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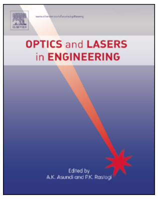

A simple method for well-defined and clean all-SiC nano-ripples in ambient air

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ABSTRACT

Well-defined and clean all-SiC nano-ripples with a period of about 150 nm are produced via the combination of 800-nm femtosecond laser irradiation and chemical selective etching with mixture solution of 65 wt% HNO_3 acid (20 mL) and 40 wt% HF acid (20 mL). The incorporation mechanism of oxygen (O) species into the laser induced obscured nano-ripples is attributed to femtosecond laser induced trapping effect of dangling bonds, while that of chemical etching induced well-defined and clean nano-ripples is assigned to chemical reactions between mixture acid solution and amorphous silicon carbide (SiC) or silicon oxide (SiO_2). Results from EDX analysis show that the incorporated foreign O species (atomic percentages of 9.39%) was eliminated effectively via chemical etching, while the atomic percentages of silicon (Si) and carbon (C) were about 47.82% and 52.18% respectively, which were similar to those of original SiC material. And the influences of laser irradiation parameters on the nano-ripples are also discussed.

1. Introduction

Silicon carbide (SiC) is an attractive semiconductor material which has been applied for devices working in harsh environments because of its outstanding properties such as: high thermal resistance, high mechanical hardness, high chemical inertness, and excellent electrical properties. Meanwhile, as an effective and powerful tool for material processing, femtosecond (fs) laser has been employed to micromachining various kinds of materials, such as: glasses [1,2], metals [3], semiconductors [4–7] and so on. Recent years, with the rapid development of fs laser micromachining, various kinds of microstructures, such as micro-holes [8], micro-spikes [9] and nano-ripples [10–12], have been produced on/in SiC material. And these SiC microstructures could find some practical applications in the fields of microelectromechanical systems [13], optical temperature sensors [14], microelectronics and so on.

Since these microstructures mentioned above are mainly fabricated in ambient air, the fs laser ablation induced debris surrounding or covering these microstructures could not be removed efficiently via usual cleaning method. This makes these structures be obscured. Meanwhile, as the fs laser ablation process is conducted in ambient air, the photoionized O_2 would be incorporated into the microstructures. This makes these structures be not pure any more. Meanwhile, the incorporation of foreign oxygen (O) species may influence the integration quality of SiC semiconductor devices with these structures to the external circuits. Therefore, the fabrication of well-defined and clean all-SiC microstructures has attracted more and more research interests. In order to achieve this purpose, researchers have tried to produce all-SiC microstructures via alcohol-assisted photoetching with fs laser pulses [15]. However, alcohol is a kind of liquid with strong volatility and fluidity, the distance between the planes of SiC substrates and alcohol surface would become shorter and shorter during fs laser irradiation. This would not be beneficial to produce uniform and large area microstructures. Therefore, finding a simple and practical method for all-SiC microstructures with better quality is needed.

In this paper, a simple and practical method for the fabrication of well-defined and clean all-SiC nano-ripples in ambient air has been proposed, in which 800-nm fs laser irradiation and chemical selective etching were combined chronologically. After fs laser irradiation, obscured nano-ripples covered with much debris were

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induced at the laser affected zones (LAZ); via the chemical etching with mixture solution of HNO_3 acid and HF acid, well-defined and clean nano-ripples with a period (Λ) of about 150 nm were produced. In our experiments, a scanning electronic microscope (SEM) equipped with an energy dispersive X-ray spectroscopy (EDX) was employed to characterize the morphology and chemical composition of the nano-ripples respectively. Results from EDX analysis show that the incorporated foreign O species was eliminated effectively via chemical selective etching, while the atomic percentages of silicon (Si) and carbon (C) were about 47.82% and 52.18% respectively, which were similar to those of original SiC material. The incorporation mechanism of foreign oxygen (O) species and the formation mechanism of chemical etching induced well-defined and clean nano-ripples (or the removing mechanism of the laser ablation induced debris) were discussed respectively. Meanwhile, the influences of laser irradiation parameters (such as laser scanning velocity and laser average power) on the nano-ripples were also discussed.

2. Experimental details

2.1. Technological process

The technological process for fabrication of all-SiC nano-ripples is divided chronologically to two steps, which are illustrated in Fig. 1.

2.1.1. Laser irradiation

Prior to the fs laser irradiation, the double polished 6H–SiC wafers, with the orientation of (0001) and thickness of 350 μm. Acetone and ethanol are two kinds of volatility liquid, which are commonly employed to eliminate the contaminant on the sample surface during the ultrasonic cleaning, therefore, for the purpose of eliminating the contaminant on the surface of SiC, the wafers were rinsed successively in an ultrasonic cleaner with acetone, ethanol, and de-ionized water for 15 min, respectively. Then the SiC wafers were mounted horizontally on a computer controlled three dimensional (3D) translation stage (Pro Scan II™) with a step resolution of 40 nm at *x*, *y*, *z* directions respectively, as shown in Fig. 1(a). An amplified Ti: sapphire fs laser system (Coherent Inc., U.S.A.) was employed to provide laser pulses with 50-fs pulse duration, 800-nm central wavelength, and 1-kHz repetition rate. The intensity distribution of the incident laser beam was Gaussian-type. The laser average power (P) could be continuously varied by rotating a variable neutral density filter (NDF), and the access of the laser beam was controlled via a mechanical shutter connected to a computer. The horizontally polarized fs laser beam was focused perpendicularly onto the 6H–SiC wafers by a microscope objective lens (Nikon, NA=0.90, 100 ×), the size of the laser spot ($D=10\pm1$ μm) was generally determined by calculating the average widths of the photoinduced lines via ten single laser scannings on the same SiC substrate. The laser scanning direction (u) was parallel to the polarization direction (E) of the incident laser, which was shown as double blue arrow in Fig. 1(a). The laser scanning velocity (υ), the scanning length (L), the distance between two adjacent scanning lines (Δd) and the number of the scanning lines (M) could be set via the software equipped with the 3D translation stage. In our experiments, the number of the laser pulse accumulated on per laser spot was estimated by the formula $N=(D\bullet f)/\upsilon$, where f was the repetition rate of the incident laser, D was the laser spot, and υ was the laser scanning velocity.

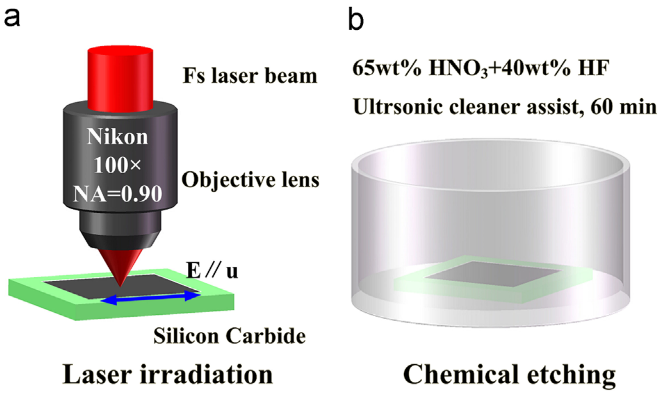


Fig. 1. Technological process for the fabrication of all-SiC nano-ripples. (a) Laser irradiation; (b) chemical etching. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

A charge coupled device (CCD) camera connected with the computer was employed to monitor the laser irradiation process and the focus state of the incident laser on the surface of SiC online. Meanwhile, the whole laser irradiation process was conducted in ambient air, with the temperature and humidity of (25 ± 1) °C and (45 ± 5)%, respectively.

2.1.2. Chemical selective etching

After fs laser irradiation, the 6H–SiC wafers were etched with mixture solution of 65 wt% HNO_3 acid (20 mL) and 40 wt% HF acid (20 mL) in an ultrasonic cleaner for about 60 min, as shown in Fig. 1(b). There were two advantages for the chemical etching performed in ultrasonic environment. Firstly, due to the fast flowing of surrounding solution during ultrasonic shocking process, the byproducts of chemical etching could quickly escape from SiC surface into ambient solutions, which would weaken the solute loading effect during the chemical etching process. This was beneficial to the chemical etching. Secondly, the temperature of ambient solution rose from 25 °C to 50 °C because of the ultrasonic shocking of surrounding water. This would provide appropriate temperature for the chemical reaction between the mixture acid solution and the laser induced amorphous SiC or silicon oxide (SiO_2), and then the velocity of chemical etching would be accelerated. During the chemical etching process, for the purpose of reducing the volatility of mixture acid solution, the beaker which contained the mixture acid solutions was closed with some necessary plastic thin film.

2.2. Characterization of nano-ripples

After laser irradiation or chemical etching, in order to eliminate the laser ablation induced debris, all the SiC wafers were rinsed chronologically with ethanol and de-ionized water in the ultrasonic cleaner for 10 min respectively. And then the morphology and chemical composition of the fs laser induced obscured nano-ripples and the chemical selective etching induced well-defined and clean nano-ripples were characterized by a scanning electronic microscopy (SEM, FEI Quanta 250 FEG Serials) which was equipped with an energy dispersive X-ray spectroscopy (EDX, TEAM™ Serials), respectively.

3. Results and discussion

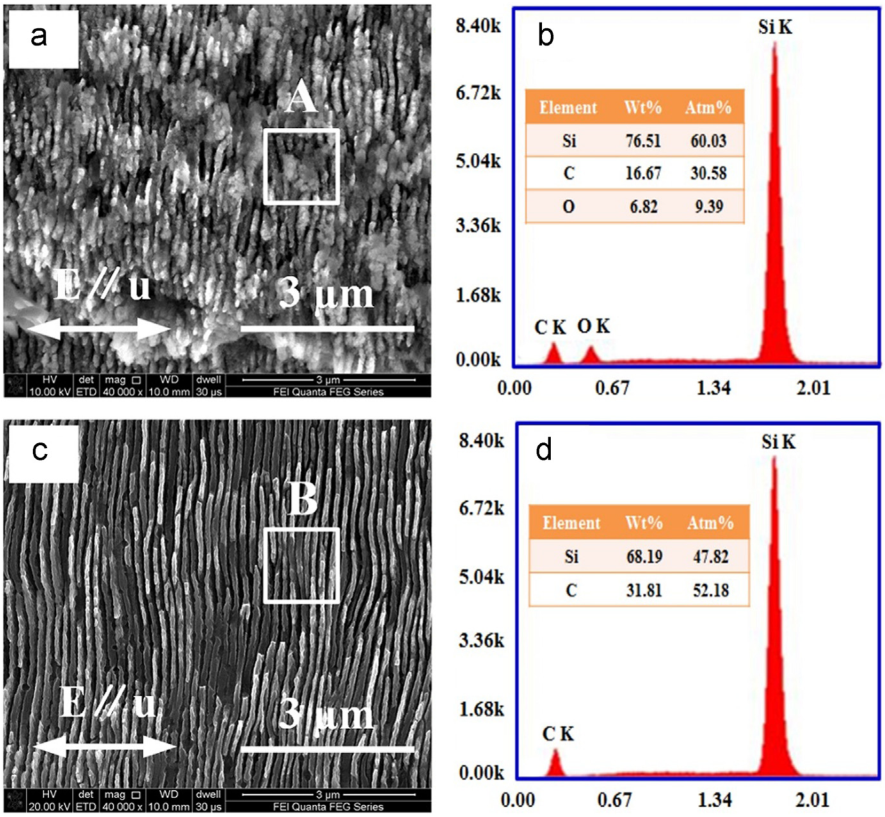
3.1. Formation mechanism of well-defined and clean nano-ripples

Fig. 2 illustrates the morphology and chemical composition of the laser induced obscured nano-ripples and the chemical etching induced well-defined and clean nano-ripples. The laser irradiation parameters were: $P=40.0$ mW, $\upsilon=200$ μm.s^{-1}, $L=100$ μm, $\Delta d=$ 3 μm, $M=5$, $N=50$, NA=0.90 (100 ×); and the chemical etching parameters were: mixture solution of 65 wt% HNO_3 acid (20 mL) and 40 wt% HF acid (20 mL), $t=60$ min, with ultrasonic cleaner assisting.

It can be seen from Fig. 2(a) that obscured nano-ripples covered or surrounded with much debris were produced at the laser affected zones (LAZ). In present reports, there are mainly two popular formation mechanisms for the fs laser induced periodic surface ripples according to the period (Λ) of the nano-ripples: Firstly, if the period (Λ) is closed to the wavelength (λ) of incident laser, the formation mechanism of the laser induced ripples is attributed to the interference between the incident light and the surface scattering wave [16]. Secondly, if the period (Λ) is much smaller than λ, and could be expressed as $\Lambda=\lambda/(2n)$ (n is the refractive index of the material), the formation mechanism of the ripples is assigned to the interference between the incident laser and the surface plasmons [17]. In our experiments, the refractive index (n) of the 6H–SiC material was 2.648, the wavelength (λ) of the incident laser was 800 nm, and the period of the laser induced nano-ripples (150 nm) was closed to the value of $\lambda/(2n)$.Therefore, the formation mechanism of the laser induced obscured nano-ripples could be attributed to the interference between the incident laser and the surface plasmons. During the laser irradiation, the former arrived laser interaction with SiC, and there would be surface plasmons come into being [17]. Because of the interference between the incident laser beam and the surface plasmons, the laser energy accumulated on the SiC surface was modulated, and the nano-ripples formed due to the modulated ablation. It can be also seen from Fig. 2(a) that the orientation of the nano-ripples was perpendicular to the direction of the incident laser polarization. This was in well accordance with the present literatures [10–12,17].

After fs laser irradiation, in order to remove the laser ablation induced debris, the SiC wafers were rinsed chronologically with ethanol and de-ionized water for 10 min respectively. According to the results of EDX analysis illustrated in Fig. 2(b), the main chemical compositions of the obscured nano-ripples were silicon (Si), carbon (C) and oxygen (O) species, with the atomic percentages of 60.03%, 30.58% and 9.39%, and the relative measurement errors of 2.60%, 13.77% and 10.34%, respectively. This indicates that foreign O species was incorporated into SiC material during the process of fs laser irradiation in ambient air. As for the incorporation mechanism of O species into the SiC material, the fs laser induced trapping effect of dangling bonds could be employed to explain it. When the SiC material was irradiated with fs laser pulse, original regular crystal structures of SiC material were destroyed, there would be some defect formed in SiC material, and some crystalline SiC transformed to amorphous SiC [18,19,23–25]. This amorphous SiC was abundant with dangling bonds. These dangling bonds trapped the photoionized O atoms from ambient air into SiC material [18,19], and these trapped O species existed in amorphous SiC material in the form of silicon oxide (SiO_2). When these obscured nano-ripples rich in amorphous SiC and SiO_2 in Fig. 2 (a) were etched in the mixture solution of HNO_3 and HF acid, the debris surrounding or covering these nano-ripples was removed efficiently, and the nano-ripples transformed from obscured to well-defined and clean, with a clear period (Λ) of about 150 nm, which have been shown in Fig. 2(c). The formation mechanism of the chemical selective etching induced well-defined and clean nano-ripples (or the removing mechanism of the laser ablation induced debris) could be explained via the following chemical reactions [20–25]:

$$\mathrm{SiC}\xrightarrow{\text{Laser}}\mathrm{SiO_2}+\mathrm{SiC_{amorphous}} \tag{1}$$



Element	Wt%	Atm%
Si	76.51	60.03
C	16.67	30.58
O	6.82	9.39

Element	Wt%	Atm%
Si	68.19	47.82
C	31.81	52.18

Fig. 2. SEM morphology and chemical composition from EDX analysis of nano-ripples. (a) SEM image of laser induced obscured nano-ripples; (b) chemical composition of area A marked in (a); (c) SEM images of chemical etching induced well-defined and clean nano-ripples; (d) chemical composition of area B marked in (c). Both the laser polarization direction and the scanning direction are marked as double arrows in (a) and (c).

$$\mathrm{SiC_{amorphous}} + 2\mathrm{HNO_3(aq)} + 2\mathrm{H_2O} \rightarrow$$
$$2\mathrm{HNO_2(aq)} + \mathrm{SiO_2(s)} + \mathrm{CO_2(g)}\uparrow + 2\mathrm{H_2(g)}\uparrow \qquad (2)$$

$$\mathrm{SiO_2(s)} + 6\mathrm{HF(aq)} \rightarrow \mathrm{H_2SiF_6(aq)}\uparrow + 2\mathrm{H_2O(aq)} \qquad (3)$$

The variation of SiC material during the fs laser irradiation and chemical etching could also be illustrated clearly in Fig. 3. Amorphous SiC and SiO_2 formed after fs laser irradiation, as shown in reaction (1). When the irradiated SiC was rinsed in the mixture acid solution, because of the strong oxidizability of HNO_3 acid, amorphous SiC was oxidized to silicon oxide (SiO_2), as shown in reaction (2). Meanwhile, the byproduct SiO_2 from reaction (2) and the laser induced SiO_2 reacted with HF acid, and then the flusilicic acid (H_2SiF_6) came into being, as shown in reaction (3). Based on the analysis above, obscured nano-ripples transformed to well-defined and clean nano-ripples after the chemical etching of mixture acid solution. After chemical etching, in order to remove the byproducts from reaction (2) and (3), the SiC wafers were rinsed with ethanol and de-ionized water for 10 min respectively. According to the results of EDX analysis illustrated in Fig. 2(d), the main chemical compositions of the chemical etching induced well-defined and clean nano-ripples were Si and C, with the atomic percentages of 47.82% and 52.18%, and the relative measurement errors of 1.47% and 13.72% respectively, which were close to those of original SiC material. Therefore, these nano-ripples could be called as all-SiC nano-ripples. And these nano-ripples would be better candidates for SiC nano-gratings or antireflection structures.

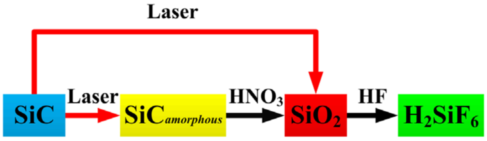


Fig. 3. Material variation of SiC during the fs laser irradiation and chemical selective etching.

The reasons that using the mixture solution of HNO_3 acid and HF acid in chemical etching process could be explained as following: Firstly, if only the HNO_3 acid was used, the fs laser induced amorphous SiC from reaction (1) could be removed, however, the byproduct SiO_2 from reaction (2) and the laser induced SiO_2 from reaction (1) left on substrates; this would make the nano-ripples contain foreign O species. Secondly, if only the HF acid was used, the laser induced SiO_2 from reaction (1) could be eliminated, however, the fs laser induced amorphous SiC from reaction (1) remained in the nano-ripples; this would make the nano-ripples be not pure any more. Therefore, mixture solution of HNO_3 acid

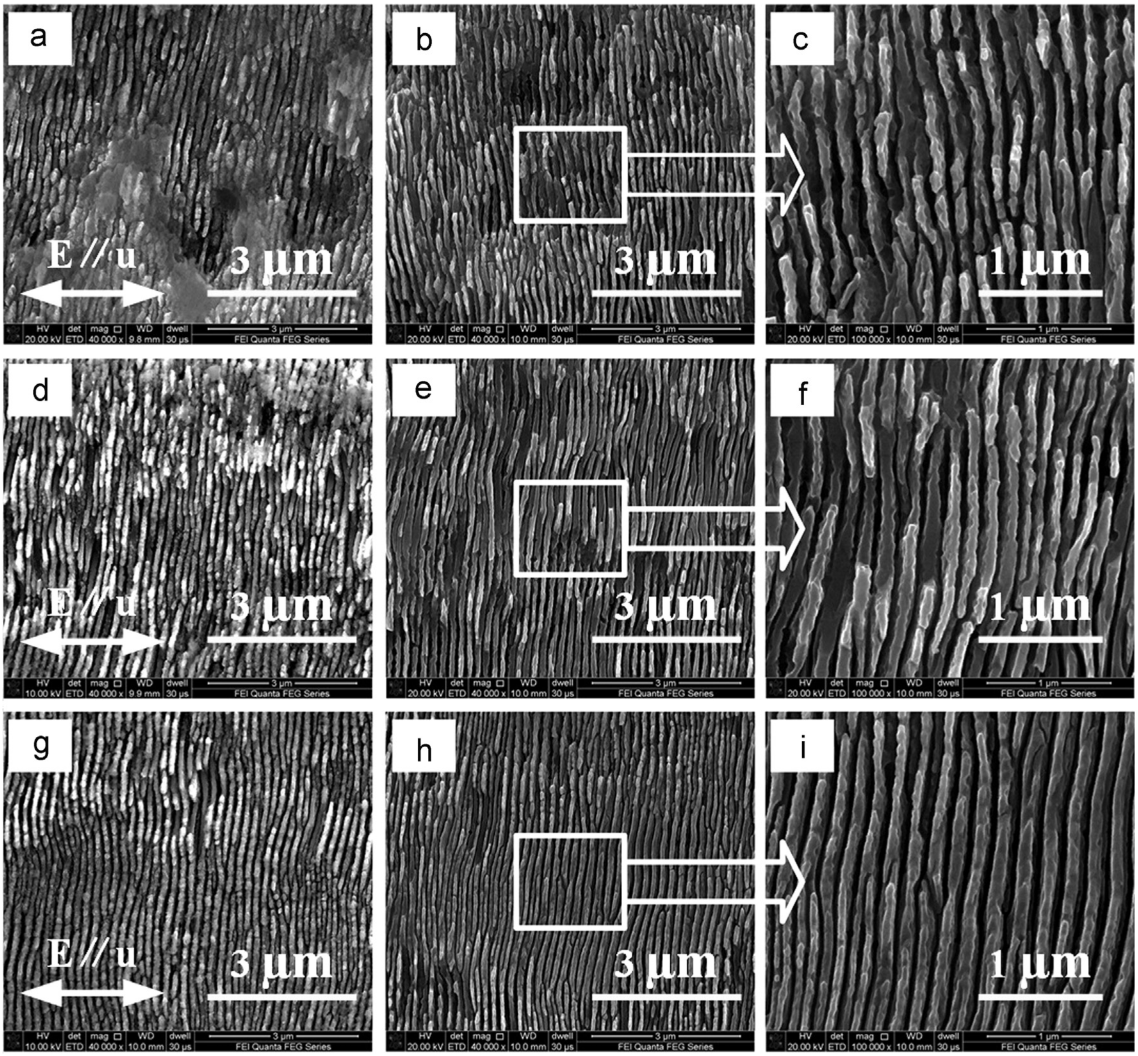


Fig. 4. SEM images of SiC nano-ripples at different laser scanning velocities. (a) $\upsilon = 100\ \mu\mathrm{m\ s^{-1}}$, before chemical etching; (b) $\upsilon = 100\ \mu\mathrm{m\ s^{-1}}$, after chemical etching; (c) detailed image of (b); (d) $\upsilon = 300\ \mu\mathrm{m\ s^{-1}}$, before chemical etching; (e) $\upsilon = 300\ \mu\mathrm{m\ s^{-1}}$, after chemical etching; (f) detailed image of (e); (g) $\upsilon = 500\ \mu\mathrm{m\ s^{-1}}$, before chemical etching; (h) $\upsilon = 500\ \mu\mathrm{m\ s^{-1}}$, after chemical etching; (i) detailed image of (h). Both the laser polarization direction and the scanning direction are marked as double arrows in (a), (d) and (g).

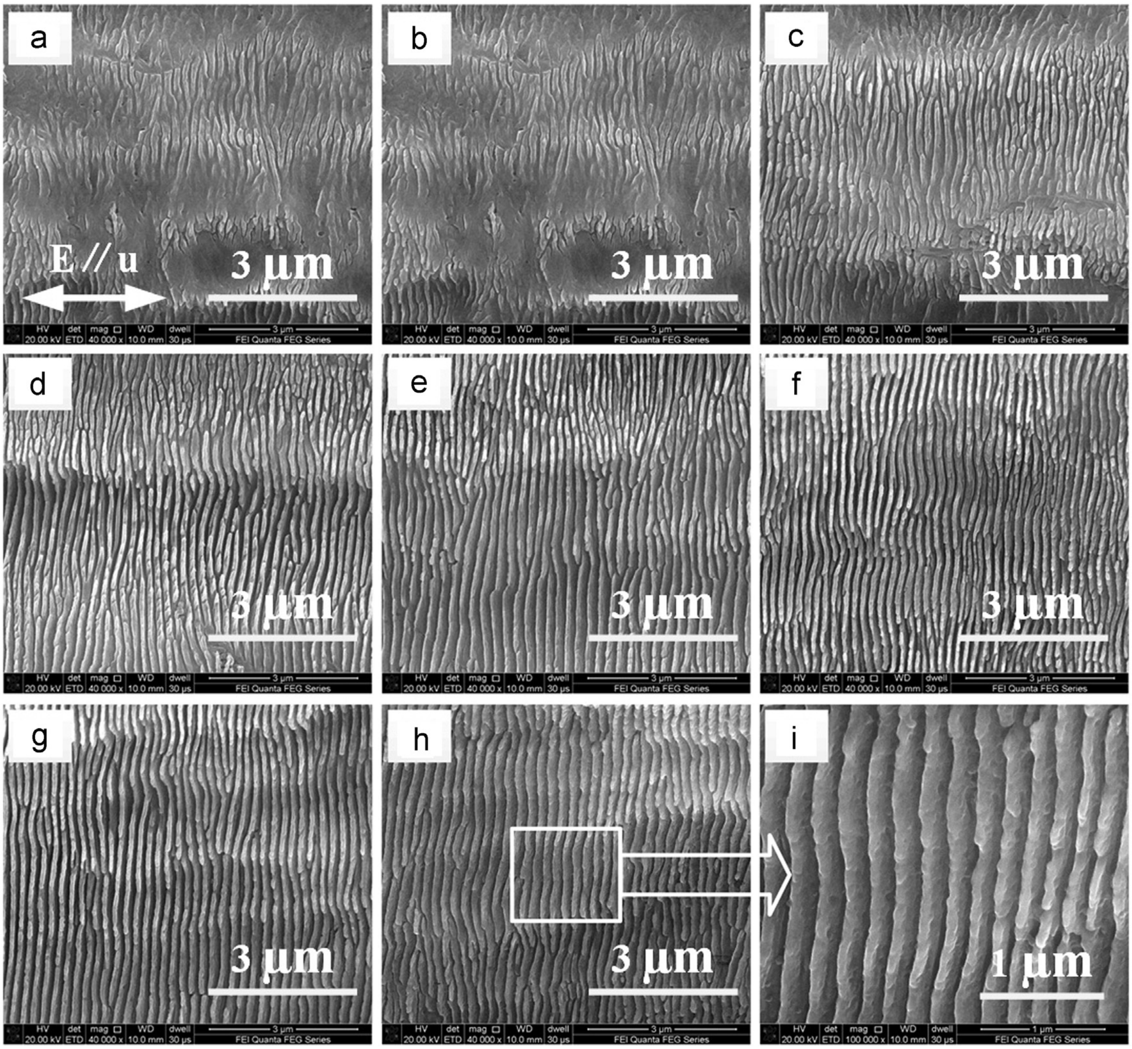


Fig. 5. SEM images of SiC nano-ripples at different laser average powers. (a) $P=10.0$ mW; (b) $P=14.0$ mW; (c) $P=18.0$ mW; (d) $P=22.0$ mW; (e) $P=26.0$ mW; (f) $P=30.0$ mW; (g) $P=35.0$ mW; (h) $P=40.0$ mW; (i) detailed image of (h). Both the laser polarization direction and the scanning direction are marked as double arrows in (a).

and HF acid was a better choice for the fabrication of well-defined and clean all-SiC nano-ripples. To the best of our investigation, there have not been any reports on the well-defined and clean all-SiC nano-ripples fabricated via this simple method. This method has the advantages as following: Firstly, the fs laser irradiation was performed in ambient air, no special gases or instruments were employed. This makes the experimental process be simpler and more practical than those conducted in special gases. Secondly, the mixture acid solution merely reacted with the laser affected zones (LAZ), original SiC material which was not irradiated by fs laser was not influenced in the chemical etching process. This suggests the selectivity of this method is high. Thirdly, since the formation mechanism of the well-defined and clean nano-ripples (or the removing mechanism of the laser ablation induced debris) was the chemical selective reaction between mixture acid solution and amorphous SiC materials or laser induced SiO_2, this method could also be applied to process SiC materials of other types or orientations, such as 3C–SiC, 4H–SiC and 6H–SiC. This indicates the flexibility of this method is high. Finally, the main chemical compositions of chemical etching induced well-defined and clean nano-ripples were Si and C, and the atomic percentages of Si and C were closed to those of original SiC material. This suggests no foreign species remains in the nano-ripples, which would improve the integration quality of SiC devices with these nano-ripples to the external circuits. Due to the high selectivity, high flexibility and high purity of the produced structures, we predict that this simple and practical method would be a better reference for the fabrication of all-SiC structures. Using this method, different kinds of micro/nano-microstructures, such as: through holes [23], microchannels [24], and deep-subwavelength nano-hole arrays [25], have been successfully produced in or on SiC material.

According to the formation mechanism of well-defined and clean nano-ripples discussed above, we predict that the quality of the nano-ripples are influenced by the fs laser irradiation parameters (such as the laser scanning velocity, the laser average power and the distance between two adjacent scanning lines) and the chemical etching parameters (such as the respective percentage of HNO_3 acid and HF acid in the mixture solution, the concentration of HNO_3 acid and HF acid, the chemical etching time). In the following section, we mainly discuss the influences of laser irradiation parameters on the induced nano-ripples.

3.2. *Influences of laser irradiation parameters on nano-ripples*

3.2.1. *Laser scanning velocity*

Fig. 4 illustrates the morphology of the nano-ripples produced at different laser scanning velocities. The laser irradiation parameters were: $P=40.0$ mW, $\upsilon=100\ \mu\text{m s}^{-1}$, $300\ \mu\text{m s}^{-1}$, $500\ \mu\text{m s}^{-1}$, $L=100\ \mu\text{m}$, $\Delta d=3\ \mu\text{m}$, $M=5$, $N=100$, 33.3, 20, NA$=0.90$ (100×); and the chemical etching parameters were: mixture solution of 65 wt%

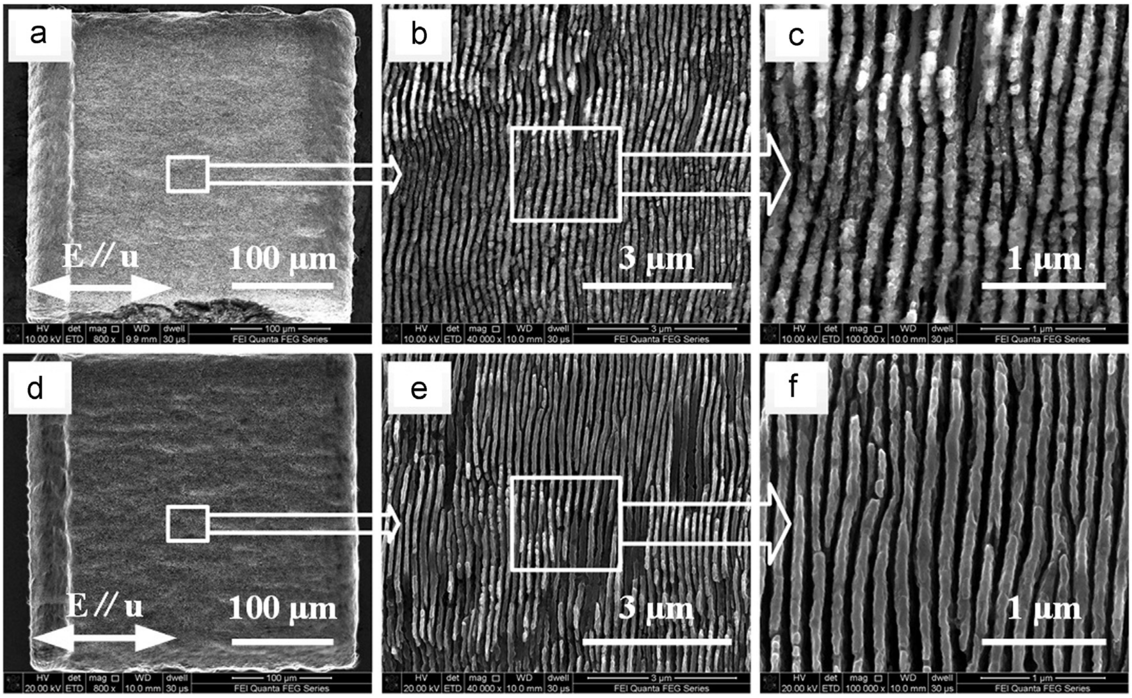


Fig. 6. SiC nano-ripples with large area. (a) Obscured nano-ripples, before chemical etching; (b) detailed image of (a); (c) detailed image of (b); (d) well-defined and clean nano-ripples, after chemical etching; (e) detailed image of (d); (f) detailed image of (e). Both the laser polarization direction and the scanning direction are marked as double arrows in (a) and (d).

HNO_3 acid (20 mL) and 40 wt% HF acid (20 mL), $t=60$ min, with ultrasonic cleaner assisting.

It can be seen from Fig. 4 that the period (Λ) of the nano-ripples keeps invariant, while the continuity of single nano-ripple becomes better with the increase of the laser scanning velocity. This would be related with the scattering effect of the laser induced debris to the incident laser pulses. Since the laser energy accumulated on unit area decreased with the increase of the laser scanning velocity, the laser ablation induced debris would be fewer under higher scanning velocity, and the scattering effect would be weaker at higher velocity; therefore, the continuity of single nano-ripple would be better. This could be seen clearly from Fig. 4(c), (f) and (i).

3.2.2. *Laser average power*

Fig. 5 illustrates the morphology of the nano-ripples produced at different laser average powers. The laser irradiation parameters were: $P=10.0$ mW, 14.0 mW, 18.0 mW, 22.0 mW, 26.0 mW, 30.0 mW, 35.0 mW, 40.0 mW, $\upsilon=500\ \mu m\ s^{-1}$, $L=100\ \mu m$, $\Delta d=3\ \mu m$, $M=5$, $N=20$, NA=0.90 (100 ×); and the chemical etching parameters were: mixture solution of 65 wt% HNO_3 acid (20 mL) and 40 wt% HF acid (20 mL), $t=60$ min, with ultrasonic cleaner assisting.

It can be seen from Fig. 5 that the continuity of single nano-ripple and the uniformity of the nano-ripples become better with the increase of laser average powers. This would be related with the overlap extent of two adjacent scanning lines. Since the intensity distribution of incident laser beam was Gaussian-type, the laser intensity decreased from the center to the periphery area along the transverse direction. This makes the nano-ripples be produced only at the central area of single scanning line. Under the same distance (Δd) between two adjacent scanning lines, the higher of the laser average fluences, the better of the continuity of single nano-ripple, and then the better of the uniformity of the nano-ripples.

In the above section, we just tried to fabricate all-SiC nano-ripples using this simple and practical method, therefore, the area (A) of the laser affect zones (LAZ) was so small, and the chemical etching time (t) was so long. Results from SEM and EDX analysis have proved it was a feasible method for the fabrication of well-defined and clean all-SiC nano-ripples. However, the fabrication efficiency was so low. We speculated that the efficiency of fabricating the nano-ripples would be further improved via increasing the laser scanning velocity or reducing the chemical etching time. In the following section, we tried to fabricate well-defined and clean all-SiC nano-ripples of larger area (A) with higher scanning velocity and shorter etching time.

3.3. *Fabrication of large area well-defined and clean nano-ripples*

Fig. 6 illustrates the morphology of nano-ripples with larger area (A) and shorter etching time (t). The laser irradiation parameters were: $P=40.0$ mW, $\upsilon=500\ \mu m\ s^{-1}$, $L=300\ \mu m$, $\Delta d=3\ \mu m$, $M=100$, $N=20$, $A=300\ \mu m \times 300\ \mu m$, NA=0.90 (100 ×); and the chemical etching parameters were: mixture solution of 65 wt% HNO_3 (20 mL) and 40 wt% HF (20 mL), $t=10$ min, with ultrasonic cleaner assisting.

It can be seen from Fig. 6 that well-defined and clean nano-ripples with large area have been successfully produced on 6H–SiC material. With fs laser irradiation, obscured nano-ripples covered or surrounded with much debris were produced at the laser affect zones (LAZ), which have been shown in Fig. 6(a)–(c). After the chemical selective etching of mixture acid solution of HNO_3 acid and HF acid, the debris were eliminated efficiently, therefore, the nano-ripples were smoother, as shown in Fig. 6(d)–(f). It should be noticed that the chemical etching time was just 10 min, and the main chemical compositions of these nano-ripples were Si and C. This indicates that the fabrication efficiency was improved.

In our experiments, the influences of fs laser irradiation parameters on the nano-ripples have been discussed. Under appropriate combination of the laser parameters, nano-ripples of high quality would be produced. Even so, the fabrication of well-defined and clean all-SiC nano-ripples should also consider the influences of chemical etching parameters. And this will be the further works we are considering to do.

4. Conclusions

In conclusion, the combination of 800-nm fs laser irradiation and chemical selective etching with mixture solution of HNO_3 acid and HF acid is a simple and practical method for fabrication of well-defined and clean all-SiC nano-ripples. After chemical selective etching, the incorporated foreign O species (atomic percentages of 9.39%) was eliminated effectively via chemical etching, while the atomic percentages of silicon (Si) and carbon (C) were about 47.82% and 52.18% respectively, which were similar to those of original SiC material. The incorporation mechanism of foreign O species into SiC material was fs laser induced trapping effect of dangling bonds, and that of the chemical etching induced well-defined and clean nano-ripples was the chemical reaction of mixture acid solution with the laser induced amorphous SiC and SiO_2. Furthermore, the laser irradiation parameters also influenced the quality of the nano-ripples. Due to the high selectivity, high flexibility, high purity of the induced microstructures of this method, we predict it would be a promising method for fabrication of all-SiC advanced functional structures or MEMS devices.

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