The effects of heat treatment on microfluidic devices fabricated in silica glass by femtosecond lasers*

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(Received 29 July 2011; revised manuscript received 6 September 2011)

We fabricated complex microfluidic devices in silica glass by water-assisted femtosecond laser ablation and subsequent heat treatment. The experimental results show that after heat treatment, the diameter of the microchannels is significantly reduced and the internal surface roughness is improved. The diameters of the fabricated microchannels can be modulated by changing the annealing temperature and the annealing time. During annealing, the temperature affects the diameter and shape of the protrusions in microfluidic devices very strongly, and these changes are mainly caused by uniform expansion and the action of surface tension.

Keywords: water-assisted femtosecond laser ablation, microchannels, heat treatment

PACS: 42.62.–b, 47.85.Np, 52.38.Mf **DOI:** 10.1088/1674-1056/21/3/034208

1. Introduction

Microfluidic devices and micro-analysis systems have been applied broadly in the fields of chemical and biological analyses.[1] Many scholars have studied and analysed the interaction between the laser beams and various media.[2−5] With the development of femtosecond laser technology, the femtosecond laser has become a unique tool for fabricating various micro/nano structures[6−9] and diffractive optical elements.[10] The microfluidic devices can also be fabricated in silica glass made by femtosecond laser writing, chemical etching[11] and water-assisted femtosecond laser ablation.[12−14] After being annealed, the internal surfaces of the fabricated microchannels have become much smoother and the cross sections of the microchannels[15,16] are also affected greatly by the heat treatment process. The fabricated microchannel has a uniform diameter and a smooth internal surface, and these are the core techniques of the fabrication. Therefore, heat treatment plays an important role in fabrication. After heat treatment, the diameter and the internal surface roughness are significantly improved, and these are the two most important parameters in identifying the quality of microfluidic devices.

In this study, we fabricated straight microchannels, microfluidic devices and micromixers in silica glass using a water-assisted femtosecond laser. After heat treatment, the diameters of the microchannels and the internal surface roughness of the microfluidic devices are significantly reduced. The size of the diameter can be controlled by changing the temperature and the heat treatment time. The shape variation of the protrusions in the fabricated micromixer can be attributed to the action of the surface tension in the heat treatment process.

2. Experimental technique

A Ti:sapphire regenerative amplified laser system (Coherent Inc) is employed in this experiment. The repetition rate, wavelength and pulse width of the femtosecond laser are 1 kHz, 800 nm and 120 fs, respectively. A circular aperture is employed to select the diameter of the laser beam in order to obtain a good quality focusing point. A mechanical shutter with a response time of 100 ms is used to select the ablation time, and a linearly polarized Gaussian laser beam is focused by an optical microscope (20×/0.45, Nikon). The sample is commercially available silica glass ($70SiO_2 \cdot 20Na_2O \cdot 10CaO$). The glass samples are cut into 6 mm × 10 mm × 2 mm with the upper and lower surfaces polished, and the side surfaces of the silica glass are polished into small angle slopes. The glass sample is placed on a computer-controlled

*Project supported by the Science and Technology Foundation of Heilongjiang Province, China (Grant No. A200912) and the Program of Excellence Team in the Harbin Institute of Technology, China.

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transition stage with a resolution of 100 nm, and the whole experimental fabrication process can be monitored by a charge-coupled device (CCD) in real time.

The silica glass is placed in a container with the distilled water connecting the slopes. The laser beam is focused on the slope surface of the silica glass from the top surface, as shown in Fig. 1(a). We set the repetition rate, the laser pulse energy and the scanning speed to be 1000 Hz, 6 μJ and 200 μm/s so as to efficiently ablate the silica glass into debris. As the silica moves along the designed pattern by controlling the transition stage, the femtosecond laser beam pulses ablate the interface between the glass and the water, as shown in Fig. 1(b). The scanning cell is a rectangularity with a size of 100 μm×60 μm, and we set the moving step of the scanning cell to be 20 μm, as shown in Fig. 1(c). In the fabrication process, the laser-generated bubbles can eject the ablated debris from the fabricated microchannel. Then distilled water can be introduced into the fabricated microchannel for further cleaning. The effects of blocking and redeposition are therefore greatly reduced and we can fabricate long microchannels and complex microfluidic devices in silica glass.

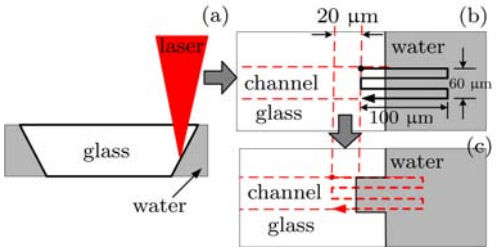


Fig. 1. (colour online) The procedure for fabricating the microchannels: (a) focusing the laser on the slope, and (b) and (c) scanning the interface between the glass and water.

3. Results and discussion

The fabricated microchannels are 500 μm below the surface of the silica glass, as can be seen in Fig. 2(a). The cross sections of these microchannels are shown in Figs. 2(b) and 2(c), respectively, and from the figures we can see that the diameters are uniform from the opening regions to the middle regions. The diameters of the two microchannels are about 50 μm. Then we anneal the microchannels to investigate the effects of heat treatment on the diameter of the microchannels. We put the silica sample in a furnace, then the temperature rises to 750 °C in 40 minutes. When the temperature in the furnace reaches 750 °C, the power of the furnace is shut off. When the temperature in the furnace decreases to room temperature, we can take out the sample. The microchannels, as shown in Fig. 2(d), are more transparent and smoother than those without annealing. The cross sections of the microchannels are shown in Figs. 2(e) and 2(f). We can see that the diameter of the microchannels is reduced to about 30 μm, which means that the heat treatment can affect the diameter of the microchannels greatly. When the silica glass is heated, it will expand uniformly. Therefore, the diameters of the fabricated microchannels will be reduced in the process of heat treatment. At 750 °C the surface silica, including the internal surface silica of the microchannels, will be melted, and then the internal surface of the microchannels becomes smooth and transparent.

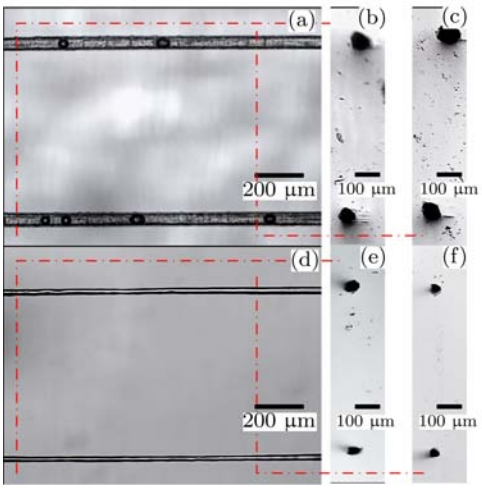


Fig. 2. (colour online) (a) The fabricated microchannels, (b) and (c) the cross sections of the microchannels before heat treatment, (d) the microchannels after heat treatment, and (e) and (f) the cross sections of the microchannels after heat treatment.

In addition, we also investigated the effects of heat treatment on microfluidic devices with microchannels and micro-pools. The microfluidic devices are fabricated at a depth of 500 μm under the surface of the silica glass, as depicted in Fig. 3(a). The diameter of the microchannels is about 50 μm. The micro-pools are 240 μm × 180 μm and 500 μm × 180 μm, respectively. From the enlarged version, as shown in Figs. 3(c) and 3(d), we can observe the obvious traces of laser ablation on the internal surface of the micro-pools. To obtain a smaller diameter of microchannels and a smooth internal surface of the microfluidic devices, we put the fabricated sample into the furnace for

further heat treatment and set the annealing temperature at 755 °C. After annealing, the micro-pools are of the sizes 200 μm × 110 μm and 430 μm × 110 μm, respectively, as shown in Fig. 3(b). The diameter of the microchannels is also reduced to ∼20 μm. Figures 3(e) and 3(f) show that the internal surface roughness of the micro-pools is significantly reduced after heat treatment. The ablated traces on the internal surface of the micro-pools disappear and the microfluidic device becomes transparent in comparison with the sample without heat treatment, as depicted in Fig. 3(a).

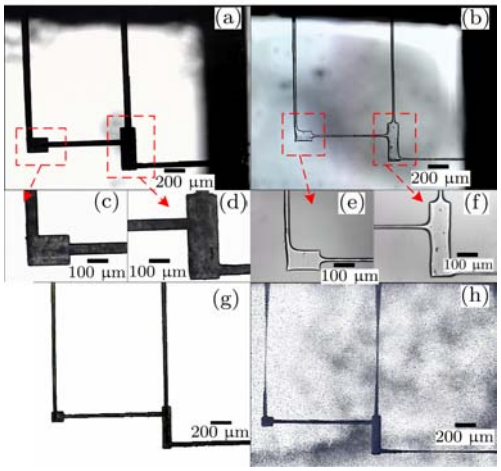


Fig. 3. (colour online) (a) The fabricated microfluidic device 500 μm under the silica surface, (b) the microfluidic device after heat treatment, (c) and (d) the micro-pools before heat treatment, (e) and (f) the micro-pools after heat treatment, (g) the fabricated microfluidic device without cleaning, and (h) the blocked microfluidic device after heat treatment.

From the experimental results we can draw a conclusion that by raising the temperature of the heat treatment, we can fabricate microchannels with smaller diameters. However, if the annealing temperature is 760 °C or the silica anneals for 10 minutes at 750 °C, the microchannels will disappear in this kind of silica glass. Moreover, before annealing we have to clean the microchannels in an ultrasonic bath to completely eject the ablated debris from the microchannels. Otherwise, after annealing the microchannels will be blocked. The uncleaned microfluidic device can be seen in Fig. 3(g), and after annealing at a temperature of 755 °C, the device can be blocked again, as shown in Fig. 3(h). From Fig. 3(h) we can observe that the internal surface of the two micro-pools and the diameter of the connecting microchannels barely change after heat treatment at 755 °C. This is because silica debris is easier to melt than bulk silica glass with the increase in temperature, and the melted silica debris will block the microchannels. As a result, in the two micro-pools and the connecting microchannels, a high-pressure environment is formed. The high pressure will cause a rise in the silica softening point, so the two micro-pools and the connecting microchannels will change little at a temperature of 755 °C.

We further investigate the cross sections of the microfluidic device after annealing. The microfluidic device is shown in Fig. 4(a), and the cross sections of the microchannels can be seen in Figs. 4(b)–4(g). The diameter of the microchannels is significantly reduced after annealing at 755 °C. The diameter of these microchannels is uniform in the opening and middle regions, about 20 μm, and smaller than the straight microchannels annealed at 750 °C. The profiles of the microchannels' cross sections are almost of a circular shape and more regular than the profiles of the straight microchannels' cross sections depicted in Figs. 2(e) and 2(f). From the experimental results we can observe that the effects of the heat treatment are not only caused by the uniform expansion, but also by the action of the surface tension, which has primary effects on the cross section profile of the microchannels. The reason for this is that the higher temperature and longer heating time cause more expanded and melted silica glass, which can change the cross-section profile of the microchannels under the action of the surface tension. The diameter and cross-section profile of microchannels can be modulated by controlling the temperature and the time of heat treatment appropriately.

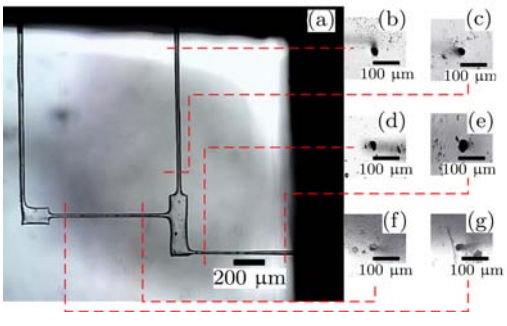


Fig. 4. (colour online) (a) The fabricated microfluidic device after heat treatment, and (b–g) a cross section of the microchannels.

Using water-assisted femtosecond laser ablation, we also fabricated a micromixer with a total length of ∼ 6 mm, which has complex protrusions in the central area 500 μm under the surface of the silica.

In the central area of the micromixer there is a circular structure which is composed of protrusions, as shown in Fig. 5(a). The diameter of the microchannels is about 50 μm. After heat treatment at 755 °C, the micromixer is shown in Fig. 5(b). The size and shape of the protrusions are significantly changed, as depicted in Fig. 5(c). From Figs. 5(d) and 5(e), we can observe that the rectangular protrusions are changed into oval-shaped and circular protrusions. The major and minor axes of the bigger oval-shaped protrusion in Fig. 5(d) are 193 and 65 μm, respectively. We can draw a conclusion by comparing with the protrusions without heat treatment that the action of surface tension plays an important role in modifying the structure of the device in the process of heat treatment at 755 °C. From Fig. 5(e) we can observe that the square protrusions become circular after heat treatment, and the diameter of the circular protrusion is 54 μm, which also strongly proves that the action of surface tension plays an important role in heat treatment. By comparing the sizes of the protrusions before and after heat treatment, we find that the melted silica glass diffuses evenly in the heat treatment process.

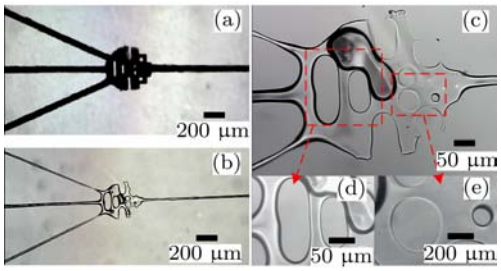


Fig. 5. (colour online) (a) The fabricated micro-mixer at 500 μm below the surface of silica, (b) the fabricated micro-mixer after heat treatment, (c) the critical component of the micro-mixer, (d) the oval-shaped protrusions, and (e) the circular protrusions.

4. Conclusion

In conclusion, heat treatment has a great influence on the quality of fabricated microfluidic devices. The diameter of the microchannels and the internal surface roughness of microfluidic devices are significantly reduced after heat treatment. The change in diameter is mainly caused by the expansion and the change in the shape of the protrusions. The cross-section profile of the microchannels is mainly caused by the action of surface tension. It is expected that this technique will be applied to fabricating high-quality complex microfluidic devices in silica glass for special applications, such as the quantitative experiments in biology and chemistry.

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