nano channel and also a system of connecting nano channels. For the test involving a nano channel between two micro channels the dye was injected at t=0 when the nano channel was dry and resistance cannot be measured. At t=1sec, the dye has propagated through the micro and nano channel to yield a measurable resistance. The resistance decreases until t=4.5sec where it begins to increase due to dye depletion as the bulk moves to other interconnecting nano channels. A plot of this resistance measurement over time is shown in Figure 8.

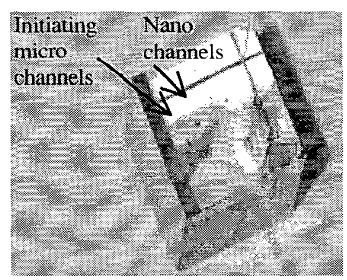


Figure 7. Nano channels tinted with dye. The dye was injected from the micro channels and the capillary force transported it into the nano channels.

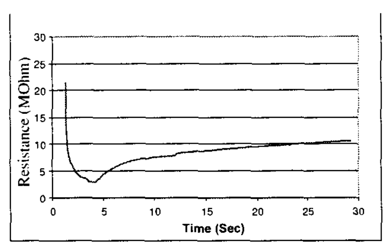


Figure 8. Test to validate the throughness of a nano channel.

Currently we are investigating various cellular-sample insertion methods to the inlet micro channels. This is in preparation for nano filtration through the nano channels. We have successfully injected saccharomyces cerevisiae cells via capillary transport shown in Figure 9 as well as insertion of the cells by electro-osmosis shown in Figure 10. Our goal is to perform nano filtration to separate bacteria (nano scale) from cells (micro scale).

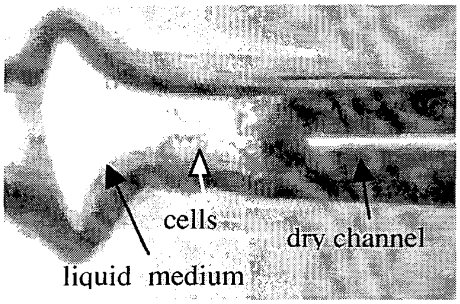


Figure 9. Capillary transported saccharomyces cerevisiae cells into source micro channel.

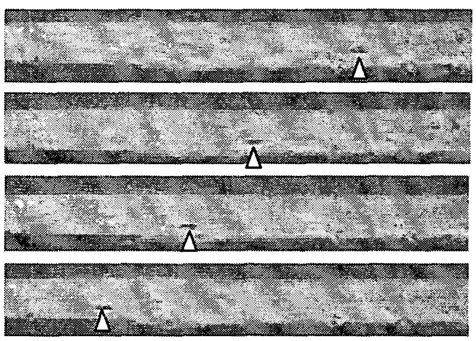
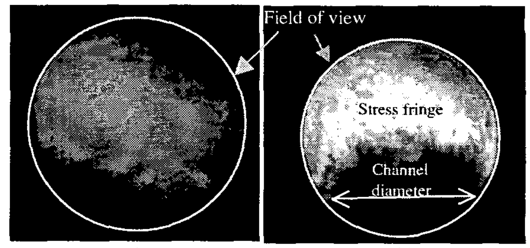


Figure 10. Electro-osmosis driven flow to insert cells into source micro channel. The arrow follows a cell strand flowing from right to left in electric field of 100V/cm. (Cell photo is color enhanced for easier visualization)

3.2 Physical Interpretation for the Creation of the Laser-induced Nano Channels

We speculate that these nano fractures are caused by thermally induced stress during and after laser drilling; this is evident by the stress fields around the initiating micro channels and shown in Figure 11. The figure shows the polariscope images obtained using photoelastic method for the visualization of internal stresses near the initiating micro channels. The bright/dark field fringes indicate areas of stress concentration. The stress gradient extends radially, and hence, we speculate that when enough micro channels are aligned on an axis in the substrate, the total stress is significant enough to cause a nano "fracture" along the micro channels. More detailed experimental tests on determining the nano channel geometries with the locations of the micro channels are underway.



(a) (b)

Figure 11. (a) Small stress can be seen on un-processed crystalline quartz substrate; for fused silica it is even less. (b) Stress on fused silica after making the initiating micro channel.

4. Discussion

The ability to fabricate nano-sized channels and cavity will be important for the advancement of making many fluidic systems useful in improve performance and add new functionality to many applications such as chemical, blood, DNA, and environment science analyses. Applications include those for cell-to-cell interaction, and cell culturing where nano-holes can be used to filter out virus and damaging particulates and allow nutrients to get to the cell, thus allowing the cell to live for a very long time. However, not many nano channels have been reported because of a fundamental problem. Conventional surface micromachining techniques can be used to make nano channels if the sacrificial layer is made to be nano-meter height. However, during the etching process, the process depends on diffusion of etching, and hence if the sacrificial layer is too thin, diffusion can be very slow. Also, if the channels are too wide, the structural diaphragm can collapse. Our work overcome these difficulties and problems by offering a fast and cheap process to connect inlet and outlet micro channels with nano channels. Future work involves fluid flow visualization of the nano channels, cell culturing, DNA and molecular manipulation, and cell to cell interaction studies in the nano channels.

5. Summary

A novel laser processing technique for making channels in the nano regime is presented in this paper. A Nd:YAG laser has been used to dry fabricate micro channels (60µm-100µm diameter) in $1cm^3$ fused silica and crystalline quartz substrates by thermal-induced processing. By controlling the locations of the initiating micro channels on the substrates, a 1D-controllable self-connecting nano fracture can be formed as a rectangular channel. This nano channel is smooth with extremely high aspect ratio (~500nm-1.5μm) in width and 1cm depth. The length is controlled by the separation of the initiating micro channels. We have also demonstrated that these nano channels are thoroughly connected to the inlet and outlet micro channels by visual and resistance measurement tests. This laser-based nano channel fabrication technique is fast, and inexpensive with potential applications in capillary electrophoresis, and electro-osmosis driven nano-filtration.

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7. References

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