

Femtosecond laser machining of fluidic microchannels for miniaturized bioanalytical systems

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ABSTRACT

Femtosecond laser ablation of borosilicate (BorofloatTM) glass has been studied to machine fluidic micro-channel geometries (high aspect ratio and variable depth) not possible through traditional micro-lithographic techniques. Utilizing a 1 kHz repetition rate femtosecond laser system (Positive Light, Spitfire) and a long-working distance 5x objective lens, groove patterns 10 μm wide and as deep as 30 μm have been produced. The experiments were performed in air and the samples were cleaned after the ablation with sodium hydroxide dissolved in water to remove the debris. The substrates were mounted on a computer controlled x-y translation stage. The quality of the micro-channels showed dependency on the scanning speed of the sample. The surrounding area of the channels was smooth at scanning rates greater than 400 $\mu\text{m/s}$ and smaller than 10 $\mu\text{m/s}$. Whereas, cracks appeared around the channels at scanning rates between 200 to 50 $\mu\text{m/s}$. Surface morphology is studied using optical, electron and atomic force microscopies. For a quantitative evaluation of ablation threshold and ablation rates, single-shot experiments in vacuum were performed. We found that the damage threshold for borosilicate glass is around 1.7 J/cm². With single pulse laser fluence of 30 J/cm², a 600 nm deep crater could be ablated. A ring, higher than the surface, appeared around the craters and was most probably created by adiabatic compression of glass due to the high-pressure plasma generated in the early stages of the process.

Keywords: Ultrafast lasers, micromachining, borosilicate glass, damage threshold, ablation rate, optical penetration depth, micro-channels, microfluidics

1. INTRODUCTION

The advent of lasers with sub-picosecond pulse durations and millijoule output power has opened new opportunities for precision laser machining of metals, dielectrics, and biological materials. The rapid coupling of energy allows material removal before significant heating occurs, resulting in extremely precise and controllable processing with nearly undetectable collateral damage

At Stanford University, we have recently established a new ultrafast laser laboratory with the hardware necessary to carry out studies of ultrafast laser/material interactions and applications to machining of a variety of materials. In this paper, the initial studies on the use of laser machining of borosilicate glass to generate fluidic micro-channel geometries are presented. Because of the isotropic nature of wet chemical etching, only shallow channels can be produced using these photolithographic techniques. However, fabrication of deeper channels has a number of important practical advantages. Deeper channels provide an increased path length for optical detection that yields a linear increase in signal. An ultrashort pulse laser can machine high aspect ratio channels on borosilicate glass to be used in the development of miniaturized bioanalytical systems.

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The paper summarizes the measurements of damage threshold and ablation rate of borosilicate glass when machined with femtosecond laser pulses and the influence of scanning rate on the machining process of micro-channels for miniaturized bioanalytical systems. Surface morphology of grooves machined with femtosecond pulse laser is analyzed using optical, electron and atomic force microscopies.

2. EXPERIMENTAL APPROACH

The experimental facility for ultrafast laser microfabrication consists of two parts: 1) the laser system, 2) a microscope for beam delivery to the sample. A kHz repetition rate Ti:sapphire amplifier system (Positive Light, "Spitfire") is used to generate ultrashort pulse lasers with output energies up to 0.9 J per pulse at 780 nm. The chirped-pulse amplification (CPA) technique used in the amplification system allows us to vary the pulse duration from 200 fs up to 20 ps. The pulse duration is measured by means of a single-shot, second harmonic generation autocorrelator. For the current experiments, 300fs duration pulses are used.

The laser fluence on the target can be precisely varied from 10 nJ to 0.9 J using two energy attenuators. Each energy attenuator involves a half-wave plate that rotates the polarization of the laser beam and a cube beam splitter in which the intensity of the transmitted light depends on its polarization. The attenuated laser-pulse energies are measured by a pyroelectric detector. The output of the laser system is linearly polarized. A fast mechanical shutter is used to select the desired number of pulses for the ablation experiments.

The laser beam is delivered to the surface by a long working distance objective lens (Mitutoya, 5x) attached to the microscope. To obtain a homogeneous and well-defined distribution on the target, a circular aperture (5mm in diameter) is placed in the beam path just before the objective lens. In the surface plane, a Gaussian spatial beam profile with a radius ($1/e^2$) of $\omega_0=5.8 \mu\text{m}$ is obtained. The same microscope can be used to inspect in-situ the irradiated surface during the ablation process.

For micro-structuring, the substrates are mounted on a computer controlled x-y translation stage of the microscope. The surface of the sample is positioned to be perpendicular to the direction of the incident beam. The borosilicate glass substrates (Borofloat™ 1.1 mm thick, Precision Glass and Optics, Ltd.) used in the current experiments are cleaned ultrasonically with alcohol before the experiments and with a 0.5 Molar sodium oxide (NaOH) solution after the ablation to remove debris.

Figure 1 shows a picture of the microscope with objective lenses for beam focusing and the vacuum chamber mounted on the x-y translation stage. A Lab-VIEW program controls both the laser energy by rotating the half-wave plate and the position of x-y stage for machining of micro-channels (grooves).

The laser ablated borosilicate glass surface is analyzed by optical and scanning electron microscopes for its morphology and by an atomic force microscope (AFM) for the measurement of the profile, that is, the size and depth of the machined surface. AFM is an excellent tool to investigate craters with small depths ($< 6 \mu\text{m}$). From these measurements a quantitative evaluation of the crater depth, the ablated volume and the crater profiles is possible.

In this work, the effects of variety of parameters such as laser fluence, pulse repetition rate, and speed of scanning rate on the quality of micro-channels were studied.

3. RESULTS AND DISCUSSION

Figure 2 shows a comparison between nanosecond and femtosecond pulse laser ablation of borosilicate glass in air. Thermal effects, which dominate the long pulse laser ablation, cause cracks with non-reproducible machining quality. Ablation of borosilicate glass with an ultrashort laser pulse is, in contrary to the processing with long pulse lasers, free of thermal diffusion providing a precise and reproducible processing ability. These results demonstrate that for high quality machining of glass substrates ultrashort pulse lasers are needed.

In the following sections the properties of the single shot laser ablation of borosilicate glass will be first discussed. Second, the damage threshold and ablation rate data will be presented. The third part will summarize the properties of the micro-machined channels (grooves).

3.1. Properties of Single Shot Craters

AFM studies were performed to examine the properties of craters generated using a single laser pulse in vacuum with different energies. Figure 3 presents three examples of AFM images of craters ablated with laser pulse energies of 13.6,

18.8, and 30 μJ . These AFM pictures depict no cracks around the ablated crater and that the dimensions of the craters increase with increasing laser energy. These images also show the existence of a white ring around the ablated crater from which thin strips of melted material are extending away. Three-dimensional representation of AFM data shows that the height of these features around the crater is higher than the surface. The ring formation might be attributed to an adiabatic compression of glass due to high-pressure plasma generated in the very early stages of the ablation process. The plasma starts to expand within 10 ps above the surface having enough time to compress the material around though with negligible heat diffusion. The thin strips expanding away from the crater might be the result of the recasting of ablated material that could not escape from the surface and melted back.

3.2. Ablation Rate

To determine the threshold fluence of borosilicate glass ablation, we plotted the ablated depth h of single-shot craters as a function of laser fluence F_0 (Fig. 4). To precisely measure the ablation threshold, namely, to avoid nonlinear beam distortion in air the experiments were performed in a vacuum chamber at a pressure below 10^{-4} mbar. The linear relation between the depth and the logarithm of the laser fluence can clearly be observed in Fig. 4. This logarithmic dependence of the ablation depth on the laser pulse fluence was demonstrated in early studies¹ for the ablation of metal targets with femtosecond laser pulses and can be described by

$$h = \kappa \ln \left(\frac{F_0}{F_{th}} \right), \quad (1)$$

where κ can be interpreted as the “effective optical penetration depth” as expected from the Beer’s law². The slope of the linear fit yields the optical penetration depth. A value of $\kappa = 221$ nm was obtained for borosilicate glass. The extrapolation of the fit to $h = 0$ resulted in a damage threshold of $F_{th} = 1.7$ J/cm². This result is consistent with earlier studies^{3,4} where a multi-shot damage threshold of F_{th} ($N = 50$, 300fs) = 1.7 ± 0.4 J/cm² was found.

3.2. Machining of Microchannels

Micro-channels on the surface of borosilicate sample were machined by moving the sample with a certain speed relative to the stationary-pulsed laser beam. The speed at which the samples are moved is called the scanning rate. Figure 5 shows optical images of channels machined at different scanning rates at 200 Hz pulse repetition rate. These experiments were performed in air at laser fluence of $F_0 = 28$ J/cm².

A minimum number of overlapping laser pulses was required to create a continuous channel. At scanning rates below 400 $\mu\text{m/s}$ an effectively continuous overlap of multiple laser pulses was reached, leading to ablation depth of several microns. The ablation depth of micro-channels depends on the number of overlapped laser pulses N_{eff} , which can be defined as:

$$N_{eff} = \frac{D \cdot RR}{SR}, \quad (2)$$

where D is the diameter of a single pulse crater, RR is the repetition rate of the laser system and SR is the scanning rate at which the samples are moved. Assuming 10 μm for the diameter of a single pulse crater, the minimum number of pulses required to obtain an effectively continuous channel was $N_{eff} = 5$ (scanning rate of 400 $\mu\text{m/s}$ at 200 Hz repetition rate.)

The quality of the microchannels was found to be a strong function of the scanning rate or in other words overlapping number of pulses. As Fig. 5 indicates, at intermediate speeds ($SR = 200$ -50 $\mu\text{m/s}$), cracks along the sidewalls of channels are obtained when the laser repetition rate is at 200 Hz. At speeds lower and higher than these intermediate values cracks could be eliminated. Uniform and highly reproducible channels are achieved at $SR > 400$ $\mu\text{m/s}$ and $SR < 50$ $\mu\text{m/s}$.

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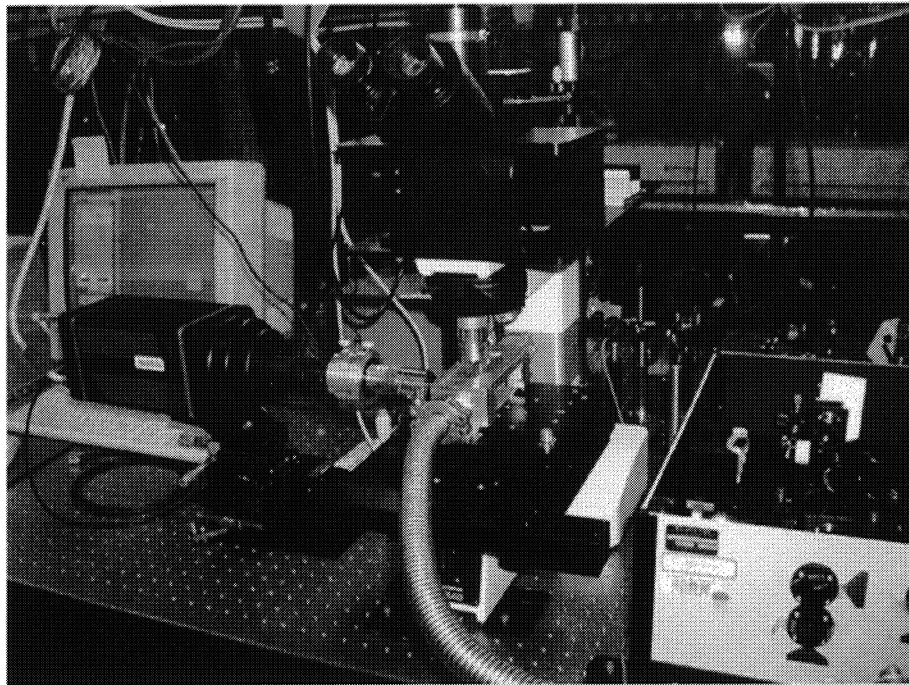
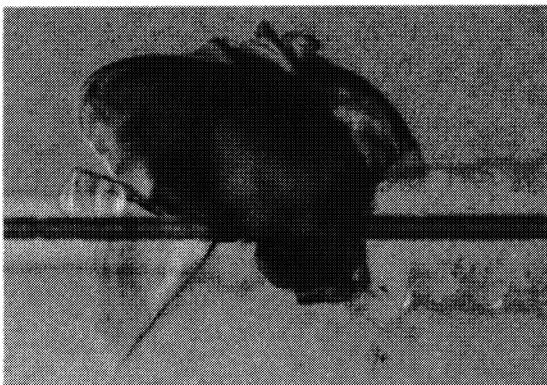


Figure 1. A picture of the experimental facility.

(a) $\tau=10$ ns.



(b) $\tau=300$ fs.

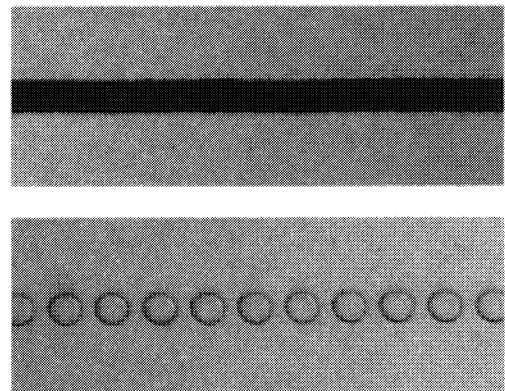


Figure 2. Comparison of ns and fs-pulse laser machining of borosilicate glass at $\lambda=780$ nm.

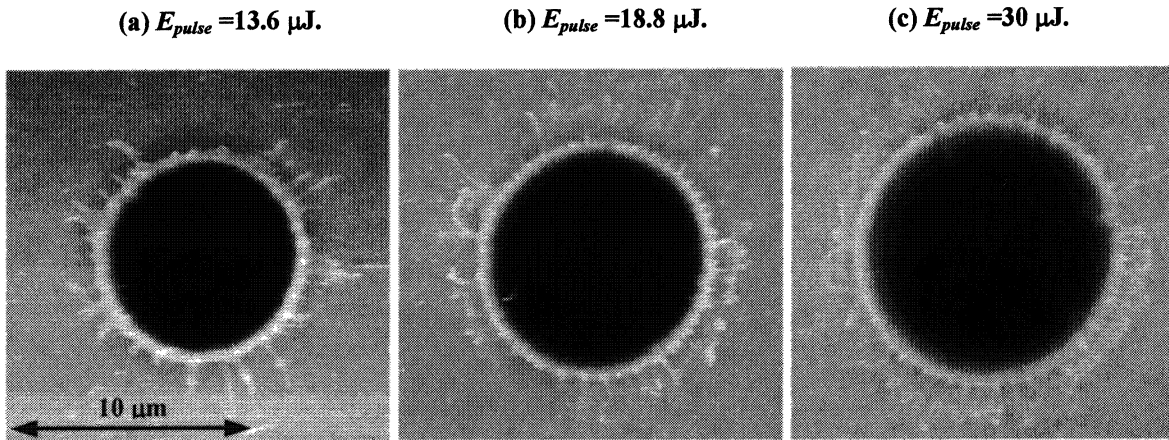


Figure 3. Samples of atomic force microscopy images demonstrating the properties of single shot craters for different laser pulse energies.

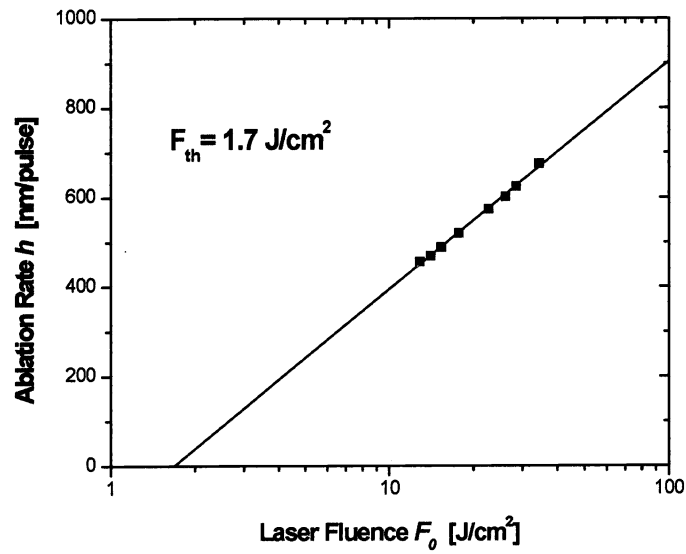


Figure 4. Plot of ablated depth of borosilicate glass as a function of the laser fluence.

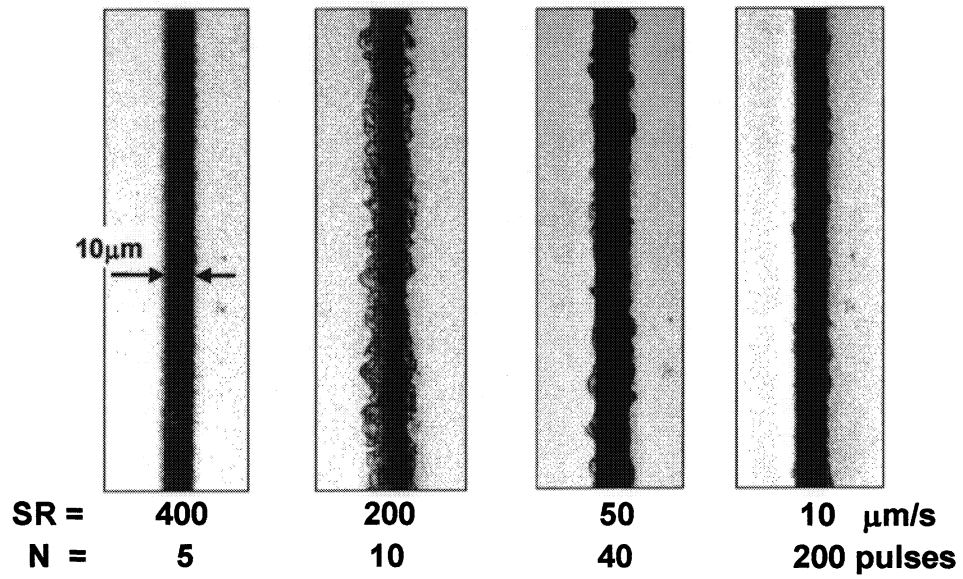


Figure 5. Effect of the scanning rate in machining of continuous micro-channels. Laser repetition rate was 200 Hz. Laser pulse energy was $E_{pulse}=29 \mu\text{J}$ ($F_0=27.5 \text{ J/cm}^2$.)