Anti-reflection for monocrystalline silicon from diamond-like carbon films deposited by magnetron sputtering

Xin Tan, Hang Zhai, Keke Meng and Zhongyi Zhang

College of Mechanical Engineering, Inner Mongolia University of Science & Technology, Baotou 014010, People’s Republic of China

* Authors to whom any correspondence should be addressed.
E-mail: heart_tan@126.com and mengkk@imust.edu.cn

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Abstract

In this work, diamond-like carbon (DLC) films used as anti-reflective coatings for monocrystalline silicon were deposited by magnetron sputtering for potential application in solar cells. The microstructural and optical properties of the films were investigated as a function of substrate temperature over a wide range during deposition. It showed that, when the substrate temperature increased from RT to 800 °C, the hybridized structures of the DLC films accordingly changed associated with a significant variation of refractive index between 2.22 and 1.64 at a wavelength of 550 nm. Three types of coating systems, namely single-, three- and five-layer films on monocrystalline silicon substrates, were designed based on the anti-reflection principle and fabricated in terms of the relationships of refractive index and deposition rate with substrate temperature. In particular, a well-designed three-layer film, of which the refractive index gradually changed along the thickness, that is 1.8, 1.9 and 2.0, respectively, was successfully deposited at one step on monocrystalline silicon substrates by adjusting substrate temperature and deposition time, and featured a broadband anti-reflective characteristic with low average reflectivity of 8.7% at a wide solar spectrum of 400–1100 nm. This work demonstrates that the DLC film has a promising application potential as broadband anti-reflective coatings in silicon-based solar cells.

1. Introduction

Monocrystalline silicon is an important semiconductor material in the application of solar cells [1–3]. Due to the inherently high refraction index (n = 4.0 at a wavelength of 550 nm), however, a significant Fresnel reflection loss of incident sunlight occurring at the air–silicon interface severely lowers the photovoltaic conversion efficiency of silicon-based solar cells [4–7]. It is fortunate that anti-reflective coatings widely found in some animals in nature, e.g., moth, butterfly and hawkmoths [8–10], reducing reflection losses at the interfaces. Various materials such as SiO2 [11, 12], TiO2 [13, 14], ZnO [15, 16] and SiO2–TiO2 [17] with different surface patterns have been developed to be used as anti-reflective coatings for silicon to improve solar cells performance. Particularly, due to unique properties including high-level of hardness (up to 80 GPa), high resistivity (up to 1015 Ω cm), wide band gap (1.0–4.0 eV) and adjustable refractive index (1.6–2.2), diamond-like carbon (DLC) films have been considered as a highly competitive candidate of anti-reflective coatings for silicon-based solar cells [18]. DLC has hybrid structures of diamond (sp3 bonding) and graphite (sp2 bonding), where the integrated area intensity ratio D peak to G peak together with the ratio of hydrogen (if it exists) and the size, type and ordering of sp2 clusters determines its main properties [19, 20]. Owing to the complexity of atomistic configurations and the diversity of preparation methods of the DLC films, however, it is still an challenge to meet the highly demanded optical application in solar cells.

To date, much effort has been devoted to develop DLC anti-reflective films. H Y Dai et al demonstrated that the refractive index of DLC films increased from 1.7 to 2.15 with sputtering power from 100 W to 180 W [21]. Dai et al pointed out that the refractive index of DLC films is in proportion to sp2-C content [22]. Won Seok Choi et al...
deposited DLC film on silicon substrates, and found that the reflectivity was 40% within 400–1000 nm [23]. Klyui et al proved that single or two-layer DLC antireflective coatings deposited on silicon substrates improved the solar cell performance by 1.35–1.5 times [24]. Yang et al found that the photovoltaic conversion efficiency of the solar cell increased from 4.12% to 5.2% by employing DLC films with a low surface reflectivity of approximately 14% as anti-reflective coatings on p-type (100) silicon wafers [25]. Overall, these works have made a great progress on the DLC films used as anti-reflective coatings in silicon-based solar cells, which, however, always focused on the specific and narrow visible band, but not the full utilizable solar spectrum. Here, a magnetron sputtering technique was employed to deposit DLC films on monocrystalline silicon substrates. Magnetron sputtering is preferred for industrial applications because of its versatility, its widespread use to sputter many materials, and its ease of scale up. The variation of optical properties of the DLC films with microstructures was analyzed as a function of substrate temperature during deposition. The profile of refractive index along thickness of multilayer DLC films was optimized to enhance the anti-reflective performance in a wide solar spectrum of 400–1100 nm.

2. Experimental details

DLC films were deposited on mirror-polished monocrystalline silicon substrates by magnetron sputtering technique using highly purified argon (99.99%) and graphite target (99.99%, 460 × 4 mm) as sputtering gas and carbon source, respectively. Prior to deposition, the substrates were ultrasonically cleaned in acetone, ethanol and deionized water each for 10 min. The vacuum chamber was evacuated to the base pressure of 3 × 10⁻³ Pa. Argon gas with a flow rate of 25 standard cubic centimeter per minute (SCCM) was introduced to the chamber by mass flow controller (MFC). The sputtering pressure of the vacuum chamber at about 1 Pa in all the experiments. Before the deposition, pre-spouting of the graphite target for 15 min. The sputtering power was 200W. A group of films was prepared at different substrate temperature from RT to 800°C. The film thickness was adjusted by controlling deposition time.

The surface and cross-section morphologies of the films were observed by using a field emission scanning electron microscopy (FESEM) (GAIA3, TESCAN, Czech Republic). The surface topography and roughness of the films were measured by using an atomic force microscopy (AFM) (Dimension EDGE, Bruker, Germany). The visible Raman spectra of the films were obtained by using a Raman spectroscopy (Finder Insight Pro, ZOLIX, China). The refractive indices and extinction coefficients of the films were determined by simultaneous fitting of the Forouhi-Bloomer Model [26] using an ellipsometer (M-2000, J. A. Woollam, USA) with a completeEASE software. The reflectance spectra of the films were obtained by using a spectrometer (Agilent Cary5000, Agilent, USA).

3. Results and discussion

Figure 1 shows the surface and cross-section morphology of the films deposited at different substrate temperatures for 1 h. As shown, all films surfaces are composed of plant of nanogranules equivalent in size that distribute uniformly and compact closely with each other, making the surfaces smooth, homogeneous and pinhole-free. Besides the dense film structure, a few defects like larger graphite particles exist on the surface can be also observed. The grain boundary seems to be not sharp, implying a poor crystallinity of the films. As the substrate temperature increased, the grains in the films gradually grew into round granules from initial flat blocks, and the surfaces became rougher. This appearance could be attributed to the high surface diffusion capacity of active carbon species induced by higher substrate temperature [27, 28]. In addition, as can be seen from the cross-section morphology in the insets of figure 1, there is no discernible delamination at the interfaces being indicative of good adhesion of the films to substrates which, thus, can ensure the propagation of incident sunlight along prescribed path and lower interface reflection losses. Moreover, the thickness of the films, determined from the cross-section images, increased with elevated substrate temperature. As shown in figure 2, the deposition ratio of the films increased from 3.7 nm min⁻¹ at RT to 7.3 nm min⁻¹ at 800°C.

Figure 3 shows the surface topography of the films as measured by AFM. As shown, the films were all composed of fine nanoscale particles, and had no obvious defects. A few uneven spikes were observed on the films surfaces due to the presence of larger particulates ablated from the graphite target as mentioned above. The surface roughness, Ra, of the films was measured over area of 10 × 10 μm² by AFM, as shown in figure 4. It can be seen that, for the substrate temperature no more than 600 °C, the surface roughness of the films remains at a low level of about 2.5 nm, which, however, rapidly increases to 3.9 nm when the substrate temperature increases to 800 °C. Nonetheless, the surface roughness of all the films on the order of less than 5 nm is far smaller than the wavelength of sunlight involved in photoelectric conversion in solar cells, and thus the interface scattering induced by surface rough structures could be negligible. The evolution of the surface topography could be explained from the diffusion process during films deposition, in which the carbon atom adsorbed onto film
surface gradually gather, and the surface will form clusters with more sp²-C, leading to the gradual increase in the roughness of the film [27].

Raman spectroscopy is a tool for the characterization of structure and bonding properties of carbon materials. Figure 5 shows the Raman spectra of the films with different substrate temperatures. A relatively broad band consisting of two characteristic peaks, i.e., D and G peaks, in the range of 1100–1800 cm⁻¹ indicates that typical DLC films were formed. The G peak results from stretching mode of all pairs of sp² sites in olefinic chains and aromatic rings, whereas the D peak results from breathing mode of all pairs of sp² sites in aromatic rings [29, 30]. Further, with the temperature increased, the shape of the Raman spectra significant changes. It has been demonstrated that the shape of Raman spectrum dominated by three independent factors including position of G peak (G-position), full width at half maximum of G peak (G-FWHM), and integrated area intensity ratio D peak to G peak (I_D/I_G) is associated with the structures of DLC films [29–33]. Generally, the change of G-position and I_D/I_G is related to sp² or sp³ bonding fraction in DLC films, and G-FWHM is a character of disorder degree of sp² clusters [34, 35].
Figure 3. Surface topography of the DLC films deposited under different temperatures. (a) RT, (b) 200 °C, (c) 300 °C, (d) 400 °C, (e) 500 °C, (f) 600 °C, (g) 700 °C, and (h) 800 °C.

Figure 4. Dependence of surface roughness of the DLC films with substrate temperature.

Figure 5. Raman spectra of the DLC films with different temperatures.
To extracted these factors, the Raman spectra of the DLC films were deconvoluted into two Gaussian peaks (i.e., D and G peaks) after a linear background subtraction, as shown in figure 5, and the extracted results are displayed in figure 6. As can be seen from figure 5, the D and G shoulders were gradually separated as the substrate temperature increased, generally suggesting the formation of nanocrystalline graphite by clustering aromatic rings. Expectedly, the extracted factors from the corresponding Raman spectra are supporting this inference. As observed from figure 6(a), G-position firstly shifts towards higher wave number with the increase of substrate temperature from RT to 400 °C and then declines as the substrate temperature increases further until to 800 °C. The upward shift of G-position represents higher sp² bonding fraction. However, ID/IG is found to follow the inverse change tendency with the substrate temperature as the G-position shift (figure 6(b)). It is noteworthy that the estimation of sp² or sp³ bonding fraction by G-position shifting is always affected by residual stress in the films [36], thus giving rise to a controversial result. So, a more reasonable conclusion derived from ID/IG observation is that the sp³ bonding fraction in the DLC films firstly increases, with the increase of substrate temperature from RT to 400 °C and then decreases when the substrate temperature further increases up to 800 °C. On the other hand, as shown in figure 6(a), G-FWHM gradually decreases, indicating that prepared DLC films have a high graphitic character. In fact, elevated appropriate substrate temperature can promote the diffusion and migration of carbon atoms on the film surface, which can reduce various defects inside the DLC film, and also increase sp³ bonding fraction [37]. However, When the substrate temperature exceeds appropriate substrate temperature, sp³ bonding in the DLC film may be converted into sp² bonding [37].

For the sake of quantitatively determine the bonding characteristics and hybridization states of the DLC films, Using XPS to analyze DLC film, as show in figure 7(a). C1s spectra of the DLC films were deconvoluted into three components, as typically displayed in the inset of figure 7(a), among which components around 284.5eV and 285.1eV are assigned to sp²-C and sp³-C respectively, while the little component at 286.1 eV is assigned to C–O contamination formed on the films surfaces due to adsorbe air oxygen element [38]. The sp³-C

![Figure 6](image6.png)  
**Figure 6.** Extracted factors from the Raman spectra of the DLC films. (a) G-position and G-FWHM; (b) ID/IG.

![Figure 7](image7.png)  
**Figure 7.** (a) XPS spectra and (b) sp³/sp² ratio of the DLC films.
and sp^3-C peaks areas ratio can be used to determine the sp^3/sp^2 ratio of the films, and the result is shown in figure 7(b). It is clear that the sp^3 bonding fraction firstly increases with the increase of substrate temperature from RT to 400 °C, and then decreases when the substrate temperature further increases up to 800 °C. This result agrees well with the Raman analysis discussed above. It has been reported that DLC films deposited by magnetron sputtering could contain hydrogen to some extent due to water vapor contamination in the chamber [38]. Thus, it should be noted that the sp3 component in the films arises from both sp^3 C-C and sp^3 C-H bonds. However, unfortunately, the sp^3 C-C and sp^3 C-H bonding types cannot be clearly distinguished in C1s spectra. This opinion is crucial for addressing the following optical issues of the DLC films.

Figure 8(a) shows the refractive index curves of the DLC films deposited at different substrate temperatures and silicon substrate (in the inset) at a spectral range of 400–1100 nm. It is clear that, by controlling the substrate temperature used during film deposition, the refractive index of the DLC films tune over a wide range. Particularly, increasing the substrate temperature from RT to 700 °C had a general effect of decreasing the refractive index of the DLC films at a wavelength of 550 nm ranging from 2.18 to 1.64 (figure 8(b)). Further increasing the substrate temperature to 800 °C then led to a slight increase in the refractive index from 1.64 to 1.73. The refractive index of silicon at 550 nm was determined to be around 4.0. Refractive index of an DLC film often is the representation of the photon scattering effect of the carbon atoms. So, the changes in the refractive index of the DLC films with the substrate temperature can be explained by the structure evolution as revealed by Raman and XPS spectra discussed above. Generally, the more sp^2 bonding the film contains, the smaller the refractive index is. On the other hand, the incorporation of hydrogen in DLC films will also lower the refractive index. Considering optical properties and the sp^3 or sp^2 bonding fraction from Raman and XPS analysis, we guess that when the substrate temperature increases from RT to 400 °C, the increasing level of incorporated hydrogen in the DLC films results in the decrease of refractive index, and the decrease of sp^3 bonding leads to further decrease of refractive index with the substrate temperature increases from 400 °C to 800 °C. In this context, a nonuniform DLC film with gradient change in the refractive index along thickness can be obtained according to the relationship between the refractive index, deposition rate and substrate temperature.

The extinction coefficient of the DLC films deposited at different substrate temperatures is presented in figure 9. It can be seen in figure 9(a) that the extinction coefficient of the films decreases as the wavelength increases. Meanwhile, at a certain wavelength, e.g. 550 nm, the extinction coefficient decreases first with increasing substrate temperature from RT to 400 °C and then increases as the substrate temperature further increases up to 800 °C, as shown in figure 9(b). It is clear that, the change trend of extinction coefficient with substrate temperature agrees well with that of sp^3 bonding fraction in the DLC films as revealed by Raman and XPS spectra discussed above.

As known, the refractive index for a single antireflective film should be equal to the square root of the refractive index of substrate [39], and the optical thickness is equal to quarter of target wavelength (λ). For broadband antireflection, a multilayer film with layer-number of odd is favored. The refractive index of the film reduces in step from substrate to air, and the optical thickness follows the rule of λ/4, λ/2, λ/4 … λ/2, λ/4. The λ/2 film just equal to double λ/4 films will not affect the target wavelength for anti-reflection, but can regulate the edge of the whole band to be more flat. To investigate the anti-reflective performance of the DLC films on monocrystalline silicon substrates, three types of film systems, i.e. single-, three- and five- layer DLC films as schematically illustrated in figure 10, were designed based on the principle of anti-reflection discussed above and fabricated by adjusting substrate temperature and deposition time during magnetron sputtering. For preparing
the single-layer film, the substrate temperature was fixed at 400 °C. For preparing the three-layer film, from bottom to top layer, the substrate temperature is controlled at 400 °C, 500 °C, 650 °C, 800 °C and 700 °C, respectively. Here, for the photovoltaic application, the reflectance of the DLC films at a wavelength of 550 nm is taken into consideration, which corresponds to the part of maximum solar spectral intensity. The reflectance spectra of bare silicon substrate and single-, three- and five-layer DLC films on silicon substrates at a spectral range of 400–1100 nm are displayed in figure 11. As indicated, after silicon substrates were coated by DLC films, the surface reflectance decreased significantly, suggesting that the DLC films have an excellent anti-reflection properties. It is noteworthy that the DLC films with different refractive index profiles have different anti-reflection properties. For the single-layer DLC film, the reflectance curve shows a typical V-shaped profile with the minimum reflectance of less than 0.1% at a wavelength of ∼500 nm. The slight deviation of central wavelength from the target one of 550 nm should derived from the experimental errors of refractive index and thickness of the DLC films. The average reflectivity of a single-layer anti-reflection film between 400–1100 nm was approximately 19.8%. For the three- and five-layer DLC films, the flattened reflectance curves reflect a good broadband antireflective performance. The three- and five-layer DLC films feature very low average reflectivity of 8.7% and 13.9% at a wide solar spectrum of 400–1100 nm, respectively. The deterioration of anti-reflective performance of the five-layer DLC films compared with that of three-layer one should be attributed to the enhancement of absorption and scattering of light as the number of film layer increases.

4. Conclusion

In this paper, smooth and homogenous DLC films were deposited on monocrystalline silicon substrates by using magnetron sputtering at different substrate temperatures, and the anti-reflective performance of the films was optimized for the application in silicon-based solar cells through comprehensive microstructural and optical study. The results indicated that, the fraction of sp\(^2\) sites firstly increased and then decreased as the substrate temperature varied from RT to 800 °C, giving rise to a significant variation of refractive index at a wavelength of
550 nm between 2.18 and 1.64. A single-layer DLC film featured an excellent single-band antirefection performance with the minimum reflectance of less than 0.1% at a wavelength of ~500 nm. In particular, a well-designed three-layer DLC film with optimized graded refractive index featured a good broadband antireflective performance with very low average reflectivity of 8.7% at a wide solar spectrum of 400–1100 nm. As the layer number further increased, the anti-reflective performance of the five-layer DLC film deteriorated as a result of the enhancement of absorption and scattering of light. This work demonstrates that the DLC film has a promising application potential as broadband anti-reflective coatings in silicon-based solar cells.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Declaration of interest statement

We declare that we have no financial and personal relationships with other people or organizations that can inappropriately influence our work, there is no professional or other personal interest of any nature or kind in any product, service and/or company that could be construed as influencing the position presented in, or the review of, the manuscript entitled 'Anti-reflection for Monocrystalline Silicon from Diamond-like Carbon Films Deposited by Magnetron Sputtering'.

ORCID iDs

Xin Tan @ https://orcid.org/0000-0001-8314-8032

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Figure 11. Reflectance spectra of bare silicon substrate and single-, three- and five-layer DLC films on silicon substrates.
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