



Fabrication of two-dimensional periodic structures on silicon after scanning irradiation with femtosecond laser multi-beams



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ABSTRACT

Two-dimensional (2D) periodic structures were fabricated on silicon surfaces by femtosecond laser irradiation in air and water, with the assistance of a microlens array (MLA) placed in the beam's path. By scanning the laser beam along the silicon surface, multiple grooves were simultaneously fabricated in parallel along with smaller laser-induced ripples. The 2D periodic structures contained long-periodic grooves and perpendicular short-periodic laser-induced ripples, which had periods of several microns and several hundred nanometers, respectively. We investigated the influence of laser power and scanning velocity on the morphological evolution of the 2D periodic structures in air and water. Large-area grid-like structures with ripples were fabricated by successively scanning once along each direction of the silicon's surface, which showed enhanced optical absorption. Hydrofluoric acid was then used to remove any oxygen and laser-induced defects for all-silicon structures.

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1. Introduction

Femtosecond lasers are a notably useful tool for fabricating surface and inner microstructures of various materials [1,2]. Over the past thirty years, femtosecond laser-induced periodic surface structures on metals and semiconductors have been extensively studied [3–10]. The period of these laser-induced ripples often ranges from a small value (less than 1/4 of the wavelength) to approximately the incident wavelength. The orientation, period, and morphology of the ripples are influenced by the laser processing parameters such as laser power and scanning velocity, processing environment, and the properties of the material itself. Moreover, many studies have been performed on artificial one-dimensional (1D) surface grooves fabricated by direct laser writing or laser interference methods [11,12]. However, few studies combining the fabrication of 1D surface grooves and laser-induced ripples to construct two-dimensional (2D) periodic structures on the surface have been undertaken. The fabrication of these 2D periodic structures is a promising approach for producing colorized metals or semiconductors [13–15] and surfaces with unique wettability properties [16–18]. They may also have potential applications in

the fabrication of bio-inspired structures [19] and photonic crystals [20].

In the specific realm of solar power technology, these 2D periodic structures may also be used in making anti-reflection surfaces in solar cells. In the past five years, researchers have intentionally fabricated multiscale surfaces containing both micro- and nanostructures, taking both optical performance and electrical performance into consideration [21,22]. Results show that these micro/nano combined structures can effectively reduce the thickness of black silicon on the cell surface, thereby reducing the near-surface recombination of carriers [23]. Compared with single-scale structures, the micro/nano combined structures have improved electrical performance. Again, silicon anti-reflection surfaces with either laser-induced ripples or 1D surface grooves have been fabricated [24,25], but surfaces with both types of structures have not been reported.

In 2008, researchers fabricated a combined microstructure on ZnO containing these two types of periodic structures by femtosecond laser interference [26,27]. Unfortunately, the overlapping region of two femtosecond laser beams was often no more than several hundreds of microns in diameter; it is quite difficult to fabricate large-area uniform structures using this interference method.

In this study, we fabricated 2D periodic microstructures in one step by using femtosecond laser scanning, with the assistance of a microlens array (MLA). We combined grooves that had a period of several microns with femtosecond laser-induced ripples that

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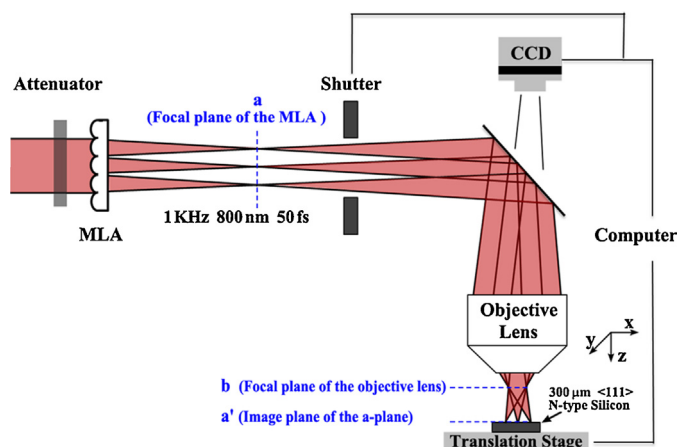


Fig. 1. Experimental setup for the femtosecond laser fabrication process.

had a period of several hundred nanometers (537 nm and 124 nm). 2D periodic microstructures were fabricated in a large area of up to $1\text{ cm} \times 1\text{ cm}$, and the scanning method helped ensuring the uniformity of the structures over long distances. Compared with lithography and reactive-ion etching methods, this method is a one-step process and is free of organic or metal contaminations. The period of grooves can be controlled by changing the objective lens or the relative positions of the MLA and the sample. The laser-induced ripples were regulated by controlling the conditions of the laser irradiation of the surface and the environmental media. We fabricated the structures at different laser powers and scanning velocities, and studied the evolution of the 2D periodic structures to find the proper fabrication conditions. The structures were fabricated in air and water to study the influence of environmental media on the ripples. Large-area grid-like structures with ripples were fabricated by successively scanning once along each direction of the silicon's surface. The increase in the optical absorption of the grid-like structure was measured. To remove the laser-induced defects and make all-silicon surfaces, we ultrasonically etched the samples by hydrofluoric (HF) acid. The morphology and optical performance of the etched surface were also tested.

2. Experimental setup

In our experiment, the samples were firstly textured with 2D periodic or grid-like structures by scanning irradiation with femtosecond laser multi-beams. Secondly, the samples were cleaned or etched for observations and measurements.

The experimental setup for parallel fabrication of 2D periodic microstructures is illustrated in Fig. 1. The 800 nm femtosecond laser was generated by a regeneratively amplified Ti:sapphire laser system, with a duration of 50 fs and a repetition rate of 1 kHz. The laser energy and pulse number were controlled by a variable optical attenuator and a mechanical shutter, respectively. After the attenuator, an MLA made of silica was used to split the femtosecond laser spatially and focus the laser beams to multiple foci in its focal plane, namely the a-plane in Fig. 1. The microlenses were square arranged with a period of $150\text{ }\mu\text{m}$ and a focal length of 5.3 mm. The laser beam was slightly less than 4 mm in diameter before the MLA, which passed through about 450 microlenses.

After passing through the MLA, the laser beams were gathered by a microscope objective lens. The objective lens was considered to image the focal plane of the MLA (the a-plane), and the corresponding image plane was the a'-plane in Fig. 1. In another word, each of the multi-beams after the MLA was focused on the a'-plane by the objective lens. Compared with the multiple foci in the a-plane, the multiple foci in the a'-plane had a shorter period and

a smaller dimension. We experimentally found that the period of multiple foci ($150\text{ }\mu\text{m}$) in a-plane was reduced by about 42 times ($3.59\text{ }\mu\text{m}$) when imaged in the a'-plane by a $10\times$ objective lens. The samples were placed in the a'-plane to be irradiated by femtosecond laser. The $300\text{-}\mu\text{m}$ thick, n-type silicon wafer with a $\langle 111 \rangle$ crystal orientation was fixed on three-dimensional translation stage with a precision of 40 nm in each direction. The 2D periodic microstructures were fabricated by scanning the femtosecond laser along the x or y direction. The directions of grooves were the same as the scanning directions, and the period of the grooves was determined from the period of multiple foci in the a'-plane, which could be regulated by changing the MLA or the objective lens, or the distance between them. The laser beam size determined the width of the textured area and the number of grooves in one scanning step. The scanning process was actuated by the stage and velocities were controlled by a computer. Grid-like structures with ripples could also be obtained by scanning successively in the x and y directions. The 2D periodic structures were fabricated in air and water at various laser powers and scanning velocities. The powers ranged from 10 mW to 30 mW and the scanning velocities ranged from $5\text{ }\mu\text{m/s}$ to $600\text{ }\mu\text{m/s}$.

After laser irradiation, samples were ultrasonically cleaned in alcohol and deionized water for 20 min each to remove debris from the silicon surface. To remove the laser-induced silicon oxides from the surface for all silicon structures, HF acid was used, which selectively reacts with SiO_x without etching the crystal silicon [28]. The etching process was carried out in an ultrasonic bath at room temperature. The morphologies of the microstructures were characterized by a scanning electron microscopy (SEM) and the chemical composition was analyzed by energy dispersive X-ray spectroscopy (EDX) analyzing. The optical reflectance and transmittance of samples were measured by a UV-vis-NIR spectrophotometer equipped with an integrated sphere (SHIMADZU UV-3600) to calculate the absorbance.

3. Results and discussion

3.1. Morphologies of the 2D periodic structures

Fig. 2(a) and (c) show 24 parallel grooves with detailed morphologies fabricated through one scanning step in air. The objective lens used here was a $10\times$ lens with a numerical aperture of 0.30. In Fig. 1, the distance between a-plane and b-plane was about 97.9 cm, including a barrel length of 5 cm and a working distance of 1.75 cm. The distance between b-plane and a'-plane was $540\text{ }\mu\text{m}$. The irradiation area in the a'-plane had a diameter of about $81.2\text{ }\mu\text{m}$. And the diameter of each multiple foci in the a'-plane was estimated to be about $0.72\text{ }\mu\text{m}$. Fig. 2(b) shows the two-dimensional Fourier-transform (2D-FT) of Fig. 2(a), which reveals the groove period of $3.59\text{ }\mu\text{m}$ and the ripple periods between 484 nm and 592 nm. The width of the valleys was approximately 700 nm [Fig. 2(c)], which was influenced by the laser spot dimension of the multiple foci in the a'-plane. Ripples with an average period of approximately 537 nm stretched across the grooves. The direction of the ripples was almost perpendicular to the horizontal polarization direction of laser. The ripples were similar to the typical laser-induced ripples on silicon in air reported in previous works [3]. The structures shown in Fig. 2(d) were fabricated by a circular polarized laser in water. We replaced the $10\times$ objective lens by a $20\times$ objective lens and obtained a smaller period (approximately $1.83\text{ }\mu\text{m}$) of the grooves, as shown in Fig. 2(d). The distance between the a-plane and the b-plane (Fig. 1) was about 97.6 cm in this case, including a barrel length of 6 cm and a working distance of 0.45 cm. We moved the samples along z-axis and found that the distance between b-plane and a'-plane was $142\text{ }\mu\text{m}$. The laser-induced ripples were

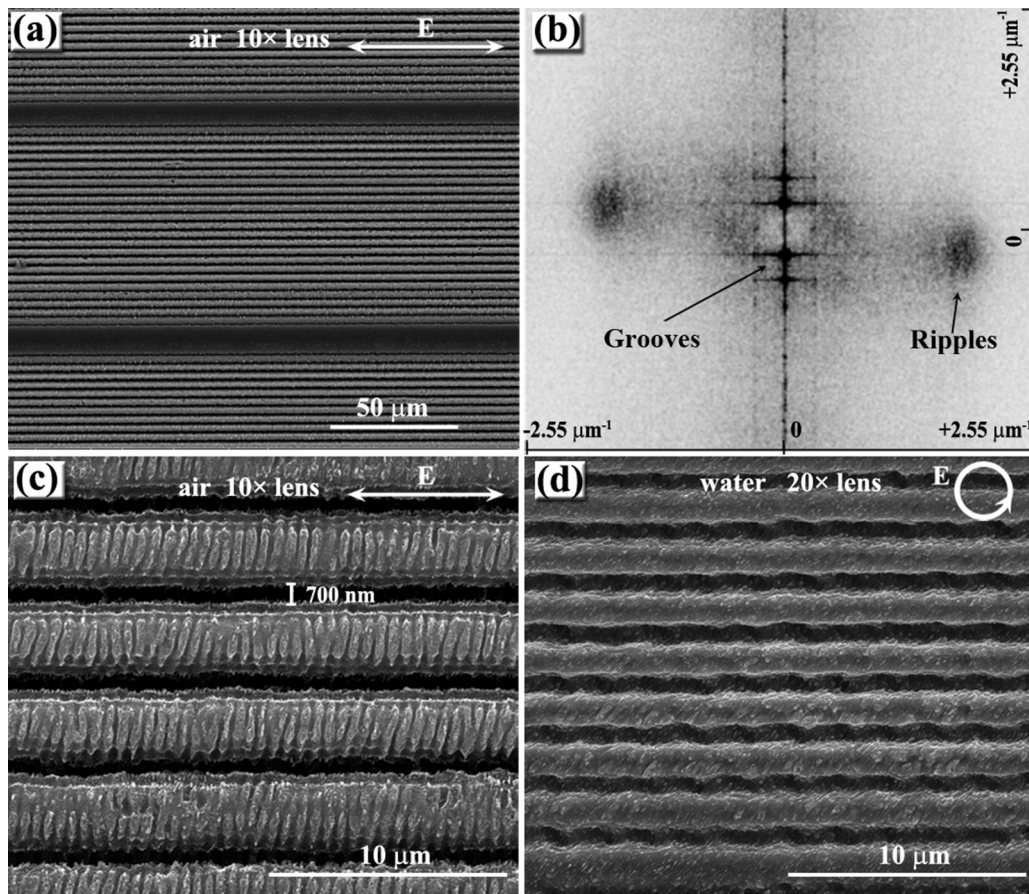


Fig. 2. SEM and 2D-FT images of the 2D periodic structures. (a) Overview of the structures fabricated in air at a laser power of 30.4 mW and a scanning velocity of 150 $\mu\text{m/s}$. The corresponding 2D-FT image was shown in Fig. 2(b). (c) Enlarged view of the structures shown in Fig. 2(a). (d) Parallel grooves fabricated by a circular polarized laser in water at a power of 31.2 mW and a velocity of 100 $\mu\text{m/s}$. The corresponding laser polarization direction was marked on each SEM image.

discontinuous and appeared as particles with a width of approximately one hundred nanometers. The circular polarization of femtosecond laser resulted in the discontinuity of ripples. However, it did not influence the direction of the grooves. Different from the grooves reported in some previous studies, whose directions were parallel to the laser polarization directions and periods were associated to inhomogeneous energy depositions [29,30], the groove directions and periods in this study were determined from the scanning directions, and the settings of the MLA and objective lens, respectively.

3.2. Morphological evolution of the 2D structures in air and water

To find the influence of the laser parameters and the environmental media on the formation of the 2D periodic structures, we fabricated the structures at different laser powers and scanning velocities in air and water, using the 10 $\times$ objective lens. Fig. 3(a)–(h) shows the results for the structures fabricated in air, and Fig. 3(i)–(l) show those for the structures fabricated in water. Fig. 3(a)–(d) show the evolution of the 2D periodic structures with increasing laser powers from 10.3 mW to 30.2 mW in air at a scanning velocity of 500 $\mu\text{m/s}$. When the laser beam had a low power as 10.3 mW [Fig. 3(a)], sequences of nanoholes were induced with a period similar to the period of the laser-induced ripples. When the laser power was slightly higher as 15.5 mW [Fig. 3(b)], shallow and short ripples appeared from the nanoholes. It is likely that the sequence of nanoholes was the primary structure that finally evolved into ripples [13]. The nanoholes were distributed in the regions where the

shaped laser energy was concentrated and therefore the nanoholes were wider and deeper than the ripples. The nanoholes became deeper to finally form grooves as the laser power increased further. Meanwhile, the shallow ripples became longer and deeper and covered the area between the grooves [Fig. 3(c) and (d)]. Fig. 3(e)–(h) show the evolution of the 2D periodic structures with increasing scanning velocities from 5 $\mu\text{m/s}$ to 600 $\mu\text{m/s}$, at a laser power of 30.1 mW. The laser energy deposition would increase as the scanning velocity decreased, giving rise to an increasing thermal effects. Thus a sufficiently low scanning velocity as 5 $\mu\text{m/s}$ resulted in the melting of the grooves and ripples, as shown in Fig. 3(e). As the scanning velocity was increased, grooves and ripples gradually became distinct. A sufficiently high velocity of 600 $\mu\text{m/s}$ also had a negative influence on ripples, as shown in Fig. 3(h). It was reported that the formation of ripples on silicon surfaces is a threshold effect which depends on the laser energy deposition [31,32]. A sufficient high scanning velocity would result in a too low energy deposition for inducing the ripples on the surface.

Fig. 3(i)–(l) show the 2D periodic structures fabricated in water. The effects of laser power and scanning velocity on the 2D periodic structures in water were similar to those seen in the structures fabricated in air. Narrow grooves on silicon with a valley width of only 500 nm could be fabricated simultaneously by scanning at a relatively high velocity of 300 $\mu\text{m/s}$, and there were no ripples on the silicon surface, as shown in Fig. 3(i). Fig. 3(j) and (k) shows that the grooves became more defined as the laser power increased from 10.2 mW to 30.2 mW. The laser-induced ripples in water had periods between 104 nm and 143 nm (2D-FT method). The average

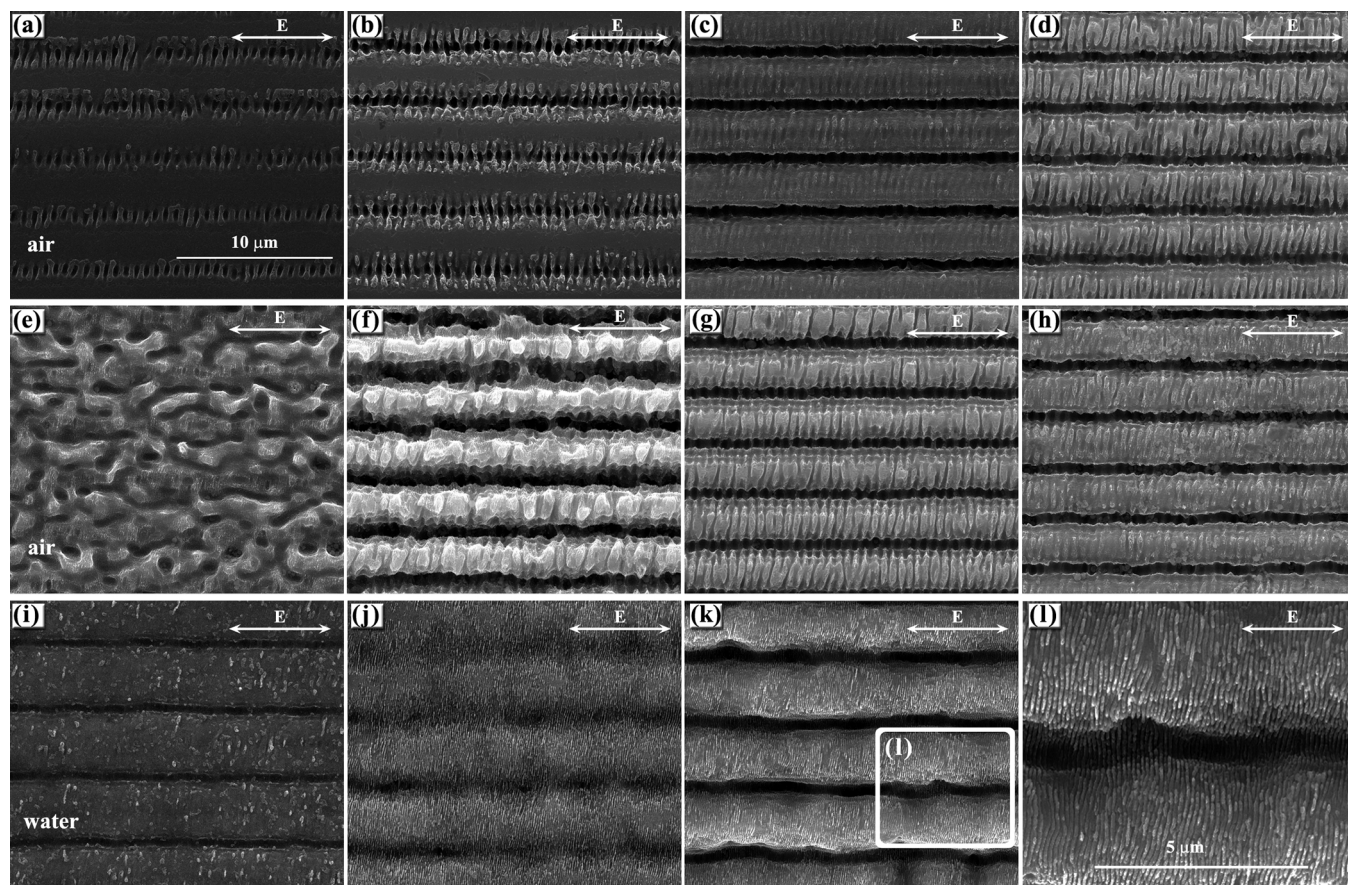


Fig. 3. Evolution of the 2D periodic structures. (a)–(d) are structures fabricated in air at a scanning velocity of 500 $\mu\text{m/s}$ and increasing laser powers of (a) 10.3 mW (b) 15.5 mW (c) 23.1 mW and (d) 30.2 mW. (e)–(h) are the structures fabricated in air at a laser power of 30.1 mW and scanning velocities of (e) 5 $\mu\text{m/s}$ (f) 80 $\mu\text{m/s}$ (g) 250 $\mu\text{m/s}$ and (h) 600 $\mu\text{m/s}$. (i)–(l) are the structures fabricated in water with laser parameters of (i) 30.2 mW, 300 $\mu\text{m/s}$ (j) 10.2 mW, 5 $\mu\text{m/s}$ and (k) 30.2 mW, 5 $\mu\text{m/s}$ (l) Detailed observation of the region marked in Fig. 3(k).

period was about 124 nm, which was much smaller than the period induced in air. The ripples also adequately spread over both the side wall and the bottom of the valley formed in between grooves. The formation of periodic ripples by femtosecond laser under water environment has drawn much attention since 2004. Much finer ripples could be induced in water with a relative significant number of pulses. The small ripple period might be related to the self-organized process, the excitation of surface plasmon polaritons, and the interplay of the liquids-related perturbations and surface melting [33–36]. To fabricate 2D periodic structures in our experiment, we needed to use a laser power of more than 20 mW to maintain the continuous grooves. In air, the ripples could be induced at a velocity between 150 $\mu\text{m/s}$ and 500 $\mu\text{m/s}$. Moreover, the ripples could be easily induced in water when the scanning velocity was less than 50 $\mu\text{m/s}$. The low scanning velocity indicated a relative large pulse number, which was necessary in forming finer ripples in water. This might be correlated to the energy scattering effect caused by cavitation bubbles and suspended particles formed in front of the irradiated area [33,35].

3.3. Grid-like structures and the optical absorbance of the structured samples

To fabricate grid-like structures with ripples, we scanned the laser beam twice successively along the x and y directions at a laser power of 30.0 mW and a scanning velocity of 150 $\mu\text{m/s}$ in air, using the 10 $\times$ objective lens. By using scanning method, it is possible to obtain textured surfaces on regions with dimensions

up to a few square millimeters or even square centimeters when necessary [15,18]. In our experiment, samples of 1 cm $\times$ 1 cm dimension were structured, as shown in the inset of Fig. 4(a). The structured samples were ultrasonically cleaned by deionized water for 20 min to remove debris. The resulting surface morphology is shown in Fig. 4(a) and (b). Fig. 4(a) shows the uniform grid-like structures over a large area and Fig. 4(b) is the enlarged view of the surface shown in Fig. 4(a). Laser-induced ripples were formed on top of the surfaces as well as in the valley formed in between grooves. There were some nanoscale particles in the bottom of the valleys [37]. The atomic percentage of oxygen at the marked point in Fig. 4(b) was measured as 12.57% by EDX analyzing. Femtosecond laser ablation can induce structural defects and oxides to the silicon surface when scanning in air. Further, the laser-induced defects can worsen the electrical performance of silicon solar cells, and the oxides may be detrimental to subsequent fabrication processes [38]. To ameliorate these concerns, we ultrasonically etched the sample with HF acid, which selectively reacts with SiO_x without etching the crystal silicon. Fig. 4(c) shows the morphology of the surface structures after etching by HF acid. The nanoscale particles in the bottom of the valleys were removed. And the EDX analyzing showed that the left structures were almost pure silicon. After etching, a pristine all-silicon surface was obtained.

Fig. 4(d) shows the absorbance of the sample with grid-like structures shown in Fig. 4(b) and (c). The unstructured silicon was also measured as a reference, as shown in Fig. 4(d). The optical absorbance of the grid-like structures was measured in the wavelength from 250 nm to 1800 nm. Results indicated a prominent

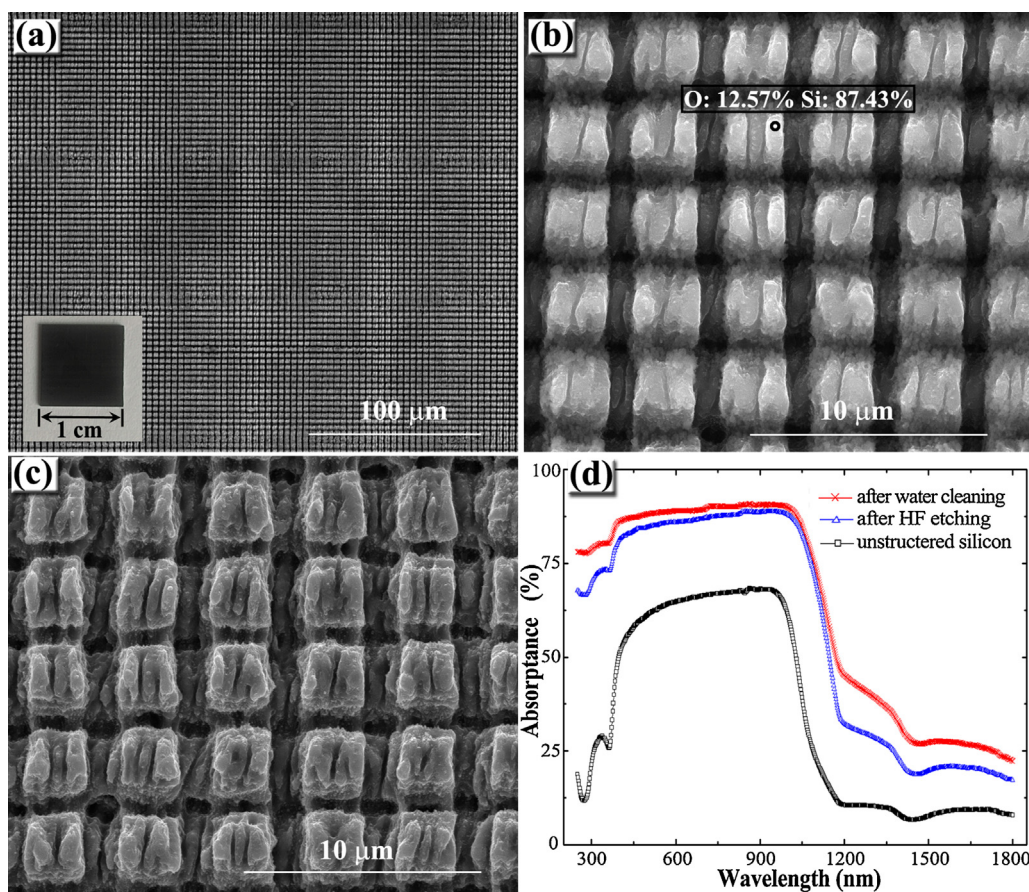


Fig. 4. (a) Overview of the grid-like structures. The inset shows the dimension of the structured sample. (b) Enlarged view of the surface shown in Fig. 4(a). The EDX analyzing point was marked in (b) and the atomic percentages of the elements were given. (c) Surface morphologies after HF etching for 60 min. (d) Absorbance of the samples as a function of wavelength.

enhancement in the shorter wavelength region (below 400 nm). Furthermore the absorbance of visible wavelengths ranged from 86% to 91%. The unstructured silicon was almost transparent above the band gap wavelength (1100 nm), while the structured silicon demonstrated an obvious improvement in absorption from 1200 nm to 1800 nm. After acid etching, the absorbance decreased to varying degrees. Two factors may contribute to the absorption enhancement of the structured surfaces: multiple reflections caused by the grid-like structures, which were primarily responsible for absorption in the visible wavelength range, and the surface defects and impurities. Researchers have found that the incorporation of chalcogens such as sulfur and tellurium can significantly enhance the optical absorption at infrared wavelengths [39]. After acid etching, the Si-O bond and some of the laser-induced defects were removed, which may have led to the decrease of optical absorbance at infrared wavelengths. Moreover, nanoscale particles in the bottom of the valleys were also removed, which may have also contributed to the decrease in absorbance at wavelengths below 350 nm.

4. Conclusion

In summary, 2D periodic structures on silicon were fabricated by femtosecond laser irradiation. Femtosecond laser beams were spatially split by an MLA and focused by an objective lens to fabricate parallel grooves with laser-induced ripples on the surface. The influence of laser power and scanning velocity on the morphological evolution of the 2D periodic structures was investigated. Grid-like structures with ripples were fabricated and their

optical absorbance properties were measured over a range of wavelengths. The method and structures in our study have potential applications in fabricating functional surfaces with different optical properties or wettability properties.

Acknowledgments

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