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Femtosecond laser machining of multi-depth microchannel networks onto silicon

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Abstract

Direct writing of multi-depth microchannel branching networks into a silicon wafer with femtosecond pulses at 200 kHz is reported. The silicon wafer with the microchannels is used as the mold for rapid prototyping of microchannels on polydimethylsiloxane. The branching network is designed to serve as a gas exchanger for use in artificial lungs and bifurcates according to Murray’s law. In the development of such micro-fluidic structures, processing speed, machining range with quality surface, and precision are significant considerations. The scan speed is found to be a key parameter to reduce the processing time, to expand the machining range, and to improve the surface quality. By fabricating a multi-depth branching network as an example, the utilization of femtosecond pulses in the development of microfluidic devices is demonstrated.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Femtosecond (fs) laser pulses have been attractive for a wide variety of micromachining applications because of their ability to fabricate micron-level features with minimal peripheral damage and associated debris. The machined quality and the resolution of such ultrashort laser pulses have been proven in many ‘proof of concept’ demonstrations [1–5]. However, the low productivity of fs pulses has limited their contribution in practical applications. This study investigates fs pulses with a high repetition rate (200 kHz) to fabricate a wide range of microchannel structures onto silicon with quality surface and increased processing speed. This is demonstrated by fabricating microchannel branching networks to replicate the flow of blood in mammalian vasculature to serve gas exchangers for use in artificial lungs.

Current generation of artificial lungs is not efficient enough to support patients with chronic irreversible progressive pulmonary failure [6–9]. Structurally, the inefficiency of current hollow fiber blood oxygenators is attributed to their non-physiological features, which include long diffusion lengths, high flow resistances, and non-uniform flow distributions [10]. The lung-like features are essential to the design of an artificial lung to enhance the gas exchange performance while maintaining the biocompatible blood flow condition. In the natural vasculature, the trade-off between the pressure drop and the diffusion lengths of the blood vessel is well balanced with uniform flow distribution. This optimal vascular architecture follows a relationship known as Murray’s law [11, 12]. For a bifurcation, as illustrated in figure 1(a), Murray’s law states that the radius of the parent vessel \( r_0 \) and the radii of daughter vessels \( r_1 \) and \( r_2 \) have the following relationship:

\[
r_0^3 = r_1^3 + r_2^3.
\] (1)

The human pulmonary vasculature consists of 15 branching levels, with diameters ranging from \( \sim 20 \mu m \) up to several millimeters [13].

In order to create an efficient gas exchange and a biocompatible flow condition, we seek to replicate natural
vascular networks. Multi-depth microchannel branching networks are proposed as gas exchangers to mimic the natural vascular structure. The network bifurcates according to Murray’s law and thus the channels become shallower and narrower at each bifurcation as illustrated in figure 1(a). To apply Murray’s law to rectangular cross sections, the hydraulic diameter is used instead of the radius. Here, the aspect ratio of the width to the depth of channels in the network is designed to be constant. While single depth branching designs can implement Murray’s law by varying the width of each generation [14], the multi-depth design is beneficial for a more compact design which could be easily implantable. In the development of the networks, a wide range of microchannel size with reasonable surface quality and resolution is necessary to realize various designs. The current phase of our artificial lung development requires multi-level structures with depths ranging from ∼20 μm to several hundreds of microns. Additionally, the surface roughness should be minimized to reduce any adverse effect of surface roughness which may initiate blood clotting and clogging [6, 10].

This study describes fs laser machining of multi-depth microchannel networks into silicon as a part of the development of artificial lungs. It is possible to build such branching networks using lithography and etching-based techniques; however, the mask-based lithographic techniques are inefficient in terms of cost and development time, and it is difficult to lithographically vary the depth of such multi-depth structures. An alternative method to reduce cost and time is the laser direct writing, which does not need any masks. In addition, the flexibility of the laser process facilitates creating multi-depth structures of such branching networks. In our previous studies [15, 16], multi-depth branching microchannel networks were laser machined into silicon with nanosecond (ns) ablation followed by acid etching. However, the channel depth range with a reasonable surface quality was not wide enough to realize various microchannel network designs. Although the chemical wet etching post-processing smooths the surface roughness, the relatively long chemical etching time required to remove debris buildup, resulting from a strong thermal reaction of irradiated silicon, limits the minimum channel size. Alternatively, fs ablation provides better surface quality and resolution than ns ablation by minimizing the thermal impact on irradiated material [1, 2].

There have been extensive studies on fs ablation of silicon [17–24]. Generally, the repetition rates (< several kHz) of the fs pulses used in these studies are accompanied by low processing speed. This is because relatively low pulse fluence is often required for high processing qualities, which is not wide enough to compensate the productivity loss by such a low repetition rate [24]. In the majority of applications including our artificial lung fabrication, however, macroscopic amounts of material removal and micron-level precision are required. Therefore, the development of silicon micromachining processes with high repetition rate (200 kHz) fs pulses would have significant impact. The machining range, the processing speed, and the machined quality are all considered in this study.

Achieving the necessary feature depth with quality surface and rapid processing speed requires efficient energy transfer from laser to the target; however, the energy transfer must be well controlled in order to maintain sufficient surface smoothness. The laser energy transferred to the target can be manipulated with processing parameters such as the pulse energy, the focal position, the scan speed, and the number of scan passes. Due to the high repetition rate of the fs laser used in this study, the number of scan passes and the scan speed are manipulated as two main parameters. First, the ablation depth range in a single pass scanning is measured to optimize the pulse energy, the focal position, and the scan speed. In multiple-pass scanning, the influence of the scan speed on the channel depth, the material removal rate (MRR), and the surface roughness is studied.

As a demonstration, a 9-level branching network obeying Murray’s law, for which the depth range is 50–320 μm, is laser machined into a silicon wafer. Actual branching networks are created out of polydimethylsiloxane (PDMS) using the laser machined silicon structures as molds.

2. Experimental method

The schematic of the fs laser micromachining setup is shown in figure 2. The laser used for this study is a prototype fiber laser operating at 1040 nm. The laser is set to produce ∼600 fs pulses with ∼10 μJ pulse energy at a repetition rate of 200 kHz. So the output average power is ∼2 W at 200 kHz. The average power is varied using a combination of a half-wave plate and a polarizer in beam delivery. A quarter-wave
In the laser scan with a single pass as illustrated in figure 3, the pulse energy \( \Delta E \), the focal position \( z \), and the scan speed \( v \) are varied to control the microchannel depth \( D_{\text{mc}} \). Figure 4(a) shows a SEM image of a fs-ablated channel with \( \Delta E = 1.14 \mu J \) at \( v = 5 \text{ mm s}^{-1} \) and \( z = 0.4 \text{ mm} \) following 20 s acid etching. This channel has a depth of \( D_{\text{mc}} \sim 24 \mu \text{m} \) and width of \( \sim 50 \mu \text{m} \). The laser ablation depth \( D_{\text{la}} \) is smaller than \( D_{\text{mc}} \) by the depth etched by the acid solution. The difference is estimated to be approximately 17 \( \mu \text{m} \). While its machined quality proves to be superior to that of longer pulses, fs pulse interaction with silicon still leads to considerable surface roughness \[18, 19, 21–24\].

To select optimized \( v \), single pass channels are created for the scan speeds varying from 1 to 30 mm s\(^{-1}\) by 1 mm s\(^{-1}\) increment with 9.72 \( \mu J \) (2.56 J cm\(^{-2}\)) pulses at the repetition rate \( f \) of 200 kHz. It is observed that when the scan speed is set to 5 mm s\(^{-1}\), the notch-like shape is not observed after 20 s acid etching. With the optimized scan speed found above, it is found that \( D_{\text{mc}} \) variation from 17 to 63 \( \mu \text{m} \) can be achieved by manipulating the pulse energy and the focal position below the surface. This is shown in figure 4(c). The minimum depth, 17 \( \mu \text{m} \), is the result of the laser removal of nitride coating only and subsequent 20 s acid etching. Thus, using single pass scanning at focus with 2.56 J cm\(^{-2}\) pulse fluence and \( v = 5 \text{ mm s}^{-1} \), the ablation depth is \( D_{\text{la}} \sim 46 \mu \text{m} \). Higher scan speed results in reduced \( D_{\text{la}} \) for single pass scan as reported in many other papers \[18, 22, 24\]. Using an effective number of pulses for single pass scan driven by Crawford et al \[22\], the ablation depth per pulse (adpp) is estimated to be \( \sim 84 \text{ nm} \). Crawford et al \[22\] performed a systematic study
on the fabrication of linear grooves in ⟨100⟩ silicon using 150 fs pulses at \( f = 1 \) kHz and the wavelength of 800 nm. The ablation depth per pulse is measured in terms of the fluence ranging from 0.1 ∼ 1 J cm\(^{-2}\) for \( v = 0.1, 0.25, \) and 0.5 mm s\(^{-1}\). The value increases up to adpp ∼ 1 μm with the fluence of 11 J cm\(^{-2}\). The fluence of ∼3 J cm\(^{-2}\) resulted in adpp of 100 ∼ 170 nm depending on \( v\). The result is similar in magnitude to ours, although their wavelength, pulse duration, and experimental conditions are different. Although our \( v = 5\) mm s\(^{-1}\) is higher by an order of magnitude, the pulse-to-pulse distance \((v/f)\) is shorter by an order of magnitude due to the high repetition rate (200 kHz). Less spacing between pulses can lead to more roughness and debris, but acid etching used in our study allows a shorter pulse-to-pulse distance while maintaining good surface quality. The productivity can be estimated by calculating \( f \cdot \text{adpp}\). For the same fluence, therefore, the productivity of our laser setup at \( f = 200\) kHz would be higher by approximately two orders of magnitude than that of the study at \( f = 1\) kHz. The productivity of the low repetition rate fs pulses can be increased by increasing \( \Delta E\). However, it is known that high \( \Delta E\) is accompanied with a quality loss. An optimal fluence range of fs pulses for quality machining of silicon is suggested by several groups [21, 24], and it is reported that the ablation depth is limited at very high \( \Delta E\) [20]. Therefore, high \( f\) with low \( \Delta E\) is recommended to improve the productivity and to minimize the quality loss.

The width of single pass channel (\( W_s\)) varies from 35 to 62 μm as \( \Delta E\) increases from 0.45 to 9.2 μJ when machined at focus followed by 20 s etching. The width of single pass channel is further increased up to 95 μm as the 9.2 μJ beam is defocused up to 1 mm at 5 mm s\(^{-1}\) scan speed. The effect of \( v\) on the width is relatively small compared to \( \Delta E\) and \( z\).

### 3.2. Multiple-pass scan

To increase the range of the microchannel depth (\( D_{mc}\)) and the width (\( W\)), multiple-pass scan is used. As illustrated in figure 5, this process is a layer-by-layer ablation process. To vary the channel width, multiple parallel lines are scanned with a separation (\( \Delta d\)) of 20 μm so that the number of lateral parallel lines (\( N_w\)) determines the width of the channel (resolution is determined by \( \Delta d\) and \( W_s\)). The number of laser-scanned layers (\( N\)) is manipulated to control \( D_{mc}\); however, the ablation depth does not always increase with \( N\) as observed in
Figure 6. SEM images of the cross section of laser machined channels onto silicon after 20 s acid etching. The scan speed is 480 mm s\(^{-1}\).

Figure 7. (a) Ablation depth into nitride coated silicon wafer versus the number of scan passes, \(N\), after 20 s acid etching. 9.72 \(\mu\)J pulses are scanned at focus with \(v = 30, 120, 480, \) and 1920 mm s\(^{-1}\). (b) The ablation depth versus \(v\) with \(N = 95\) and 2.56 J cm\(^{-2}\) fluence at focus.

\(v\) also plays a key role in the laser processing speed and the machined surface quality. The variation of the MRR with \(v\) is presented in figure 8(a). The MRR is measured for \(N = 95, 195,\) and 395. In the measurement, the effect of the acid etching is not included. The maximum MRR \(\sim 0.5\) mm\(^3\) min\(^{-1}\) is obtained at \(v \sim 750\) mm s\(^{-1}\).
or the formation of the conical spikes with N. Another reason might be the defocusing effect as the ablation depth increases, since the focal position is fixed during machining and for this case and the Rayleigh range is $\sim 300 \text{ m}$.

The dependence of $Ra$ on $v$ is similar to that of the MRR. Thus, $v$, which provides the optimal material removal, also creates a smooth channel surface. This is described by the relation between the heat accumulation and overlap of fs pulses at a high repetition rate. At low $v$, the distance between pulses is closer so that the thermal effect on the surface is increased by the heat accumulation. The thermal reaction is accompanied by debris buildup. This debris fills the channel and blocks laser energy delivery to the target surface increasing the surface roughness. As $v$ is increased, debris buildup is reduced which increases the overall MRR and reduces $Ra$. However, as $v$ is further increased, the effectiveness of material removal is reduced due to the increased pulse-to-pulse distance. This results in higher roughness because of increased peak-to-valley roughness. These competing processes create an optimal MRR and $Ra$ at certain $v$ as shown in figure 8.

$Ra$ as a function of $D_{mc}$ is plotted in figure 10. For fs ablation, $v$ is set to 480 mm s$^{-1}$ and $N$ is varied from 1 to 320 resulting in $D_{mc}$ from 28 to 236 $\mu$m after 20 s etching. For comparison, channels are also machined with a pulsed Nd:YAG laser operating at 1064 nm with 500 Hz repetition rate and 100 $\mu$s pulse width [15]. The 100 $\mu$s pulses are modulated to produce a series of 200 ns pulses with 10 $\mu$s interval. The near-Gaussian beam spot size of FW1/e$^2$M is about $30 \mu$m in diameter. The average power is varied from 0.2 to 3 W with the scan speed of 8 mm s$^{-1}$ and 0.5 mm defocus below surface. For both fs and ns laser ablation, the surface roughness increases with the ablation depth as observed in many laser ablation studies [18, 19, 24]. For 20 s etch time, the maximum ns ablation depth, with $Ra$ of $< 2 \mu$m, is $\sim 50 \mu$m, whereas, for fs ablation, a depth of more than 200 $\mu$m can be achieved with $Ra < 2 \mu$m. When the etch time for the ns-ablated channel is increased to 40 s, the maximum depth with $Ra < 2 \mu$m is increased to $\sim 130 \mu$m; however, the minimum channel depth also increases to $\sim 90 \mu$m.
Figure 10. Average roughness $Ra$ versus depth $D_{mc}$ for fs and ns laser machined channels after acid etching. For fs ablation, the scan speed is set to $v = 480\, \text{mm s}^{-1}$.

3.3. Laser machining of the multi-depth microchannel network

Based on the above study, a 9-level branching network is laser machined onto a nitride coated silicon wafer with the fs laser, as shown in figure 11(a). The SEM image in figure 11(b) shows the surface morphology of the laser machined channel surface. The network is designed to bifurcate according to Murray’s law and thus the channels become shallower and narrower at each bifurcation as illustrated in figure 1. The depth of the branching network ranges from 50 to 317 $\mu$m as presented in table 1. Here, the depth-to-width ratio of each generation remains approximately constant as well as the ratio of the depth to the length. In addition, all paths from inlet to outlet of the network are designed to have the same length to achieve the same pressure drop. To control the depth and the width of each generation of channels, $N$ and $N_w$ are varied. $N$ for desired depths is chosen by interpolating the plot in figure 7(a). The laser processing of this branching network took about 5 h. So macroscopic material removal in addition to micron level precision is achieved with fs ablation. A final blood flow/oxygen-exchange (oxygenator) device is created out of PDMS using the laser machined silicon structure as a mold as shown in figure 11(a) according to the procedure described in [15]. Then, a thin film of PDMS is placed on top of the PDMS network to make a closed channel. To function as an artificial lung, blood flows inside the channels and gases are exchanged through the gas permeable PDMS film.

4. Conclusions

 Femtosecond (fs) ablation of microchannels in silicon is studied to develop branching networks to serve gas exchangers for use in artificial lungs. Wide ranges of microchannel size with smooth surface are required to realize artificial vascular network simulating the mammalian respiratory blood vessel tree obeying Murray’s law. In the development of such devices, the total fabrication time is also a significant consideration. Using a high repetition rate significantly increases the fabrication speed of fs pulses. We show that the expansion of the machining range with smooth surface and the increase of processing speed in a large part rely on

Table 1. Depths ($D_{mc}$) and widths ($W$) of each generation of a 9-level branching network and corresponding number scan layers ($N$) and number of lateral parallel lines ($N_w$) with 20 s etching. Hydraulic diameters ($D_h$) are estimated assuming that the channel cross sections are rectangular. The pulse energy is 9.72 $\mu$J and the scan speed is 480 mm s$^{-1}$.

<table>
<thead>
<tr>
<th>Generation</th>
<th>$D_{mc}$ ($\mu$m)</th>
<th>$W$ ($\mu$m)</th>
<th>$D_h$ ($\mu$m)</th>
<th>$N$</th>
<th>$N_w$</th>
</tr>
</thead>
<tbody>
<tr>
<td>First</td>
<td>317</td>
<td>3180</td>
<td>577</td>
<td>462</td>
<td>157</td>
</tr>
<tr>
<td>Second</td>
<td>252</td>
<td>2520</td>
<td>458</td>
<td>356</td>
<td>124</td>
</tr>
<tr>
<td>Third</td>
<td>200</td>
<td>2000</td>
<td>364</td>
<td>271</td>
<td>98</td>
</tr>
<tr>
<td>Fourth</td>
<td>159</td>
<td>1600</td>
<td>289</td>
<td>204</td>
<td>78</td>
</tr>
<tr>
<td>Fifth</td>
<td>126</td>
<td>1260</td>
<td>299</td>
<td>150</td>
<td>61</td>
</tr>
<tr>
<td>Sixth</td>
<td>100</td>
<td>1000</td>
<td>182</td>
<td>108</td>
<td>48</td>
</tr>
<tr>
<td>Seventh</td>
<td>79</td>
<td>800</td>
<td>143</td>
<td>74</td>
<td>38</td>
</tr>
<tr>
<td>Eighth</td>
<td>63</td>
<td>640</td>
<td>115</td>
<td>47</td>
<td>30</td>
</tr>
<tr>
<td>Ninth</td>
<td>50</td>
<td>500</td>
<td>91</td>
<td>26</td>
<td>23</td>
</tr>
</tbody>
</table>

Figure 11. (a) 9-level branching network laser machined onto nitride coated silicon and PDMS replica. The process is done at focus with 480 mm s$^{-1}$ scan speed and the etching time is 20 s. (b) SEM image of the network in silicon.
the appropriate choice of the scan speed. Conveniently, we find that the optimal scan speed for effective material removal also produces the smoothest ablation surface. Utilizing a high repetition rate of 200 kHz fs laser system with \( \sim 10 \ \mu \text{J} \) pulse energy, the channel depth varies up to several hundreds of microns with \( \text{Ra} < 2 \ \mu \text{m} \) and MRR \( \sim 0.5 \ \text{mm}^3 \ \text{min}^{-1} \). A 9-level microchannel network is realized as a demonstration. Therefore, the laser technique developed in this study enables the fabrication of microchannels mimicking the feature of the natural vasculature as needed for the development of artificial lungs. The mimicked physiological features are expected to contribute to the further development of artificial lungs. In addition to the artificial lung development, the simplicity and flexibility of our laser technique are beneficial to the developments of MEMS, lab-on-a-chip systems, and other biotechnology applications.

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