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Non-ablative texturing of silicon surface with a continuous wave fiber laser

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Abstract: Laser surface texturing based on ablation has been widely used, but hardly any reports can be found on non-ablative laser surface texturing. Silicon is highly transparent to the infrared wavelength of fiber laser (λ = 1090 nm) and thus regarded as an unsuitable tool for the purpose of surface texturing. However, we succeeded in using a continuous wave fiber laser to produce regular arrays of sub-micron bumps on silicon surface. The approach is shown to be based on laser-induced oxidation of silicon.

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References and links

1. Introduction

Surface texturing has emerged as an important area in scientific research with a broad range of applications. The laser ablation-based surface texturing has been well studied. Micron and submicron texturing of silicon by laser ablation [1–5] has drawn strong interest due to its wide use in microelectronics and solar cells. Several groups have also reported use of pulsed lasers to produce grooves [6] or pits [7] on surfaces of many other materials such as Ge [8], metals [9,10] and thin films [11,12] under diverse experimental conditions. Near-field enhanced laser irradiation with micro-nano sized spheres [13,14] and near field scanning optical microscope (NSOM) [15,16] are used to form micron, submicron, and nano patterns, but the approaches are also based on the target ablation and redistribution of melt materials.

Hardly any reports can be found in the public domain on use of non-ablative laser surface texturing to form uniform micro-bumps. Silicon is known to be highly transparent to the infrared wavelength of fiber laser ($\lambda = 1090 \text{ nm}$), so fiber laser is not regarded as a suitable tool for ablative texturing of silicon. However, we succeeded in producing arrays of sub-micron bumps on silicon surface through non-ablative texturing with a CW fiber laser. The non-ablative texturing method provides potential possibilities for micro/nano-device applications and clean marking of silicon wafers.

2. Material and experimental procedures

The material used in the study was a single crystal n-type (phosphorous doped) silicon wafer with (100) crystal orientation, resistivity $\rho = 1-20 \cdot \Omega\text{cm}$ and thickness of 300 $\mu$m. The wafer was cut into smaller pieces in the size of 10 mm by 10 mm.

Ultrasonic cleaning of the samples was carried out in methanol for 10 min, acetone for another 10 min, and then de-ionized water for 30 min. To minimize influence of native oxide layer, the cleaned samples were kept in de-ionized water till being used.

The samples were mounted on a motorized X-Y precision stage and irradiated using a CW fibre laser with wavelength $\lambda = 1090 \text{ nm}$. The laser scanning was carried out in an area of 2 mm by 2 mm with a point scanning pitch of 20 $\mu$m, as shown in Fig. 1(a). A lens of 50 mm focal length was used to focus the laser beam to the size of $\approx 40 \mu$m.

![Fig. 1. (a) Dimension and point scanning pitch of laser irradiated area. (b) Fiber laser irradiation and coaxial nozzle to deliver gas onto the laser spot.](image_url)

The laser experiments were carried out at a fixed dwell time of 40 ms but using varying laser power (11.5, 19, 23.3 or 29.3 W) in either ambient, oxygen or argon gas atmosphere. Oxygen and argon were delivered onto the Si surface in three different pressures (0.25, 0.5, or 0.75 bar) through a coaxial nozzle with an exit diameter of 2 mm and placed about 2 mm above the surface, as shown in Fig. 1(b).
The laser irradiated surfaces were characterized using a confocal microscope. Energy dispersive X-ray spectroscopy (EDX) was used to examine chemical composition of the surface features.

3. Results and discussion
Surfaces irradiated with the fiber laser in various conditions were carefully characterized. At the lowest power of 11.5 W, no morphologically modified surfaces were observed. When the power was increased to 19 W, the laser was able to change morphology of the surfaces. The typical results are shown in Fig. 2 and Fig. 3.

After the irradiation in the ambient environment, the surfaces were found to be roughened without regular patterns, as shown in Fig. 2(a). It is very interesting to observe that the irradiated surfaces rose a few microns above the original surface, as shown in Fig. 3(a). This indicates swelling of the material during the laser irradiation. We attribute the swelling to laser-induced thermal oxidation of Si. It is well known that 56% of the oxide thickness lies above the original surface if a bare silicon surface is oxidized. Indeed, EDX analysis of the laser irradiated surface showed the presence of silicon oxide.

If thermal oxidation is really the cause of the swelling in the ambient environment, then no such swelling should be expected to occur during the laser irradiation in the inert environment of Ar. This was indeed found to be the case. The laser irradiation of Si in Ar resulted in roughened irregular surfaces (Fig. 2(b)) but the average surface height is at the original surface level, as showed in Fig. 3(a). EDX analysis of the surface irradiated in Ar did not detect any oxygen, as shown in Fig. 4(a).

Fig. 2. Confocal microscope images of the Si surface irradiated using laser power 19 W and dwell time 40 ms (a) in ambient, (b) in Ar (0.5 bar), (c) in O₂ (0.25 bar), (d) in O₂ (0.5 bar) and (e) in O₂ (0.75 bar).
An idea came to our mind to make use of the laser-induced thermal oxidation to produce regularly-patterned surface bumps. To this aim, Si surfaces were irradiated in pure O$_2$ to enhance oxidation.

Various laser powers (11.5, 19, 23.3 or 29.3 W) were tried. At the lowest power of 11.5 W, the laser was found to be too weak to modify morphology of the Si surfaces. When the laser power was increased to 19 W, uniformly patterned submicron bumps were formed under either of the three different O$_2$ pressures (0.25, 0.5, or 0.75 bar), as shown in Figs. 2(c), 2(d), 2(e), 3(b), 3(c) and 3(d). When the laser power was further increased to 23.3 W or 29.3 W, the regular bumps disappeared and the surface patterns became random.

EDX was used to examine chemical composition of the laser-induced submicron bumps. Silicon and oxygen were detected, as shown in Fig. 4, indicating that the bumps are made of silicon dioxide.

The oxidation of Si can be attributed to heating under irradiation of the laser. It can be deduced that only localized heating can lead to formation of the regular bumps. This explains why uniform patterned surface was achieved only within a narrow processing window (power of 19 W). When laser power was too high, extensive surface melting occurred, resulting in random surface patterns.
Figure 5 shows schematically the conventional growth of SiO$_2$ using uniform heating source. The uniform heating oxidizes the Si surface uniformly and thus produces uniform swelling of the surface due to formation of SiO$_2$, as shown in Fig. 5(b). In this case, no discrete bumps are formed on the surface.

However, when the laser beam moves from one point to another (as illustrated in Fig. 1), it acts as a localized point heating source, therefore formation of discrete bumps is expected to occur at the laser dwelling locations if the laser power is sufficiently high to activate rapid oxidation and in the meantime low enough to avoid extensive surface melting, as shown schematically in Fig. 6(a). It can be seen from Fig. 3 that pitch of the bumps is 20 µm and it indeed matches the laser scanning pitch, lending further support to the above analysis.

Introducing O$_2$ flux into the locally heated region could provide more diffusive and oxidizing atmosphere and therefore help to enhance the laser-induced thermal oxidation process, making it easier to form surface bumps.

Figure 6(b) illustrates the microscopic mechanism of the laser-induced thermal oxidation (photothermal oxidation). For photon energies (i.e., $E_{\text{photon}} = h\nu$) near Si bandgap (1.12 eV), the enhancement in oxidation rate is mainly due to the laser surface heating and the increased electron population in the Si-SiO$_2$ interface. Considering that Si bandgap decreases with increasing temperature [17], laser radiation with the 1090 nm wavelength ($E_{\text{photon}} = 1.138$ eV) causes the electron emission from Si into the Si-SiO$_2$ interface and O$_2$ diffusion. The electrons are also transferred from Si to adsorbed O$_2$ and enhance the diffusion of O$_2$ through the oxide layer, as shown in Fig. 6(b). Therefore, electron tunneling and O$_2$ diffusion may explain the initial rapid oxidation.

Fig. 5. Schematic diagram of SiO$_2$ growth on Si surface by uniform heating source, showing the changes in thickness (a) before oxidation, and (b) after thermal oxidation.

Fig. 6. (a) Laser-induced SiO$_2$ bumps by localized Gaussian heating source; (b) microscopic model for the laser-induced surface oxidation, ↑ and ↓ indicate increasing and decreasing concentrations, respectively.
A further enhancement of the oxidation rate by the laser radiation is due to the heating of both the oxide and Si which favors the O$_2$ diffusion and the hot electrons emission. On the way towards the Si-SiO$_2$ interface, O$_2$ will pick up electrons which are trapped in the oxide. As a result, the concentration of O$_2$ decreases, while that of O$_2^-$ increases. Diffusion of O$_2^-$ is also enhanced by the electric field related to the positively charged holes at the Si surface. In the layer near to Si-SiO$_2$ interface, diffusion of O$_2^-$ and O$_2$ becomes blocked due to lattice mismatch. However, hot electrons emitted into the SiO$_2$ can easily penetrate the blocking layer and enhance the oxidation rate.

Careful observation of the surface profile shown in Fig. 3 reveals that some regions around the bumps go slightly below the original surface of the silicon wafer. This suggests that the possibility of some surface melting at a power of 19 W could not be excluded. The pattern generation process at a power of 19 W might be a combination of both surface melting and oxidation but is dominated by oxidation. However, other possibilities may exist, for example, diffusion of Si atoms to the bump from the surrounding area could occur during localized laser heating and the diffusion could result in the slightly lower surface around the bumps.

The O$_2$ pressures were found to affect the bump height. At the pressure of 0.25 bar, the height was found to be 850 nm. When the pressure was increased to 0.5 bar, the bump height decreased to 720 nm. Increase of the pressure further to 0.75 bar resulted in bumps with even lower height (660 nm). The purpose of varying the pressure was to change the volume flow rate (flux) of O$_2$ gas delivered to the surface. Though introducing oxygen flux provides more oxidizing atmosphere, an increase in the volume flow rate (as the result of increasing pressure) enhances the convective dissipation of thermal energy and thus slows the oxidation rate.

4. Conclusions

A regular array of micro-bumps was fabricated on Si substrate based on laser induced non-ablative texturing. The bumps were found to be SiO$_2$ based on EDX analyses. Formation of the bumps can be explained qualitatively by laser-induced localized Si oxidation. Potential industrial applications of this process are: (i) flexible, selective and fast local rapid thermal oxidation; (ii) debris-free wafer marking and SiO$_2$ structures for optical uses, and (iii) surface textures for new surface functionality.