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表面改性硅的超快激光脉冲制备及光电特性研究

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摘　要：为将单晶硅的吸收限拓展至近红外波段以满足光通讯之需求，利用纳秒激光脉冲辐照单晶硅表面制得了表面结构化的硅 . 研究了能量密度从 0.39 J/cm^{2}到 24 J/cm^{2}的激光脉冲辐照后结构化硅表面的形貌差异 . 测试并分析了不同能量密度的激光脉冲辐照后结构化硅的反射率、透过率和吸收率等光学性质 . 研究发现，相比于单晶硅衬底，所有结构化硅样品都表现出近红外吸收增强特性，对 1 500 nm 的近红外光的吸收率高达 55%. 进而对改性硅样品的红外吸收的热稳定性进行了研究 . 在 473 ～1 073 K 的温度范围内对改性硅样品进行退火，通过分析改性硅的反射率、透过率和吸收率随退火温度的变化规律，发现热退火处理会轻微降低改性硅样品的红外区吸收率，吸收率降低幅度低于 10%. 最后，通过分析脉冲激光改性硅的拉曼光谱，获得了改性硅的晶体结构信息 . 经过纳秒激光脉冲辐照后，硅表面处于无序化状态，形成非晶或多晶相，但是后热退火工艺可以有效改善结构化硅表面的晶体质量 .

关键词：结构化；红外吸收；热退火；硅；脉冲激光
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Research on Fabrication and Optoelectronic Properties of Surface Modified Silicon by Ultrafast Laser Pulse

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Abstract：In order to extend the absorption edge of crystalline silicon to the near infrared band to meet the requirement of optical communication，the textured silicon was obtained by irradiation of nanosecond laser pulses. The surface morphology of modified silicon under irradiation with different laser fluence from 0.39 J/cm^2 to 24 J/cm^2 was investigated. Then，the optical properties including reflectance，transmittance，and absorptance of silicon samples by nanosecond laser irradiation with different laser fluence were measured. The results show that the absorptance below the bandgap of silicon enhances for all the textured silicon samples and it is up to 55% for near infrared light of 1 500 nm. Next，the thermal stability of infrared absorption for the textured silicon samples was investigated. The thermal annealing process at temperatures of 473～1 073 K can slightly reduce the absorptance below the bandgap of silicon and the magnitude of reduction only varied within 10%. Moreover，the dependence of reflectance，transmittance，and

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absorptance of modified silicon on the annealing temperature was studied on. Lastly, the crystal structures of modified silicon were determined by Raman spectroscope. After irradiation of nanosecond laser pulses, the crystalline silicon surface is disordered and amorphous or polycrystalline phases are formed. However, the crystal quality of textured silicon can be improved by post-thermal annealing process.

Key words: Texturing; Infrared absorption; Thermal annealing; Silicon; Pulsed laser

OCIS Codes: 140.3390; 140.3330; 140.3538; 160.1890; 320.7090; 350.3850; 320.4240

0 Introduction

After interaction with ultrafast laser pulses, the dielectric and metal materials will be modified due to the ultra-intense energy of ultrafast laser pulses, such as femtosecond (fs) laser, picosecond laser, and nanosecond (ns) laser[1-5]. Similarly, ultrafast laser pulses will also change the electrical, optical, and structural properties of semiconductor materials and broad the application of semiconductor materials in the field of optoelectronics[6-9]. It is different from fs laser pulses, the ns laser pulses are often applied in the laser annealing process. Owing to the remarkable thermal effect, ns laser pulses can repair lattice damaged by ion implantation and electrically activate doped atoms[10-13]. However, the fluence of the ns laser pulse for laser annealing process is lower than the ablation threshold of semiconductor materials. If the fluence of ns laser pulse is greater than the ablation threshold of semiconductor materials, surface of the materials will be modified. Therefore, many novel properties and functions will be obtained for the modified semiconductor materials[14-17].

Silicon (Si), as one of the most important semiconductor materials, has been widely applied in the field of microelectronics[18-20]. However, crystalline Si can not absorb infrared (IR) light owing to its bandgap of 1.12 eV, so this will limit the application of crystal Si as IR photodetector in optoelectronic integration. It is exciting that the absorption edge of crystalline Si can be extend to near infrared (NIR) wavelength of 2.5 μm after irradiation by fs laser pulses. The microstructured Si fabricated by fs laser pulses has been widely studied[21-25]. However, after ablation using fs laser pulses, the crystal structures and phases in Si surface layer are very complicated because of an extremely fast cooling speed during the resolidification process. The disordered layer of Si surface contains crystalline core, polycrystalline, or microcrystalline, or nanocrystalline[26]. As comparison, the surface of the ns-formed structures is not as disordered as that of the fs-formed structures, the ns-formed structures also have crystalline core, but the disordered layer is even thinner (200 nm or less) and covers only parts of the structures. Consequently, the modified Si by ns laser pulses is better candidate material for the application to photovoltaic devices. This work focuses on the features of damaged layer and particularly on the optical absorption properties in IR region (>1.1 μm). However, the IR absorption mechanism is very complicated and it may include many contributions such as sub-bandgap absorption and free carrier absorption induced by hyperdoping of the impurity atoms, structural defects absorption and dangling bond defects absorption in the amorphous Si (α-Si) phase induced by ns laser ablation process. In this paper, a modified Si surface has been obtained using ns laser pulses ablation in a high pure Argon (Ar) atmosphere instead of gaseous medium with functions of doping or chemical etching. Hence, the absorption contribution from hyperdoping of impurity atoms can be excluded and the defect-absorption contribution induced by ns laser ablation has been emphasized and discussed in detail. Moreover, under these non-doping conditions the IR absorption of the modified Si was observably enhanced even using a very low ns laser fluence of 0.39 J/cm^2. The modified Si shows thermostable NIR absorption, which indicates that this material has potential application in the field of infrared photodetection.

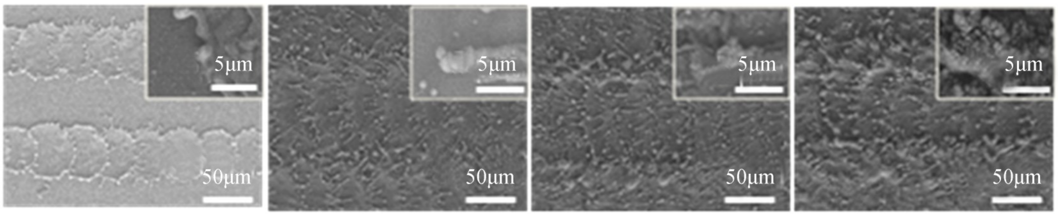
1 Experimental details

N-Si (111) substrates with 250 μm thick and 3 000 Ω·cm resistivity were cleaned using a standard cleaning solution [27]. Next, the cleaned Si substrates were loaded into a vacuum chamber. The vacuum chamber was pumped to 5 Pa (background pressure) firstly and then was filled with high pure Ar (99.999%) atmosphere of 0.1 MPa. Afterward, the vacuum cavity was fixed onto a three-dimensional (3D) translation stage controlled by a computer program. For experiments of laser irradiation, the Si substrates were irradiated by a frequency-tripled, Q-Switched, Nd:YAG ns laser (spectral physics, a 10-ns pulse duration, a 355-nm wavelength, and a 10-Hz repetition rate)[25]. Laser spot was shaped to 8-mm diameter by an expansion system and a diaphragm.

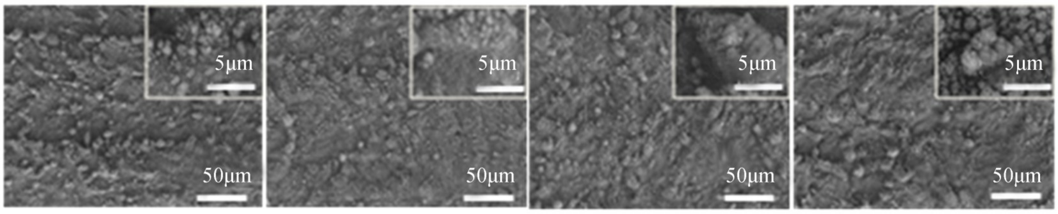
After shaping the laser spot was focused to 180-μm diameters by a 600-mm lens. To obtain modified Si surface with large area, the vacuum chamber was moved in the S-line sweep route (Fig.1(i)) at a rate of 250 μm/s, and the spacing between two adjusted lines is 100 μm, so that each spot got an average number of 7 pulses. The surface morphology was characterized by a field emission scanning electron microscope (SEM, JSM-7 500F, JEOL, and Japan). The optical properties of all Si samples were obtained using a Shimadzu UV-3 600 spectrophotometer equipped with an integrating sphere (LISR-UV3100). The reflectance (R) and transmittance (T) of samples were measured in the range of 250～2 500 nm in 2 nm increments, as a result the absorptance of samples ($A=1-R-T$) can be calculated too. Raman spectroscopy was produced by a LabRAM HR evolution Raman spectrometer by HORIBA scientific with a 532 nm laser. In the experiments, the measured results for reflectance, transmittance, and Raman spectroscopy can be repeated very well and meet accuracy requirements.

2 Results and discussion

After irradiation of ns laser pulses with certain laser fluence, the Si surface is ablated and textured. Fig.1(a)～(h) show the surface morphologies of modified Si samples fabricated at different laser fluences of 0.39 J/cm^2, 2.0 J/cm^2, 3.9 J/cm^2, 7.9 J/cm^2, 12 J/cm^2, 16 J/cm^2, 20 J/cm^2, and 24 J/cm^2. For the laser fluence larger than melting threshold, Si substrate will absorb laser energy. After the lattice reaches temperatures exceeding the melting point of crystalline Si, a molten Si layer will develop. Melting followed by resolidification will change the atomic structure of Si materials[28, 29]. The laser fluence larger than 0.39 J/cm^2 can cause boiling at the melting surface. So the resulting superheating of the liquid phase and high nucleation rates of the gas phase eject material from the Si surface in a process known as ablation. From the SEM image of textured Si with laser fluence of 0.39 J/cm^2, an obvious ablation region related to the shape of laser spot can be observed. In addition, radiated splash can be observed clearly at the edge of the laser spot, which is dependent of the heating effect from ns laser pulses.



(a) 0.39 J/cm^2 (b) 2.0 J/cm^2 (c) 3.9 J/cm^2 (d) 7.9 J/cm^2



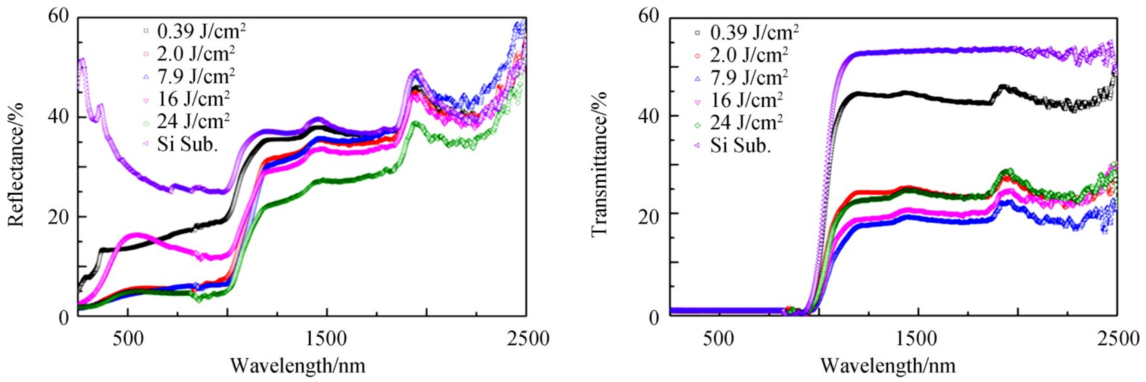
(e) 12 J/cm^2 (f) 16 J/cm^2 (g) 20 J/cm^2 (h) 24 J/cm^2

(i) 15 mm×15 mm samples (j) 1 mm×1 mm samples

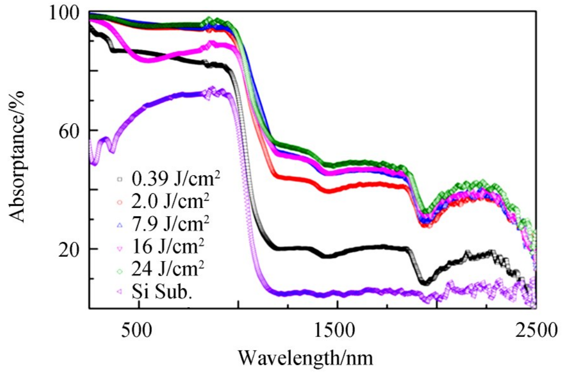
Fig.1 Top-view SEM images of ns laser irradiated samples with the different laser fluence, 15 mm×15 mm samples and the laser fluence for A～H is corresponding to the laser fluence in (a)～(h), the real pictures of 1 mm×1 mm samples

After irradiation of higher laser fluences (>3.9 J/cm^2) more substantial material will be removed from the Si surface and ablation damage becomes more observable. With increase of the laser fluence, the size of ablation zone increases. As a result, the adjacent scanning line overlaps each other. At the same time, the borderline is vague between the two adjacent lines, as a result the ablation zone corresponding to the shape of laser spot can not be observed clearly. The magnified details of the surface microstructures show as insets in Fig.1(a)~(h). Further increase the laser fluence can result in larger structures that go deeper into the material and many micro-spheres particles splashed from the adjacent lines in the Si surface. Furthermore, an increase of the laser fluence can also enlarge the diameters of the sputtered micro-spheres particles and surface roughness of textured Si. In addition, some secondary nanostructures are observed on the micro-spheres surface with the increase of the laser fluence. Fig.1(i) shows the pictures (15 mm × 15 mm) of real Si samples irradiated by ns laser pulses at different laser fluences. The used laser fluence of pictures A~H in Fig.1(i) correspond to that of Fig.1 (a)~(h), respectively. It can be found that the modified Si samples show different surface colours for the different laser fluence. Fig. 1 (j) shows pictures (1 mm×1 mm) of the real Si samples and the adopted laser fluence of pictures A~H in Fig.1(j) correspond to that of Fig.1(a)~(h), respectively. From these pictures, dimension of the whole melting zone increases with increase of laser fluence due to the heating effect of ns laser. A linear absorption process of ns laser pulse will lead deeper melt depths and slower resolidification-front speed, as a result the thermal ablation process of ns laser pulse will cause to the large heat-affected zone in Fig.1(j) [30].

The optical properties of Si samples are modified after the ablation of ns laser pulses. The reflectance (250~2 500 nm) of parts of Si samples is shown in Fig.2(a). From Fig.2(a), for all the modified samples, the reflectance above the bandgap of Si decreases ($\lambda=250\sim1\ 100$ nm) when compared to the reflectance of crystalline Si substrate. The reduction of reflectance in this region can be attributed to scattering of incident light on the rough Si surface [31]. At the same time, for majority samples, the reflectance below the bandgap of Si ($\lambda=1\ 100\sim2\ 500$ nm) is lower than the one of Si substrate. Unlike the reflectance in region of 250~1 100 nm, the reflectance below the bandgap of Si is related to the structure defects induced by the ns laser ablation process [32, 33].



(a) Reflectance of ns laser irradiated samples (b) Transmittance of ns laser irradiated samples



(c) The calculated absorptance of ns laser irradiated samples

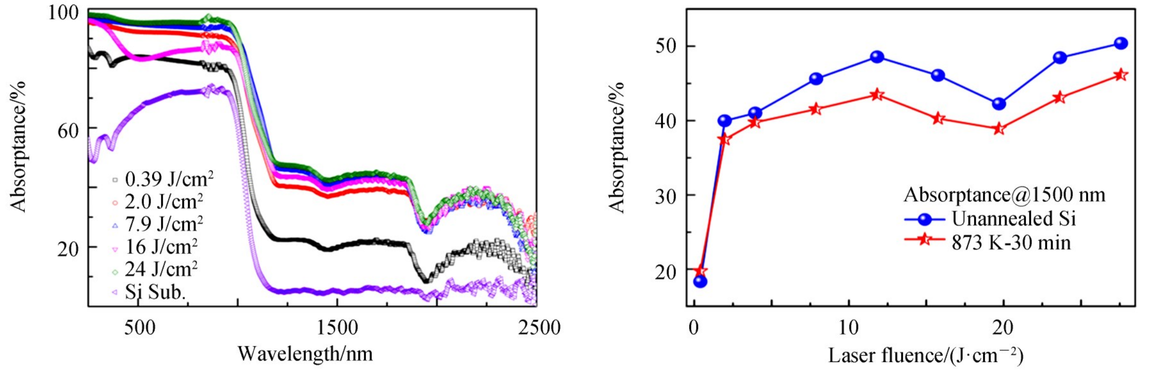
Fig.2 Reflectance, transmittance and absorptance of ns laser irradiated samples with different laser fluence

Similarly, the transmittance of Si samples in the range of 250～2 500 nm is shown in Fig.2(b). From Fig.2 (b) it can be found that the irradiation of ns laser pulses don't change the transmittance above the bandgap of Si (λ=250～1 100 nm). In other words, the light in this region of wavelengths can not pass through the modified Si samples. However, after laser irradiation the transmittance below the bandgap of Si (λ=1 100～2 500 nm) remarkable decreases. In addition, the transmittance of fabricated Si samples decreases firstly (<7.9 J/cm^2) and then increases (>7.9 J/cm^2) with the increase of laser fluence.

Then the absorptance of modified Si samples can be calculated according to energy conservation of light. The absorptance of modified Si samples and Si substrate is shown in Fig. 2(c). In comparison with the absorptance of crystal Si substrate, the absorptance of modified Si samples obviously enhances in whole wavebands. For majority of modified samples the absorptance above the bandgap of Si is higher than 90%. Moreover, the absorptance in the range of 1 100～2 500 nm basically increases with increase of the laser fluence. For all the modified Si samples, the absorptance gradually decreases with increase of the wavelength in the range of 1 100～1 800 nm. The decrease of the absorptance is related to the structural defects induced by ns laser ablation [34].

In sum, for the modified Si samples, the reduction of reflectance and transmittance below the bandgap of Si can be attributed to the increase of light absorption. In addition, the absorption below the bandgap of Si are mainly induced by two mechanisms: Urbach states from α-Si in the re-solidified surface layer [35], deep-level absorption of structural defects induced by the fast melt cooling process[36]. As increase of the laser fluence, the surface roughness and defects density of the ablated Si increase simultaneously due to more serious damage in Si surface layer. As a result the absorption of ablated Si increases, too. According to the relationship ($A+T+R=1$) among the A, R, and T, enhanced absorptance below the bandgap of Si will cause corresponding decrease of reflection or transmission.

To investigate the thermal stability of NIR absorption of ablated Si by ns laser pulses, the modified Si samples have been thermally annealed at temperature of 873 K for 30 min. The absorptance of modified Si samples is shown in Fig.3(a). The absorptance below the bandgap of Si is slightly reduced by the annealing process (magnitude of reduction is lower than 10%) while the absorptance above the bandgap of Si nearly doesn't change after the thermal annealing. In comparison with the absorptance of unannealed Si samples, the absorptance of the annealed samples do not gradually decrease with increase of the wavelength (1 100～1 800 nm), which is related to reduced density of defect states in modified Si layer by the annealing process. However, the dependence of absorptance on laser fluence is nearly the same as the one before annealing. In order to clearly observe the variation tendency of absorptance of modified Si samples vs laser fluence, the dependence of sample's absorptance at 1 500 nm on the laser fluence (0.39～28 J/cm^2) is described in Fig. 3(b). The absorptance (18.3%) is low for the Si sample ablated by laser fluence of 0.39 J/cm^2 while the absorptance (39.9%) rapidly rises for the sample irradiated under laser fluence of 2.0 J/cm^2. Then, the absorptance of samples basically increases with increase of the laser fluence. After annealing, the variation of absorptance vs laser fluence shows similar tendency to that before annealing. But the annealing process reduces the absorptance

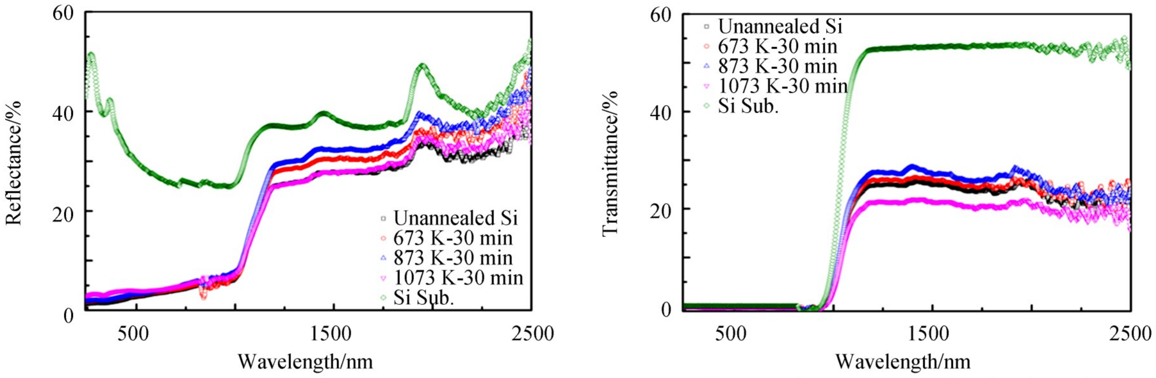


(a) Absorptance of ns laser irradiated samples after thermal annealing

(b) The absorptance of samples before and after annealing at wavelength of 1500 nm

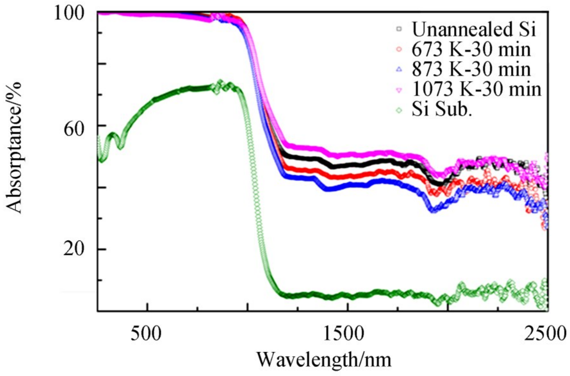
Fig.3 Effect of thermal annealing on absorptance of ns laser irradiated samples of all the modified Si samples. For example, the absorptance decreases from 48.6% to 43.5% for the sample irradiated under laser fluence of 12 J/cm^2.

To determine the dependence of optical properties on annealing temperature, the modified Si samples have been thermally annealed at different temperatures (473～1 073 K) for the same duration time (30 min). Using a high-temperature tubular furnace, the samples were annealed at a flowing Ar atmosphere (99.999%) with pressure of 1 atm. The reflectance and transmittance of annealed Si sample (irradiated by ns laser pulses at fluence of 12 J/cm^2) show as Fig.4(a) and (b), respectively. Annealing at 473 K nearly does not affect optical properties of the samples, thus the optical properties of 473 K are not provided in the Fig.4. After annealing at the temperature of 473～873 K, the reflectance above the bandgap of Si is almost unaltered. As a contrast, the reflectance below the bandgap of Si increases with increase of the annealing temperature. However, after annealing at the temperature of 1 073 K the reflectance of modified Si sample has little variation in the whole measured range. Next, from Fig.4(b), the transmittance above the bandgap of Si does not change after annealing at all the temperatures, whereas the transmittance below the bandgap of Si increases with increase of the annealing temperature (473～873 K). It is interesting that further increasing temperature (1 073 K) will oppositely reduce the transmittance of modified Si samples.



(a) Reflectance of ns laser irradiated sample (12 J·cm^{-2}) after annealing

(b) Transmittance of ns laser irradiated samples after annealing

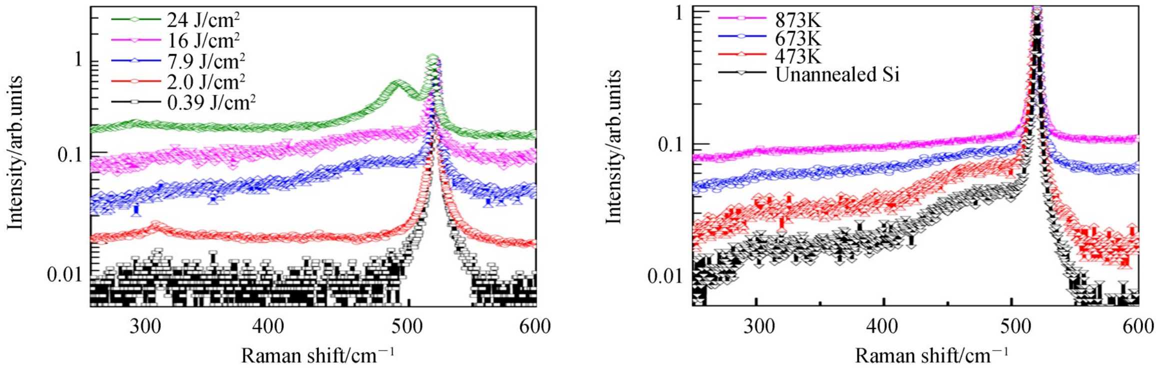


(c) The absorptance of ns laser irradiated samples after annealing

Fig.4 Reflectance, transmittance and absorptance of ns laser irradiated sample(12 J/cm^2) after annealing at different temperatures

The absorptance of annealed samples is calculated in Fig.4(c). Similarly, thermal annealing process nearly doesn't impact the absorptance above the bandgap of Si. But the annealing process can reduce the absorptance below the bandgap of Si for the annealing temperature of 473～873 K. In addition, the NIR absorptance of modified Si sample decreases with increase of the annealing temperature. In contrary, the absorptance of annealed Si samples at the temperature of 1 073 K increases compared to one of unannealed Si sample. After thermal annealing at temperature of 473～873 K, the reduction of absorptance below the bandgap of Si can be attributed to the weakly thermal stability of defect states contributing to the NIR absorption. However, after annealing at higher temperature of 1 073 K, the enhanced absorptance maybe come from the point defects stabilized at high temperature and this phenomenon is called absorption reactivation[37].

To clarify the structures of modified Si by ns laser pulses, the Raman spectroscopy of ablated Si samples is measured. Fig.5(a) is the Raman spectrum of Si surface layer irradiated by ns laser pulses at different fluences. From the Raman spectrum in Fig.5(a), all samples include a Raman vibration peak centred at 520 cm^{-1} corresponding to vibration of Si-Si atoms of the crystalline Si (Si-I)[38, 39]. Another vibration peak centred at 302 cm^{-1} of crystalline Si can also be clearly observed for the samples irradiated under lower laser fluence of 0.39 J/cm^2 and 2.0 J/cm^2. In addition, no other vibration peaks are observed in the Raman spectrum of these two samples. For further increasing laser fluence, two broader vibration peaks centred at 302 cm^{-1} and 476 cm^{-1} can be observed in the Raman spectrum, which comes from ns laser induced α-Si phase. The relative intensity of vibration peak centred at 476 cm^{-1} increases with increase of the laser fluence owing to the laser-induced α-Si phase. Especially, a strong vibration peak centred 490 cm^{-1} appears in the Raman spectrum for the sample irradiated at higher laser fluence of 24 J/cm^2 and this peak may related to the polycrystalline phase induced by stronger ablation of ns laser pulses. Fig.5(b) is the Raman spectrum of annealed Si samples (laser fluence is 12 J/cm^2) at different annealing temperatures. From the Fig.5(b), lower annealing temperature such as 473 K has no effect on Raman spectrum of modified Si sample. With increase of the annealing temperature, vibration peak at 476 cm^{-1} disappears, which means annealing process may cause a transformation of the phase from α-Si to crystalline Si. With this process, the crystal quality of modified Si can be improved and the metastable defects existing in these phases will vanish during this thermal process.



(a) Raman spectrum of laser irradiated samples with different laser fluence

(b) Raman spectrum of laser irradiated sample (12 J·cm^{-2}) with different annealing temperatures

Fig.5 Effect of thermal annealing on Raman spectrum of ns laser irradiated samples

3 Conclusion

In this paper, nanosecond laser pulses have been used to modify the crystalline Si surface. From the SEM images, it can be found that the larger laser fluence will induce the larger surface roughness and larger-scale ablation, which is related to heating effect of nanosecond laser pulses. In addition, different surface colours have been observed for the real pictures of textured Si samples fabricated at different laser fluences. The study on optical properties indicates that the infrared absorptance of crystalline Si can be enhanced by nanosecond laser irradiation and the infrared absorptance increases with increase of the laser fluence within the specific limits. Although the thermal annealing process can slightly reduce the infrared absorptance of textured Si, the magnitude of reduction in the absorptance is unobvious. Furthermore, the infrared absorptance decreases with increase of the annealing temperatures until 873 K. Finally, from the measurement for Raman spectroscopy, the crystal structures of modified Si by nanosecond laser pulses have been changed. The Si surface has been disordered by the melting and resolidification process. Next, the amorphous or polycrystalline phases have been formed in the disordered Si surface layer. Consequently, the infrared absorption of samples may be related to these new phases formed in the modified Si layer. Further thermal annealing will improve the crystal quality of modified Si with the reduction of density of defect states in Si surface layer.

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