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Femtosecond laser-induced mesoporous structures on silicon surface

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ABSTRACT

By femtosecond laser line-by-line scanning irradiating, large-scale microstructures were formed on the surface of silicon with dimensions of 1×1 mm^2. Scanning electron microscope investigations exhibited that homogeneous surface microstructures, such as directional-arranged bacilliform mesoporous structures, have been successfully prepared. The dependence of the surface morphology on laser pulse energy was analyzed, and the results indicated that the bacilliform mesoporous structures only can be textured within a certain energy range. The optical reflective spectrum measurement revealed that the presence of bacilliform mesoporous structures can significantly enhance the absorptivity of silicon at visible light range. This work would help to control the formation of surface micro/nanostructures on silicon and other materials, which has potential applications in solar energy, photoelectronics, biology and material science.

1. Introduction

Surface microstructures on silicon have attracted extensive attention in the past few decades because well-defined micro-patterned silicon has potential applications in photoelectronic devices, sensor technologies, solar cells, and so on [1–5]. Consequently, much research activity has been concentrated on controllable texturing of silicon via a versatile and efficient process. Reviewing previous works, multiple surface structures have been prepared on the silicon by various methods. For example, chemical treatments could be utilized to fabricate porous silicon which could be used as the material for the fabrication of micro-hotplates in low-power thermal sensors and thermal-effect micro-systems [6–8]. Other intriguing microstructures, such as silicon spikes, have tremendous potential applications as light absorbers for solar cells [9] and as micro-needles transdermal drug delivery [10]. The formation of the silicon spikes could be accomplished by means of laser-induced chemical etching [11–13] and plasma etching [14–16]. Furthermore, it has been reported that ripples in sub-micrometer scales would develop on the silicon surface after being irradiated by nanosecond laser pulses [17].

Recently, femtosecond laser pulses became a popular tool for texturing of solid materials, such as metals, dielectrics and semiconductors [18–20]. Femtosecond laser texturing of silicon is of interest due to its single-step process and controllable morphologies. A typical femtosecond laser-induced surface structure was subwavelength ripples. When irradiated by femtosecond laser pulses, ripples with interspacing of hundreds of nanometers grow on the silicon surface. The ripples were self-organized and the formation mechanisms might be related to the interference of incident laser light and the excited surface electromagnetic wave [21–24]. In addition, it has been proved that silicon spikes also can be obtained by femtosecond laser irradiation, which is attributed to capillary forces [25,26]. However, the isolated and individual functionalized spots with a dimension of several micrometers usually are useless for practical applications. The large-scale homogeneous surface microstructures, generally speaking, were prepared by means of chemical treatments; but chemical treatments will introduce chemical pollutions, which is unacceptable forbiological and other special applications.

In this paper, we present a fabrication of large-scale homogenous mesoporous structures on the surface of silicon wafers by a femtosecond laser scanning irradiation. By altering the processing parameters, such as incident laser energy, scanning speed, laser polarization and focal objectives, various surface structures, including the directional-arranged bacilliform mesoporous structures and island protrusions, were produced in regions with dimensions of 1×1 mm^2. We analyze the dependence of surface morphology on laser pulse energy, and believe that the bacilliform mesoporous structures only can be textured within a certain energy range. The optical reflective performances of textured areas also have been measured, which revealed the presence of bacilliform mesoporous structures could significantly enhance the visible light absorptivity of sample.

2. Experimental

The femtosecond laser resource employed in our experiments is a multi-pass Ti:sapphire amplifier, which delivered 800 nm, 30 fs pulses

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at a repetition rate of 1 kHz (FEMTOPOWER compact Pro, FEMTOLASERS). The pulse energy could be continuously varied by a variable neutral density filter and a mechanical shutter could turn the laser on and off. The laser pulses were focused via a 10× microscope objective (Nikon, N. A. = 0.3) or a 20× microscope objective (Nikon, N. A. = 0.45). The samples used in our experiments are 400-μm thick Si (100) wafers. Before femtosecond laser irradiation, the wafers are cleaned by ultrasonic bath for 15 min in acetone and rinsed in deionized water.

The samples are positioned horizontally on a three-dimensional computer-controlled stage. After scanning irradiation according to the path shown in Fig. 1, the samples are treated by ultrasonic bath for 5 min both in acetone and deionized water in order to clean the residual ejections off the surface. Results are investigated by a scanning electron microscope (SEM, JEOL, JSM-6390A).

3. Results

The SEM images of surface microstructures induced by femtosecond laser pulses with different processing parameters are shown in Figs. 2–5.

Fig. 2 shows the micrographs of surface microstructures (1 × 1 mm^2) induced by horizontal polarized laser pulses in air. The incident laser pulse energy was set as 1.1 μJ, the scanning direction was parallel to the polarization orientation, and the interspacing between scanning lines was 4 μm. Respectively, the scanning speed and the employed focal microscope objective were: (a) 100 μm/s, 10× (N. A. = 0.3); (b) 50 μm/s, 10× (N. A. = 0.3); and (c) 100 μm/s, 20× (N. A. = 0.45).

Fig. 3 shows the micrographs of surface microstructures (1 × 1 mm^2) induced by vertical polarized laser pulses in air. The incident laser pulse energy was set as 1.1 μJ, and the laser pulses were focused via a 10× (N. A. = 0.3) microscope objective; the scanning direction was perpendicular to the polarization orientation. Respectively, the scanning speeds and the intervals were: (a) 100 μm/s, 4 μm; (b) 50 μm/s, 4 μm; and (c) 100 μm/s, 8 μm.

Fig. 4 shows the micrographs of surface microstructures (1 × 1 mm^2) induced by 45° polarized laser pulses in air. The incident laser pulse energy was set as 1.1 μJ, and the laser pulses were focused via a 10× (N. A. = 0.3) microscope objective; the polarization orientation was 45° to the scanning direction. Respectively, the scanning speeds and the intervals were: (a) 100 μm/s, 4 μm; (b) 50 μm/s, 4 μm; and (c) 100 μm/s, 8 μm.

Fig. 5 shows the micrographs of surface microstructures (200 × 100 μm^2) induced by horizontal polarized laser pulses in air with much higher incident laser energy densities. The laser pulses were focused via a 10× (N. A. = 0.3) microscope objective, the scanning direction was parallel to the polarization orientation, and the intervals between scanning lines were 2 μm. For Fig. 5(a), the incident laser pulse energy was set as 2.5 μJ, and the scanning speed was 160 μm/s; for Fig. 5(b), the incident laser pulse energy was 3.5 μJ and the scanning speed was 80 μm/s.

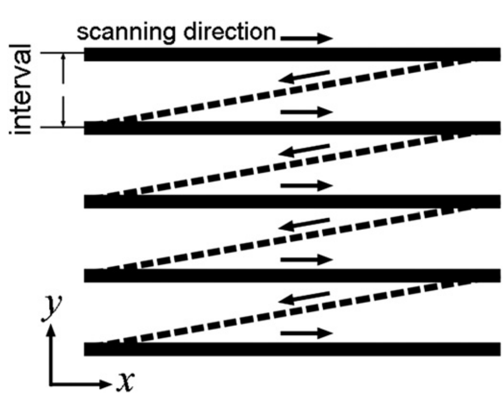

Fig. 1. The illustration of scanning path. The scanning path is zigzag: during the process, the laser pulses were irradiated horizontally along *x* direction; after reaching the end point of one scanning line, a mechanical shutter will occlude the laser beam and the stage moves the sample to the start point of next scanning line. The solid lines denote irradiated lines and the dash lines represent the un-irradiated lines.

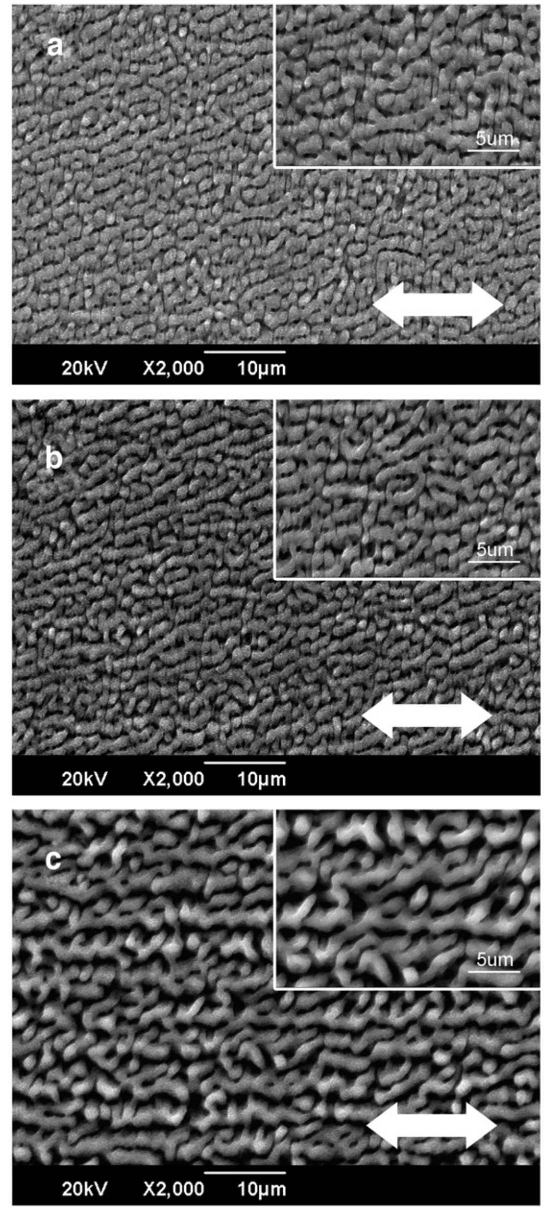

Fig. 2. The SEM images of surface microstructures (1 × 1 mm^2) induced by horizontal polarized laser pulses in air. The incident laser pulse energy was set as 1.1 μJ, the scanning direction was parallel to the polarization orientation, and the interval was 4 μm. Respectively, the scanning speed and the employed focal microscope objective were: (a) 100 μm/s, 10× (N. A. = 0.3); (b) 50 μm/s, 10× (N. A. = 0.3); and (c) 100 μm/s, 20× (N. A. = 0.45). The insert in each figure was the higher magnification image and the white arrow represented laser polarization.

Fig. 6 shows the reflective spectrums of untreated area compared to the micro-structured areas shown in Fig. 2(a), Fig. 3(a) and Fig. 4 (a). The light resource is a board-band light source (UV/VIS light source, DT 1000CE, Analytical Instrument Systems Inc.), and the incident light energies and angles are kept as under the same measurement conditions.

4. Discussion

Comparing with the femtosecond laser-induced ripples or spikes reported in the previous works, the microstructures induced in our