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Fabrication of Anti-reflecting Si Nano-structures with Low Aspect Ratio by Nano-sphere Lithography Technique

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Abstract: Nano-structured photon management is currently an interesting topic since it can enhance the optical absorption and reduce the surface reflection which will improve the performance of many kinds of optoelectronic devices, such as Si-based solar cells and light emitting diodes. Here, we report the fabrication of periodically nano-patterned Si structures by using polystyrene nano-sphere lithography technique. By changing the diameter of nano-spheres and the dry etching parameters, such as etching time and etching power, the morphologies of formed Si nano-structures can be well controlled as revealed by atomic force microscopy. A good broadband antireflection property has been achieved for the formed periodically nano-patterned Si structures though they have the low aspect ratio (<0.53). The reflection can be significantly reduced compared with that of flat Si substrate in a wavelength range from 400 nm to 1200 nm. The weighted mean reflection under the AM1.5 solar spectrum irradiation can be as low as 3.92% and the corresponding optical absorption is significantly improved, which indicates that the present Si periodic nano-structures can be used in Si-based thin film solar cells.

Keywords: Nano-sphere lithograph; Nano-patterned Si structures; Antireflection

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Introduction

Due to the increased environment problems and the decreased availability of fossil fuel sources, many research efforts has been in developing the clean renewable energy technologies. Among the various energy projects, solar cells which harvest energy directly from sunlight are considered as the most promising candidate for future energy resources [1]. Although a substantial drop of cost and efficiency improvement has been achieved in last decades, significant improvements in both device performance and the manufacturing process are still demanded to keep it economically competitive.

Currently the photovoltaic market is dominated by Si-based materials such as crystalline and polycrystalline Si, of which around half of the cost is from silicon wafers. Therefore, the research work on the development of thin-film solar cells that do not require the use of thick silicon wafers has attracted much attention. Thin film solar cells with a typical thickness of 1∼2 μm can be fabricated at a much reduced cost. For the practical application of Si-based thin film solar cells, the development for high throughput processes is required.

For both performance improvement and cost reduc-

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tion, photon management is critical in thin film Si-based solar cells, which can not only reduce the optical losses, but also shorten carrier collection length to reduce transport losses. So far, an efficient photon management is still rather challenging because it should work in a broad spectral regime due to the broadband of solar spectrum. The pyramid structure with a feature size of tens of microns is usually applied for the light trapping in crystalline silicon solar cells. However, for thin-film solar cells, the minority-carrier diffusion length is around 300 nm while the light absorption depth is around 1 μm. Hence it is essential to realize an efficient light management which can work in a broad spectral range with a feature size in the sub-wavelength regime. Nano-structures with a scale comparable to the wavelength of most utilized solar spectrum are considered as the promising candidates for the advanced photon management.

Advanced light management by using semiconductor nano-structures, such as nano-wires, nano-columnar, nano-cones etc, is currently proposed to enhance the absorption and reduce the surface reflection in a broad spectral range for high performance solar cells [2-6]. For example, by fabricating Si nano-wires, the peak absorption can be as high as 96% due to the light trapping effect [4]; the optical absorption enhancement has also been observed in Si nano-cones arrays prepared by using SiO_2 nano-sphere lithography technique [6]. In many cases, good antireflection characteristics were achieved in Si nano-structures with high aspect ratio, which cannot be used in thin film solar cells due to the thickness limit. Moreover, high aspect ratio may introduce a high level of surface defects to reduce the carrier collection efficiency [7,8]. Recently, the theoretical results suggested that surface feature with low aspect ratio can also enhance the light absorption without or with less reducing the electrical properties [7].

In our previous work, the periodical nano-structures fabricated by polystyrene (PS) nano-sphere lithography technique was introduced to optical devices containing nano-crystalline Si/SiO_2 multilayers and an enhanced photoluminescence (PL) and electroluminescent (EL) was observed [9,10]. The nano-structure was also used in hetero-junction thin film solar cells and the enhanced light absorption and significant improvement of cell performance have been achieved [11,12]. However, the influences of formed nano-patterned structures under the various preparation conditions on the reflection and absorption characteristics are still unknown and need further investigation.

In this paper, we reported the fabrication of periodically nano-patterned Si structures using PS nano-sphere lithography technique. By controlling the diameters of PS nano-spheres and the etching parameters, periodically nano-patterned Si structures can be achieved with various periodicities and depths in a large scale. We systematically studied the morphologies and the reflection characteristics of Si nano-patterned structures in order to optimize the fabrication conditions. We found that the reflection can be obviously suppressed and the reflection can be lower than 5% in a broad wavelength regime (400~1200 nm) even with the low aspect ratio. Correspondingly, the optical absorption is significantly enhanced, which indicates the potential applications of the present nano-patterned Si structures prepared by a cheap and easy approach in the thin film solar cells for improving the device performance.

Experiment

Polystyrene nano-sphere lithography technique was used to fabricate the periodically patterned Si nano-structures on (100) p-type Si wafer ($1.5\sim3\ \Omega\cdot$cm). The Si wafers were cut into 2 cm × 2 cm squares and pre-cleaned with standard RCA process and then rinsed in deionized water for several times to get the clean surface. The fabrication process includes the self-assembly of monolayer PS nano-spheres and the subsequent dry etching [13]. First, the cleaned silicon wafers used for transferring were pre-processed by immersing in the 5 wt% Sodium dodecyl sulfate (SDS) solution for more than 24 hours. The blend solution of PS is prepared with the mixture of PS solution and ethanol of a volume ratio 1:4. A drop of the blend solution was dropped on the transfer wafer to form a thin film of PS nano-spheres. Then the thin film of PS nano-spheres was transferred to the water surface which is processed with a few drops of SDS solution and a monolayer of PS nano-spheres was formed on the water surface, the color of which depends on the diameter of PS nano-spheres. The monolayer of PS nano-spheres was transferred to the substrates, and it sticks onto the Si substrates after the water evaporated. Then, the Si wafers covered by the monolayer of PS nano-spheres were set into the conventional reaction ion etching (RIE) system. By using PS nano-spheres as a mask, the patterned structures can be formed on Si wafer after etching. During the etching process, the radio frequency (r.f) power varies from 20 W to 48 W and the reaction chamber pressure is kept at 3.3 Pa by controlling the pumping speed. The CF_4 with flow meter of 30 Sccm (Standard cubic centimeters per minute) was used as etching gas and etching time varies from 10 min to 15 min. After RIE process, PS nano-spheres were removed in tetrahydrofuran (THF) solution.

We measured the surface morphology by using atomic force microscopy (AFM) with the tapping mode. The etching depths under different r.f power were explored by summarizing the AFM measurement results. The reflection spectra were measured by using

Shimadzu UV-3600 spectrometer without integrating sphere in the wavelength range (400∼1200 nm). In our previous work, we found that the reflection spectra are almost the same whether using the integrating sphere or not, which indicates the angle-independent antireflection characteristic of the nano-structures [11]. The reflection spectra of nano-patterned structures etched under different r.f power were measured to investigate the antireflection characteristics of nano-patterned structures and compare with the results of the flat Si substrate without any treatment.

Results and discussion

The morphologies of the nano-patterned Si structures obtained by using PS nano-spheres lithography technique was characterized by AFM measurements. The diameter of used PS nano-spheres is 220 nm and 300 nm, respectively. The scanning area is about 2 μm × 2 μm. As shown in Fig. 1(a) and (b), the ordered and nano-patterned Si structures can be clearly identified for both samples. It is worth pointing out that we measured the various places of the samples and observed the similar surface morphologies which indicates that the periodically nano-patterned Si structures can be formed in a large area (in our case, at least 1 cm^2). It is also shown in AFM images that the nearly close-packed Si structures are formed and the periodic length of formed Si nano-structures is consistent with the diameter of used PS nano-spheres, which suggests that one can control the periodicity of Si nano-patterned structures by choosing PS nano-spheres with the suitable sizes.

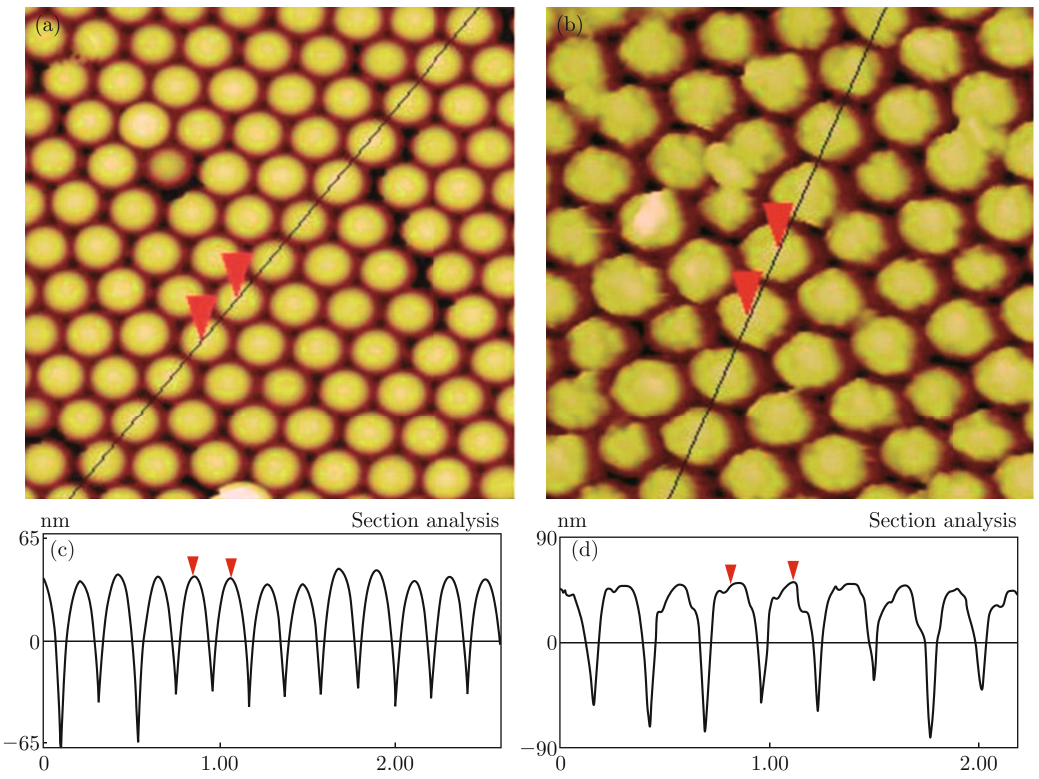


Fig. 1 AFM image of nano-patterned Si structure prepared by using PS nano-spheres with (a) diameter of 220 nm; (b) diameter of 300 nm. (c) and (d) The cross-sectional AFM images for corresponding AFM images indicated by solid line.

The cross sectional AFM images are also given in Fig. 1(c) and (d). The depth from the top of the nano-structures to the bottom can be roughly estimated from the cross-sectional AFM images. It was found that the average depth is respectively about 94.8 nm and 117.5 nm for 220 nm and 300 nm periodically nano-patterned structures. Figure 2 is the change of etching depth estimated from AFM images with the etching time both for 220 nm and 300 nm periodic structures while the r.f etching power is kept at 20 W. It is shown that the etching depth is gradually increased with the etching time and the etching depth is almost same both for 220 nm and 300 nm periodic structures, especially under the short etching time. After 10 min dry etching, the depth is about 40 nm for both samples and it reaches about 100 nm after 15 min etching for 220 nm periodic nano-structures and exceeds 110 nm for 300 nm periodic one.

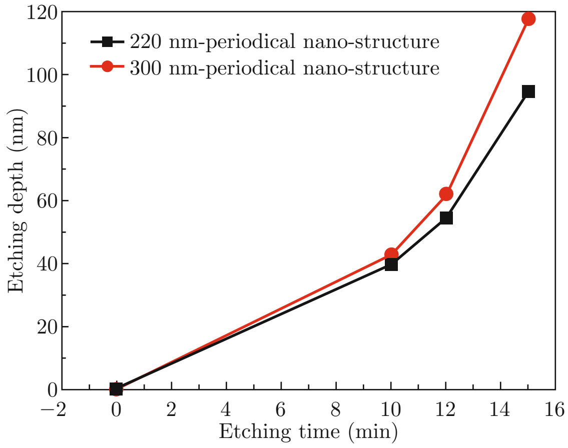


Fig. 2 The etching depth estimated by AFM images as a function of etching time for 20 nm and 300 nm periodically nano-patterned Si structures. The etching time for both samples is 10 min and the r.f etching power is 20 W.

It was found that, besides the etching time, the depth of the prepared nano-structures is also strongly depended by the other etching parameters, especially the etching r.f power. Here, we also estimated the etching depths for 300 nm periodic samples under different r.f power by using AFM measurements. Figure 3 is the change of etching depth as a function of etching r.f power. It was found that the etching depth is increased from 41.3 nm for 20 W etched structures to 157.8 nm for 48 W etched structures. Increasing the r.f power can decompose the reactive gas more efficiently, which results in the higher etching rate to get a large etching depth at the same etching time as revealed in our experimental results. We also measured the etching depth by using cross sectional transmission electron microscopy (TEM) for some selected samples. It was also found that the AFM results is slightly under-estimated compared with that that obtained from TEM images [10]. However, the depths obtained from AFM images do reflect the depth increase with the development of etching time and r.f power. Our experiment results demonstrate that PS nano-sphere lithography technique is an effective way to get uniform and periodically nano-patterned Si structures in a large area and the surface morphology of nano-patterned structures can be well controlled by changing the etching parameters such as r.f power and time.

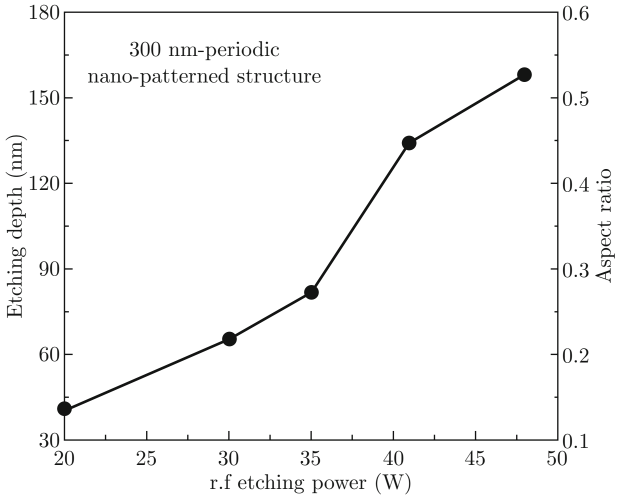


Fig. 3 The etching depths and the corresponding aspect ratio of 300 nm periodically nano-patterned Si structures as function of r.f etching power.

The reflection spectra in the wavelength range from 400 nm to 1200 nm were measured for 220 nm and 300 nm periodically nano-patterned Si structures obtained under the r.f power of 48 W and etching time of 10 min. Figure 4 shows the measurement results. The reflection spectrum of flat Si wafer is also given in the figure for reference. It was found that the reflection for flat Si substrate without patterning is quite high. The reflection is higher than 30% in the whole measurement range and even higher than 40% in the visible light region due to high refractive index of Si material. However, the reflection is obviously reduced for samples with periodically nano-patterned structures. The reflection is less than 20% for 220 nm periodic Si nanostructures and less than 10% for 300 nm periodic one in a whole spectral range. In the visible light region, the reflection of 300 nm periodically nano-patterned structures is even less than 5%. In our previous work, we investigated the reflection spectra for 220 nm periodic Si nano-structures etched under the various etching time [10]. It was found that the reflection can be further reduced by increasing the etching time. The reduced reflection suggests that the present periodically nano-patterned Si structures have the good antireflective characteristics even though they have the low aspect ratio. In the present case, the two kinds of Si nano-structures are obtained under the same etching time (10 min). At the wavelength regime below 800 nm, the two kinds of nano-structures have almost the same antireflection characteristics while at long wavelengths, 300 nm-periodical nano-structures show the better antireflection characteristic. According to the scattering effect theory, when the diameter of nanostructure becomes comparable to the light, the incident light will greatly scattered therefore increase the light path length, which means that 300 nm periodical nano-structure has a broader antireflection wavelength region. As seen in Fig. 1(c) and (d), we can identify that the depth of 300 nm-periodic structures is slightly larger than that of 220 nm-periodic one, which may result in the better antireflection characteristics of 300 nm periodic nano-structures.

In order to further understand the influences of formed nano-structures on the antireflection behaviors, we studied the reflection behaviors of nano-patterned Si structures obtained under the various r.f etching power. Figure 5 shows the reflection spectra of 300 nm periodically nano-patterned Si structures etched under the various r.f powers (20∼48 W). It is clearly shown that the reflection is gradually reduced with increasing the r.f power in a whole spectral range (400∼1200 nm). The maximum reflection for 20 W etched sample is about 25%, while for the reflection 48 W etched one is lower than 5% in the whole measurement spectral range. The similar change is also observed for patterned Si nano-structures with periodicity of 220 nm. It is suggested that the present patterned Si nano-structures exhibit the good antireflective characteristics and the reflection can be modulated by controlling the fabrication parameters.

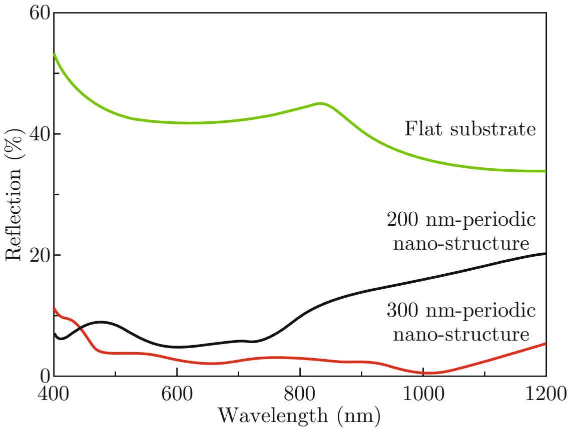


Fig. 4 Reflection spectra of nano-patterned Si structures with periodicities of 220 nm and 300 nm etched at 48 W for 10 min. The reflection spectrum of flat Si substrate is also given as a reference.

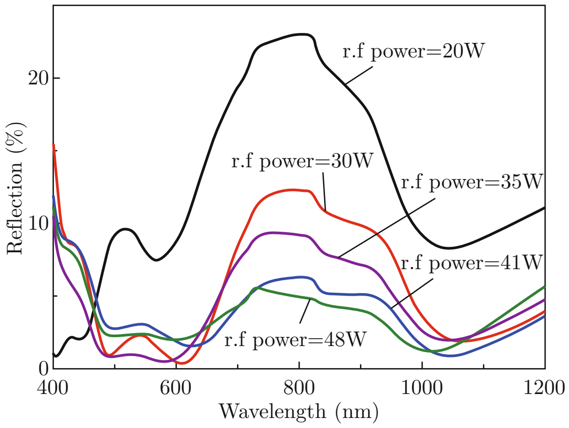


Fig. 5 Reflection spectra of 300 nm periodically nano-patterned Si structures prepared under the various r.f etching power.

It is well known that a significant fraction of sunlight (∼30%) will be reflected on the Si surface without any treatment and surface roughening is an effective approach to suppress the surface reflection and improve the device performance. The roughening structures are usually prepared by lithography technique or wet etching process and the feature size are around tens of microns [11]. For the application in the film solar cells, it is high desired to develop the technology of photon management in the sub-wavelength regime. Our results demonstrate that PS nano-sphere lithography technique is a simple and low-cost way to provide a sub-wavelength periodically patterned structures with good antireflective properties.

The similar antireflective behaviors have also been reported by other groups. X. Li et al. studied the efficient antireflective properties of periodically aligned Si nano-pillar arrays [14]. They fabricated the periodically nano-patterned Si structures by using silver catalyzed chemical etching process and found that the reflection can be remarkably reduced in the wavelength range of 200∼1000 nm. However, the reflection was increased obviously when the wavelength is longer than 1000 nm. M. A. Tsai et al. also reported the reduced reflective behaviors of crystalline Si with frustum nano-rod arrays. They reported that the reflection as low as 10% can be achieved in nano-rod Si arrays, which is lower than that of KOH textured structures in the wavelength range from 400 nm to 1000 nm [15]. In our case, we fabricated the periodically nano-patterned Si structures and the reflection can be lower than 10% in a more wide spectral range (400∼1200 nm) under the suitable preparation conditions.

As mentioned before, the reduction of reflection is associated with the morphologies of formed nano-structures [6,16]. The broad band antireflection characteristics can be understood by the effective medium theory [7,17]. According to the effective medium theory, a single layer with sub-wavelength roughness on the surface can be approximated as a set of multi-layers of the “effective medium” with the intermediate refractive index between the substrate and the air. The effective refractive index is a function of the volume fractions of the individual rough layer [17] and gradual reduction of the effective refraction index can be realized which can reduce the reflection in a wide spectral range and incident angles [5,16]. It was also reported that reduced reflection for the long wavelength photons can be attributed to the porous-like layer of nano-structures, which the refractive index discontinuity between air and Si substrate can be effectively buffered [15,18]. In our work, the formed Si nano-patterned structures have the cone-shape which causes the gradually increase of fractional area occupied by Si from top to bottom. The gradually changing of Si filling factor form a gradual effective refractive index from top to bottom which can suppress the reflection in a wide spectral range. Based on the AFM observations, it is found that, with increasing the etching time as well as the r.f etching power, the etching depth is gradually increased while the lateral sizes of formed nano-structure is almost unchanged due to the protection of covered PS nano-spheres. As a result, the aspect ratio becomes larger and the Si filling factor is changed with the etching time and r.f power and in turn, the improved antireflection characteristics are achieved in a wide spectral range.

Considering the application of nano-patterned Si structures in thin film solar cells, we estimated the weighted mean reflection R_w by calculating the reflection ratio of the periodically nano-patterned Si structures to the incident AM (Air Mass) 1.5 light at the each wavelength and averaging the results in the 400∼1200 nm. The weighted mean reflection is calculated as follow:

$$R_w = \frac{\int_{\lambda_2}^{\lambda_2} F(\lambda)R(\lambda)d\lambda}{\int_{\lambda_2}^{\lambda_2} F(\lambda)d\lambda}$$

where $F(\lambda)$ and $R(\lambda)$ is the flux of incident light and reflection of nano-patterned structures at wavelength of λ, and the λ_1 and λ_2 is 400 nm and 1200 nm, respectively. Figure 6 shows the weighted mean reflection for 300 nm periodic Si nano-structures as a function of etching r.f power. It shows the trend that the weighted mean reflection is decreased with increasing the r.f power. The weighted mean reflection R_w is decreased from 12.7% to 4.24% with increasing the r.f power from 20 W to 48 W while the lowest reflection is 3.92% for 41 W. This trend is almost consistent with the change of etching depth and a best etching depth exists for the lowest weighted reflection. The lowest reflection around 3.92% is better than the reflection obtained from nano-scale textured Si surface prepared by wet chemical etching technique [19]. Our experimental results indicate that the present nano-patterned Si structures can efficiently reduce the optical reflection loss in 400∼1200 nm which covers most of the useful solar spectrum and is a promising candidate for application in the next generation Si-based thin film solar cells.

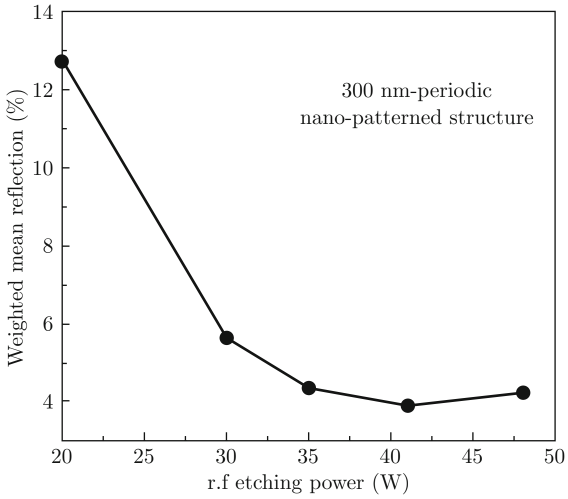
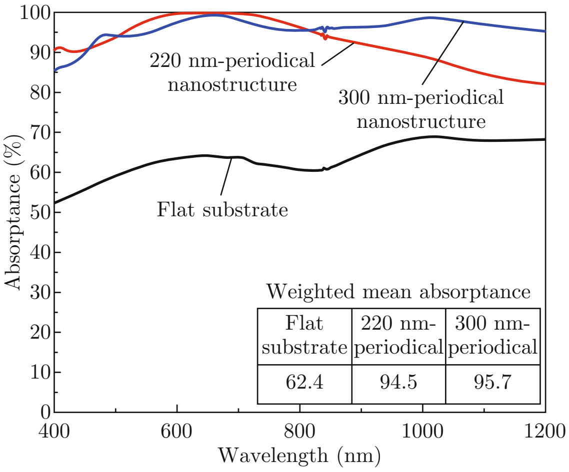


Fig. 6 Weighted mean reflection by AM 1.5 solar spectrum of 300 nm periodically nano-patterned Si structures as a function of r.f etching power.

In order to investigate the optical absorption of nano-patterned Si structures, we measured the reflection (R) and transmission (T) spectra for nano-patterned Si samples. The optical absorptance (A) was calculated by using $A = 1 - R - T$. Figure 7 shows the optical absorption spectra of periodically nano-patterned Si structures with 220 and 300 nm periodicities, respectively. The etching time and r.f power is the same for both two samples. For comparison, the optical absorptance of the flat Si substrate is also given in the same figure. As shown in Fig. 7, the optical absorption of the nano-patterned Si structures is significantly enhanced in a broad wavelength region compared with that of flat Si wafer. It is found that the optical absorptance of nano-patterned Si structures is higher than 80% in the whole measurement wavelength range. For 300 nm periodic nano-patterned Si structures, the optical absorptance is even higher than 90% in a spectral range of 400∼1200 nm. The enhanced optical absorption has been reported in Si nano-dome structures [6]. It was found that the optical absorption is obviously improved even in a large incident angles. The enhanced optical absorption can be attributed to the suppressed surface reflection and multiple absorption of the scattering light in the nano-patterned structures.



Weighted mean absorptance

Flat substrate	220 nm-periodical	300 nm-periodical
62.4	94.5	95.7

Fig. 7 The optical absorption spectra of 220 nm and 300 nm periodically nano-patterned Si structures after cell deposition. The absorption spectrum of flat Si substrate is also given for comparison. Inset is the summarized mean optical absorptance weighted by the AM 1.5 solar spectrum.

We also calculated the mean absorption weighted to AM 1.5 (1 sun) solar spectrum and the results are summarized in Fig. 7. The mean weighted optical absorptance of the flat Si wafer is about 62.4% while it is enhanced to 94.5% and 95.7% to 220 nm periodic and 300 nm periodic nano-patterned Si structures, respectively, which is close to the best results reported previously [6,20]. It is implied that the most of the incident sunlight can be utilized by the Si substrates with nano-patterned structures.

The suppressed surface reflection and enhanced optical absorption can be realized by using the present nano-patterned Si structures formed by the nano-sphere lithography technique because they provide the grading refraction index between the air and Si interface as we discussed before. It was also reported that the optical absorption can be enhanced by using periodically sub-wavelength nano-structures. Since the feature size of surface pattern is smaller than wavelength, the incident electromagnetic wave can be coupled with the whole surface sub-wavelength structures which can trap the light to enhance the light harvesting in a wide spectral range [21,22].

Usually, the good antireflective properties and enhanced optical absorption have been achieved by introducing the Si nano-structures with high aspect-ratio. However, in our case, compared with the previous Si nano-structures, such as Si nano-wires (the length is 67 μm in micrometer scale) [4] and Si nano-cones (the length is around 600 nm) prepared by using SiO_2 nano-sphere lithography technique [6], the depth of nano-patterned structures is quite low as revealed by AFM observations. The small etching depth indicates the low aspect ratio (<1) of our nano-patterned structures. The aspect ratios of the 300 nm periodically nano-patterned structures etched under the various r.f powers are calculated according to the AFM measurements and the results are also shown in Fig. 3. The aspect ratio is increased from 0.14 to 0.53 with increasing the etching power from 20 W to 48 W. More recently, J. Li et al. theoretically discussed the light-trapping capability of nano-structures with low aspect ratio [7]. They suggested that surface reflection can be reduced especially for the high energy photons (short wavelength region) and the optical absorption can be significantly enhanced even in the nano-patterned structures with low aspect-ratio.

Conclusion

Periodically nano-patterned Si structures have been fabricated by using PS nano-sphere lithography technique. AFM images revealed that the periodic structures can be formed by using PS monolayer as an etching mask. The lateral size and periodicity can be changed by using the PS nano-spheres with various sizes and the depths can be controlled by changing the etching parameters such as etching time and r.f power during the etching process. The formed nano-patterned Si structures exhibit the good antireflection characteristics though they have low aspect ratios. With increasing the diameter of nano-spheres and etching r.f power, the reflection can be significantly suppressed in a broad spectral range (400∼1200 nm) compared to the flat Si substrate. The reflection of nano-patterned Si structures can be lower than 10% in the whole measurement range. The mean reflection weighted by AM1.5 sunlight spectrum of the patterned Si nano-structures can be as low as 3.8% and the corresponding weighted mean absorption can be obviously enhanced to 95.7%, which is close to the best values reported previously. Our experimental results indicate that the periodically nano-patterned Si structures can reduce the surface reflection and enhance the optical absorption which can be potentially applied in the new generation Si-based thin film solar cells.

Acknowledgements

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