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Simple method for fabrication of microchannels in silicon carbide

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By using 800-nm femtosecond laser irradiation and chemical selective etching with hydrofluoric acid, microchannels are fabricated in silicon carbide. The diameter of the microchannel is about $1.5 \,\mu$ m. The morphology of the channel is characterized by using scanning electronic microscopy equipped with an energy dispersive X-ray spectroscopy. The formation mechanism of silicon carbide channels is attributed to the formation of laser-induced structural change zones in silicon carbide and the reaction of the laser-induced structural change zones with hydrofluoric acid. In addition, the influences of the laser average power and scanning velocity on the position of the microchannel are discussed. © 2015 Laser Institute of America.

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Key words: microchannel, chemical etching, femtosecond laser, 6H-SiC

I. INTRODUCTION

Silicon carbide (SiC) is preferred for applications in harsh environments because of its outstanding physical and chemical properties.^{1,2} SiC-based devices are capable of working in harsh temperatures, corrosion, shock, and radiated environment.^{3–5} Especially, electrical, mechanical, and thermal properties of SiC make it a very unique material for biosensor application.⁶ Accordingly, SiC has been used for applications of high temperature pressure sensor⁷ and gas sensor.⁸

Laser micromachining has several prominent advantages over the conventional methods, such as noncontact processing, fast removal rates, and being independent of etch masks.^{9–11} Additionally, due to the fact that the samples can be mounted onto a programmable positioning stage, laser direct writing is capable of fabricating three dimensional micromechanical devices.¹² Femtosecond laser has been proved to be a versatile tool for micromachining transparent material, because it can deposit energy into the material through high-order nonlinear absorption, inducing material change with very high resolution either on surface or in bulk.^{13,14} Specifically, it has been applied to drilling,^{15–18} cutting,¹⁹ and synthesizing²⁰ various types of microstructures and nanostructures in SiC. However, little work has been done in fabricating microchannel in SiC.

Microchannel is very important feature in many applications such as microelectronics, optoelectronics, microchemical systems, microelectromechanical systems (MEMS), microfluidic chip, and biosensor devices. Therefore, there have been many works centering on fabricating microchannel in glass^{21–23} and silicon.^{24,25} One of the most popular methods for fabricating of microchannel in transparent material is the one that combines femtosecond laser and chemical selective etching, in which the femtosecond laser with the ability to deposit high peak power into bulk material via multiphoton absorption without damaging the surface was used to induce laser affected zones which shows very high etching rate to acid solution as compared to the pristine material.⁹ As the

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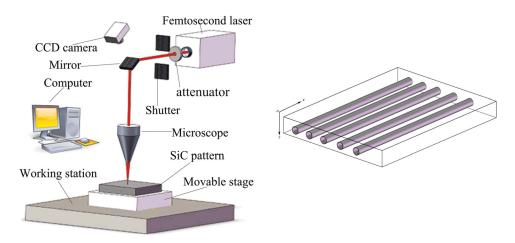


FIG. 1. Schematic diagram of fabricating microchannels in SiC and the scanning paths.

result, after the acid treatment the microchannels were produced. However, since the acid solutions are usually effective to the pristine material too, the diameter of the channel is usually not uniform with the increase of the length.

In this work, we report on the fabrication of microchannel in SiC using laser irradiation and wet etching with hydrofluoric acid (HF). First, laser-induced structural change zones (LISC) were produced with the irradiation of 800-nm femtosecond laser. Then, HF was used to selectively remove the LISC, forming the microchannel in SiC. Subsequently, a scanning electron microscope (SEM) equipped with an energy-dispersive x-ray spectroscope (EDS) was employed to characterize the morphology of the LISC and SiC microchannels.

II. EXPERIMENT DETAILS

Figure 1 shows the experimental setup for fabricating microchannels in SiC. It consists of a femtosecond laser source, an attenuator, a neutral density filter, a mechanical shutter, an *xyz* movable stage, a computer, and a CCD camera. The laser was an amplified Ti: sapphire femtosecond laser system (FEMTOPOWER Compact Pro, Austria) with pulse duration of 150 fs, wavelength of 800 nm, and repetition rate of 1 kHz.

The attenuator provided a convenient way to adjust the laser energy, and the mechanical shutter was employed to control the access of laser source. The movable stage, on which the SiC pattern could be mounted, controlled by computer program, allows us to fabricate on the pattern with high precision. The CCD camera was connected to computer for clear online observation in SiC pattern surface during fabrication. The $10 \times$ microscope objective with numerical aperture (NA) of 0.3 was employed to focus laser onto the surface of 6H-SiC. The diameter of focal spot size of the NA is about $3.2 \,\mu$ m. The polarization direction of the incident laser is parallel to *y*-axis. The inset illustrates the scanning path. In addition, ultrasonic machine was used to accelerate etching process.

In ours experiments, the 6H-SiC pattern with thickness of $350 \,\mu\text{m}$ was used. First, it was cleaned in acetone and deionized water with ultrasonic field for $10 \,\text{min}$, respectively. Then

it was mounted on the movable stage. The laser beam was focused onto the pattern through an optical microscope objective lens. Since the Rayleigh length of the microscope was only 2.8 μ m, the focus point was set on the surface by moving the sample along z direction until the damage on the surface was most severe. Note that a small laser average power should be chosen so that only the laser fluence at focus point could reach the threshold for laser ablation in SiC. The laser scanning path was set to parallel to x axis. During fabrication, the surface of the pattern could be seen either through optical microscope or on the computer screen connected to CCD camera.

After laser irradiation, the pattern was cleaned consecutively with acetone and deionized water for 10 min before being selectively etched with solution of 40 wt. % HF for 1 h. SEM equipped with EDS was employed to study the morphology of the microchannels.

III. RESULTS AND DISCUSSION

Figure 2 shows the morphologies of the SiC microchannel. The microchannel was fabricated in ambient air. The laser average power and scanning velocity were 8 mW and $2 \mu m/s$, respectively. After being irradiated with an 800-nm femtosecond laser, LISC were induced inside the material at the position of about 100 μ m below the surface as shown in Fig. 2(a) (marked with the white box). In order to confirm the continuity of the LISC, the sample was polished to a random position along the scanning direction to observe cross section of the LISC. The diameter of the LISC is about $1.5 \,\mu\text{m}$. The formation of LISC could be interpreted by the microexplosion model.²⁴⁻²⁶ Although 800-nm photons cannot meet 6H-SiC band gap energy (3.1 eV) requirements, bond breaking is induced by multiphoton absorption associated with the extreme intensity. Laser-induced heating and stress could lead to microexplosion at the focus point as long as the laser fluence reaches the threshold. As shown in Fig. 2(b), the LISC may result from the redeposition of explosion debris after the laser irradiation and it is chemically less stable than the pristine SiC. And after being treated with HF, the LISC were removed, forming the channels. The pattern was again polished to a random position to confirm the continuity of the channels. Figure 2(c) shows the channel after acid etching.

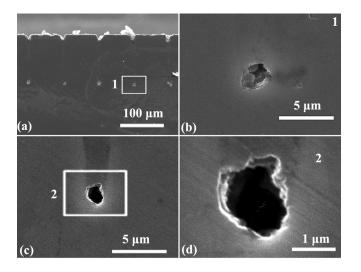


FIG. 2. SEM images of the morphology of microchannel in SiC; (a) after laser irradiation; (b) magnified view of the LISC area marked with rectangle 1 in Fig. 1(a); (c) microchannel after treatment with HF; and (d) magnified views of the microchannel.

The formation of the channels was attributed to the reaction of HF and the LISC. It is noticed that HF only removed the LISC, the surrounding area remains unchanged. As it can be seen from Figs. 2(b) and 2(c), size of the microchannel is almost the size of the LISC of which the diameter is about 1.5 μ m. As compared to the microchannel fabricated in glass using femtosecond laser and chemical etching, there are two advantages of the microchannel in SiC. For one thing, the diameter of microchannel in SiC is much smaller than that in glass (which is about 50–100 μ m). For the other, since the acid solution is only effective to the LISC, the diameter of the channel is not widened with etching time. Hence, the method could be potentially capable of fabricating uniform microchannel of which the diameter is same along the length. It can be seen from Fig. 2(c) that a black line appeared between the surface and the microchannel and the surface. The formation of the line is attributed to the change in structure of SiC caused by the formation of femtosecond filament. During irradiation, laser fluence at this place was not high enough to cause microexplosion or laser ablation, however, dangling bonds could be formed by multiphoton absorption. As a result, the structure of SiC crystal could be changed. Figure 2(d)shows the magnified view of the channel. It is obvious that the microchannel is almost circular in shape. As well known, usually high NA is used for fabricating traverse microchannel in transparent material for a circular cross section.²⁷ However, by using this method, circular shape microchannels could be obtained using NA as small as 0.3. This is of great significance because the microscope with small NA has longer work distance than that of the one with high NA.

To understand the influence of laser power on the microchannel, the experiment with several different laser average powers were conducted while the scanning speed was fixed to be 2 μ m/s. It is observed that there is no evident change in the diameter of the channels as the laser average power increases. Figure 3 shows the dependence of the position of microchannel on the laser average power. The inset shows the microchannel fabricated with laser power of 5 mW. As can be seen, the position of the microchannel increases with the increase of the laser average power. The laser power dependence of the position of the microchannel observed in SiC is different from that of the glass in which the position of the focal point of the laser beam inside the silica sample was less than the geometrical focal point, and decreased with the increase of the laser power.^{24,26} For example, with the laser average power of 2 mW the position is $38 \,\mu\text{m}$; while it is 70 μ m as laser average power is laser average power 5 mW, and with the laser average power of 8 mW the position is 105 μ m. This could be explained by considering the competition between self-focusing and self-defocusing. As the laser power increases, the thermal accumulation increases and the self-defocusing effect becomes more and more dominated. As the result, the laser beam focuses at a deeper point in the material. At the mean time, the change in refractive index of the black line which appeared between the surface and the microchannel could also make the laser beam focus at a deeper point. As mentioned above, the black line was formed by the change in structure of SiC and it contains a large number of dangling bonds, which may lead to reduction of refractive index of the material. As the laser average power increases, laser-induced dangling bonds in SiC caused by multiphoton absorption increase, leading to more change in the structure of SiC. This could reduce the refractive index of the black line, resulting in to smaller mismatch of the air and the material. Consequently, the depth of focal point increases with the increase of laser average power.

In order to study the influence of scanning velocity on the position of the microchannel, the $10 \times$ microscope objective with NA of 0.3 was used. The laser average power was set at 8 mW. And the scanning velocities were set to be 4, 5, 6, and 7 (μ m/s), respectively. It can be seen from Fig. 4 that the position of the microchannel decreases with the increase of scanning velocity. For example, with the scanning velocity of 4 μ m/s the position is 85 μ m; while it is 23 μ m as scanning velocity is 7 μ m/s.

This is because of the pulse accumulating effect. As the scanning velocity increases, the average pulses number on unit area of the SiC samples decreases. This also means that the laser energy accumulated on unit area of the SiC pattern decreases

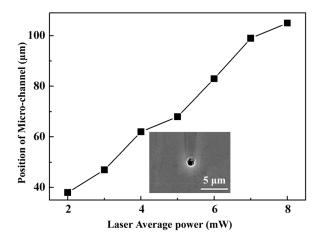


FIG. 3. The influence of laser average power on the position of the microchannel and the inset shows the cross section of microchannel fabricated with laser power of 5 mW.

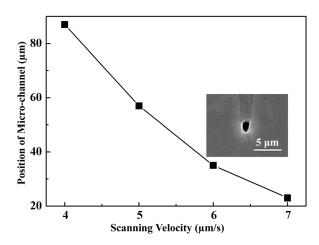


FIG. 4. The influence of scanning velocity on the position of the microchannel and the inset shows the cross section of microchannel fabricated with laser scanning velocity of $6 \mu m/s$.

and the thermal accumulation effect becomes less obvious, resulting in the decreases of the position of the microchannel.

IV. CONCLUSIONS

In conclusion, we fabricated microchannel in SiC using 800-nm femtosecond laser irradiation and chemical etching of HF. First, LISC were induced by femtosecond laser inside SiC. Then, HF was used to remove the LISC, forming the SiC microchannel. The morphology of the channel was characterized by using scanning electronic microscopy. The formation of SiC channel was attributed to the formation of LISC in SiC and the chemical reaction of the LISC with HF acid. The channel is almost circular in shape and its radius is about $1.5 \,\mu$ m. It is obvious that the position of the microchannel could be controlled by changing the laser average power and the scanning velocity. This technique has potential applications in biosensor, microelectronics and microelectromechanical system, and photonics.

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