

Research Article

Fabrication of High Transparency Diamond-Like Carbon Film Coating on D263T Glass at Room Temperature as an Antireflection Layer

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This study intends to deposit high transmittance diamond-like carbon (DLC) thin films on D263T glass substrate at room temperature via a diamond powder target using the radio frequency (RF) magnetron sputtering technique. Moreover, various process parameters were used to tune the properties of the thin films by using the Taguchi method. Experimental results show that the content of sp^3 bonded carbon decreases in accordance with the effect of the substrate temperature. In addition, the hardness of all as-deposited single-layer DLC films ranges from 13.2 to 22.5 GPa, and the RMS surface roughness was improved significantly with the decrease in sputtering pressure. The water repellent of the deposited DLC films improved significantly with the increase of the sp^3 content, and its contact angle was larger than that of the noncoated one by 1.45 times. Furthermore, the refraction index (n) of all as-deposited DLC films ranges from 1.95 to 2.1 at $\lambda = 600$ nm. These results demonstrate that the thickness increased as the reflectance increased. DLC film under an RF power of 150 W possesses high transmissive ability (>81%) and low average reflectance ability (<9.5%) in the visible wavelengths (at $\lambda = 400$ –700 nm).

1. Introduction

Diamond-like carbon (DLC) containing a large proportion of combined sp^3/sp^2 -hybrid bonds has extreme properties similar to diamonds [1–5]. It is possible to prepare DLC films for several important applications in the fields of mechanical manufacturing, solar energy devices, electricity devices, and so forth, owing to their excellent properties, including high hardness, high thermal conductivity, high electrical resistivity, low infrared (IR) absorption, transparency to visible light, hydrophobic behavior, and chemical inertness [6–11]. Recent researches and technology have focused on keeping high transmission with reduced reflectance capability to maintain enough visibility in a protectively antireflecting coating for solar energy; this type of optical coating can be applied to the surfaces of lenses and other optical devices to reduce

reflection [12, 13]. Thus, this study proposes a novel technique which can keep the surface water repellent and improve the surface by lowering reflection.

In this study, hydrogen-free DLC films were deposited at room temperature by using radio frequency (RF) magnetron sputtering with a unique target consisting of artificial nanodiamond powders. The nano-diamond powder used in this work was a commercial product, which was synthesized by the high-pressure high-temperature (HPHT) method. The diamond target fabricated in the RF magnetron cathode for DLC sputtering was composed of high-purity diamond powder with a mean particle size of about 100 nm. The nano-diamond target sputtering process easily formed high sp^3 content on the thin film at a low temperature [6, 10, 14]. For optical applications, previous studies have indicated that the optical properties of DLC films vary between those of

diamond (sp^3 bonding) and graphite (sp^2 bonding). The wavelengths of the refractive index (n) and extinction coefficient (k) of DLC depend on the C energies on the substrate [15]. On the other hand, the most important parameter of the film is the thickness. DLC films have been applied as both protective and antireflective coatings for optoelectronic devices and silicon solar cells that are even more demanding in terms of well-defined thickness and the specific optical properties of the films [16, 17]. It is well known that light transparency and hydrophobic behavior depend on D263T glass-based materials. The D263T glass substrate is a borosilicate glass that is produced by melting very pure raw materials. This type of substrate has high luminous transmittance. The properties of D263T glass represent the standard for optical glass. The performances of films deposited onto D263T glass substrate vary under different parameters such as sputtering power, working distance, sputter pressure, and deposition time in the sputtering process. Experiments have carried out to understand the effects of these process parameters. Further discussion of the results of these experiments will be provided in the following. The purpose of this work was to deposit thin films on D263T glass substrate under various process parameters to tune the properties of the thin films by using the Taguchi method, wherein the only estimation standard for steadiness and robustness was to determine a variance index identified as the signal to noise ratio (S/N) [18–22]. Moreover, a lesser number of experiments are desired in the sputtering process when using the Taguchi method. This study utilized the Taguchi method to design experiments involving as-deposited hydrogen-free DLC films on D263T glass substrates at room temperature by the RF magnetron sputtering system. The experiments focused on the significance of the process parameters of DLC films, indicating that using four factors with three levels represents an L_9 (3^4) orthogonal array. According to these analysis results, fewer experiments (only nine experimental runs) were required to study all levels of input parameters. We also filtered out some effects owing to statistical variation [21]. The four factors include sputtering power, working distance, sputtering pressure, and deposition time. The ranks of each factor were considered as major influences in the experiment via the analysis of variance (ANOVA) approach. Thus, the Taguchi method was utilized as an engineering methodology to find a correlation between control factors and product characterization. The corresponding microstructure, optical performance, and mechanical properties of the thin films were identified by a scanning electron microscope (SEM), X-ray photoemission spectroscopy (XPS), atomic force microscopy (AFM), a UV/Vis spectrophotometer, and contact angle goniometry, respectively. XPS can be summed up as the measurement of kinetic energy of the inner or valence electron ejected by used an incident photon with energy $h\nu$. The binding energy is sensitive to chemical bonds in a compound and is related to the energy of ionization. The measured kinetic energy of photoelectrons generally ranges between 0 and 1000 eV in DLC films, and the depth of inspection is about 40–50 Å. As mentioned previously, XPS is a highly sensitive analyzer which offers a better method for

the surface properties of the sample. The C1s peak of a-C:H films is in the range from 284.5 to 286.4 eV, as the full width at half maximum (FWHM) of the C1s peak of a-C:H is around 1.6–1.7 eV, relatively larger than the corresponding FWHM for diamond and for graphite. XPS was carried out the Mg K- α -line, which analyzed about the top 4 nm of the film, indicating that the spectra were determined with the C1s peak of \sim 285 eV. Also present was the O1s peak of 531 eV, owing to surface chemisorbed oxygen species. Thus, the sp^2 : sp^3 ratios determined using XPS data will represent the surface values [23–25].

2. Experimental Methods

This study employs the Taguchi methods to design the experiment in which as-deposited hydrogen-free DLC films are deposited onto D263T glass substrate ($10 \times 10 \times 0.55$ mm) at room temperature by an RF magnetron sputtering system with an artificial diamond powder target of 3 inches in diameter as the magnetron source in an argon plasma. The diamond powder as the sputter target was easily synthesized with high sp^3 content in thin film at low temperature by using the proposed sputtering method owing to the nanocrystalline diamond powders. With a larger fraction of sp^3 bonding, the properties of DLC coatings will be skewed toward diamond. The content of sp^3 is very much dependent on the ion energy during deposition [26]. The DLC films were deposited by using sputter and the sputtering power; working distance, sputter pressure, and deposition time were used as the deposition parameters. In the sputtering process, the heat flux to the substrate relates to the thermalization coefficient which is the ratio of particle energy at the substrate to that at the target. This ratio depends on the product of gas pressure and substrate-target distance [27]. Therefore, D263T glass substrates were placed at distances between 8 and 10 cm from the target with a rotation speed of 15 rpm. DLC films have been deposited at RF powers ranging from 150 to 200 W and at an rf of 13.56 MHz with capacitive coupling, and the film deposition time was in the range from 0.5 to 2 h under sputter pressure ranging from 1 to 5 mTorr. The corresponding three level experimental designs for depositing DLC films onto D263T glass substrate are listed in Tables 1 and 2. This study carried out the experiments by using the sputtering process conditions for an L_9 (3^4) orthogonal array in all as-deposited DLC films. The structured bonding of the DLC films under different deposition parameters was examined by X-ray photoelectron spectroscopy (XPS). The contact angle of the samples was measured by the sessile drop method using a First Ten Angstroms 1000B (FTA 1000B) contact angle goniometry. The liquid was dropped by using 3 μ L droplets of deionized (DI) water. All measurements were carried out at room temperature. A UV/Vis spectrophotometer was used to measure the corresponding optical transmittance of the DLC films deposited onto D263T glass substrate. The corresponding all the specimen's optical properties of refractive index (n), extinction coefficient (k), transmittance, and reflectance and film thickness were determined by

TABLE 1: The design of the experiment defines the deposition parameters of DLC films for each factor and level.

Symbol	Factor	Level 1	Level 2	Level 3
A	RF power (W)	150	175	200
B	Working distance (cm)	8	9	10
C	Sputtering pressure (mTorr)	1	3	5
D	Deposition time (h)	0.5	1	2

TABLE 2: Design of experiments (DOE) according to L_9 (3^4) orthogonal array.

Specimen (GD)	Control factors			
	RF power (W)	Working distance (cm)	Sputtering pressure (mTorr)	Deposition time (h)
1	150	8	1	0.5
2	150	9	3	1
3	150	10	5	2
4	175	8	3	2
5	175	9	5	0.5
6	175	10	1	1
7	200	8	5	1
8	200	9	1	2
9	200	10	3	0.5

using a UV/Vis spectrometer (Mission peaks optics, MP100-M) equipped with an optical microscope. The instrument with multiple gratings measures to achieve the interference between incident and reflected light with a wide spectrum ranging from UV to visible range (380–950 nm) [28].

3. Results and Discussion

3.1. XPS and Microstructure Analysis of DLC Films. Figure 1 shows the response graph of the four factors versus sp^3 content. The ranking of the order of the magnitude of the sp^3 content ranges for various levels. The leading factor affecting process parameters is deposition time, followed successively by RF power, sputtering pressure, and working distance. XPS is employed to obtain the quantitative results of film composition (sp^2 : sp^3), which are chemically specific, as each element has its own core level. The deconvolution of the C1s peak of DLC film is obtained by an XPS curve fitted by using the three-curve methods. The sp^3 fraction of DLC films was derived from XPS fitting for the C1s core peak, with peaks formed by diamond (285.1 eV), graphite (284.6 eV), and carbon-oxygen (286.5 eV) bonds. The area of each peak depends on the corresponding phase. The sp^3 content was estimated by taking the ratio of the diamond peak over the sum of the diamond, graphite, and carbon-oxygen bonds peak areas. In this study, each component was obtained by using a convolution of Gaussian (80%) and Lorentzian (20%) methods, and background subtraction was approximated by the Shirley method. The Gaussian widths of the spectra for DLC films are 1.35 eV, and the component depends on the

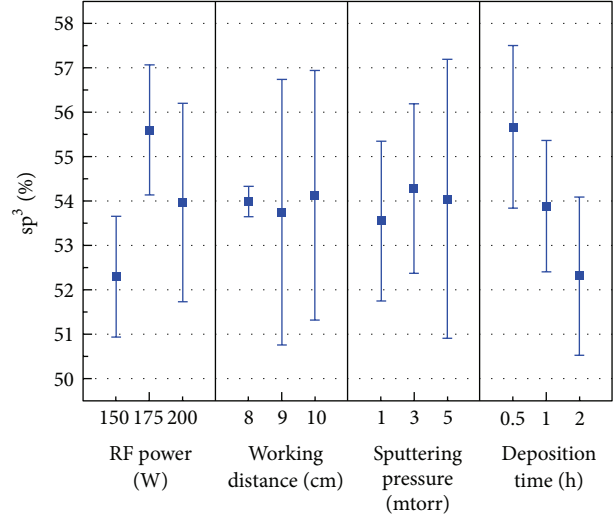


FIGURE 1: Response graph of the four factors versus sp^3 content. The ranking order of the magnitude of sp^3 content at various levels indicates that the effects of various factors on the sp^3 content are ranked in the following order: deposition time, RF power, sputtering pressure, and working distance.

sp^2 and sp^3 hybridization results in each specimen shifted by 0.5 eV, as shown in Figure 2 [23–25]. According to the XPS results, the probable sp^3 content of the specimens was reduced from 56.17% to 50.92% in correspondence with increased deposition time. The findings demonstrate that the content of the sp^3 bonded carbon becomes lower in reference to the effect of the substrate temperature. The implantation of carbon ion during deposition generates heat and warms up the substrate rapidly. The deposition time increases from 0.5 h to 2 h when significantly increasing the temperature on the substrate. The high temperature of the substrate can partially release the compressive stress induced by carbon ion implantation and obstruct the formation of sp^3 bonds. The results can affect transform of the sp^3 bond into an sp^2 bond [29]. On the other hand, this study assumes that the sp^3 content varies with RF power. For an RF power of 150 W, the low proportion of sp^3 bond in the thin film was formed from the diamond target via the sputtering process owing to the low bombardment energy. The bombardment energy is enhanced on the Ar plasma at the RF power of 175 W, indicating that the bonds of diamond can be broken by powerful bombarding atoms [10].

Figure 3 shows the typical plan view surface of SEM images, and the insets display the cross view. The results showed that as-deposited DLC films are considerable smooth morphology; the shrink and warped deformations were not observed from the surface of thin films. Figure 3(d) shows cross view of DLC film (specimen GD 3), indicating that the thickness is 30 nm. Through SEM analysis results, it clearly indicated never the existence of highly internal stress in such a thick DLC films leading to films peeling off from the D263T glass substrate. Figure 4 indicates the hardness of the DLC films analyzed by the nanoindentation test with a

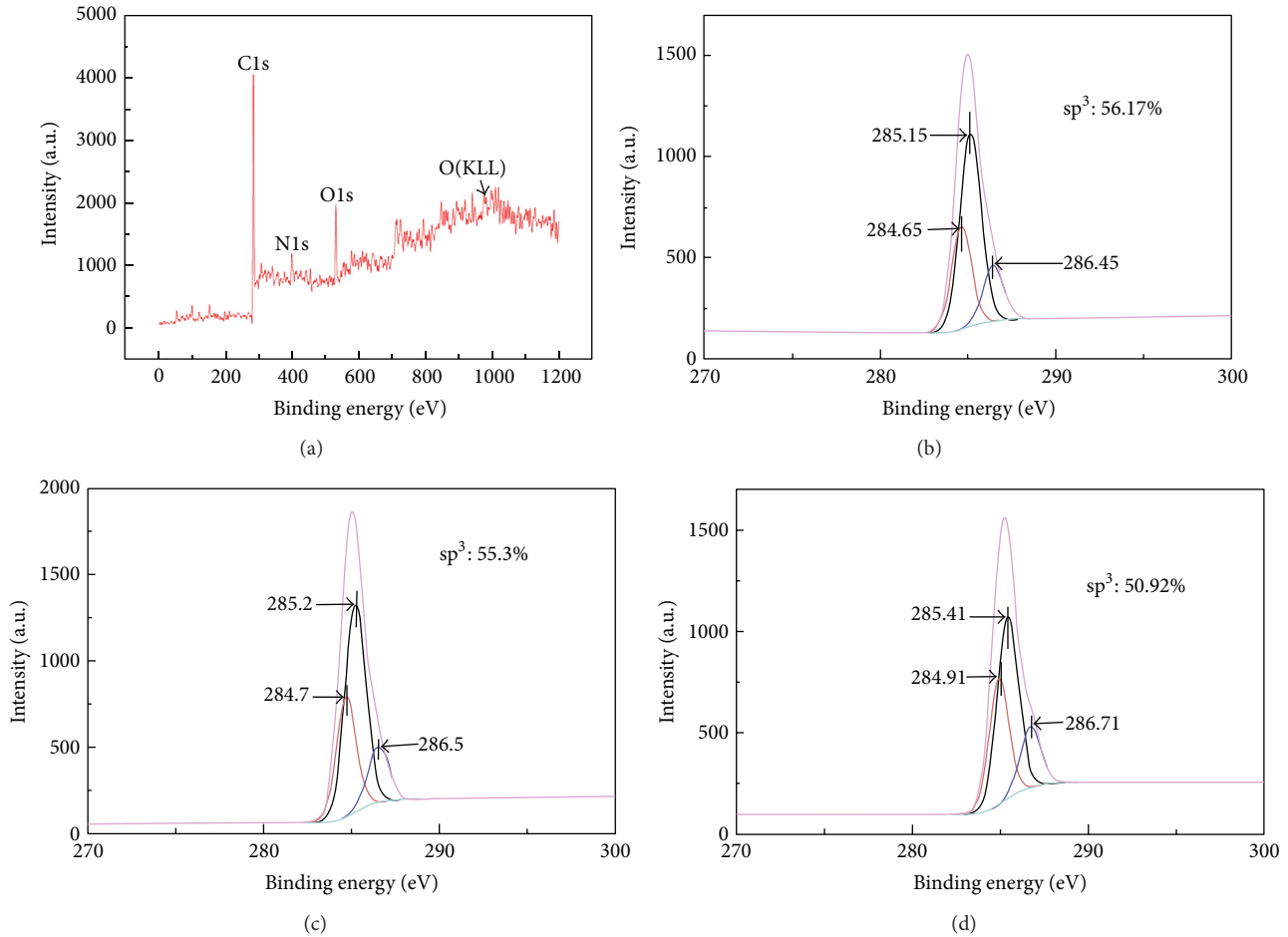


FIGURE 2: XPS survey spectrum (a) and the deconvolution of the C1s binding energy peak of the XPS spectrum of the DLC films; (b) DLC film (specimen GD 9) deposited under a sputtering power of 200 W, work distance of 10 cm, working pressure of 3 mTorr, and deposition time of 0.5 h. (c) DLC film (specimen GD 6) deposited under the sputtering power of 175 W, work distance of 10 cm, working pressure of 1 mTorr, and deposition time of 1 h. (d) DLC film (specimen GD 3) deposited under the sputtering power of 150 W, work distance of 10 cm, working pressure of 5 mTorr, and deposition time of 2 h.

large loading of 500 μN . A scanning probe microscopy (SPM) apparatus was used to conduct nanofriction, nanowear, and fractal investigations to determine the nanotribological characteristics of the deposited DLC films. Fractal analysis is applied to quantitatively characterize the surface roughness of films measured by AFM. The aforementioned NIP test results showed that the hardness behaviors of all as-deposited DLC films improved significantly with the increase of the sp^3 content, and the hardness of the as-deposited films was greater than 13.2 GPa.

3.2. RMS Surface Roughness Analysis of DLC Films. Figure 5 shows the response graph of the four factors versus RMS surface roughness. The ranking of the order of the magnitude of the RMS surface roughness differs at various levels, indicating that the effects of various factors on the RMS surface roughness increase in accordance with sputtering pressure, deposition time, working distance, and RF power. The surface roughness values of DLC films have been obtained by AFM analysis. AFM measurements presented here were obtained

in the tapping mode, using a Digital Instruments Nanoscope machine. Typical scan areas were over $30 \times 30 \mu\text{m}^2$, and the scan rate was approximately 1 Hz. Surface RMS roughness was obtained from a complete scan, using the software supplied with the AFM system. From this measurement it is clear that all as-deposited DLC films were lower than 1.9 nm with a scanning area of $30 \times 30 \mu\text{m}^2$, and the RMS surface roughness in the DLC films was improved significantly with the decrease of the sputtering pressure. In this study, the formation of nanoparticles during vapor deposition strongly depends on the sputter pressure. At higher plasma pressure, there is an increase in the number of inelastic collisions which leads to a decrease in the diffusivity of carbon in the D263T glass substrate owing to the significantly reduced energies. Thus, the films deposited at higher plasma pressure demonstrate an increased RMS surface roughness. At low pressures, it is evident that smooth DLC films are formed. This result is consistent with previous results, and this can be understood on the basis of the large differences between the atomic masses of carbon and argon which causes carbon

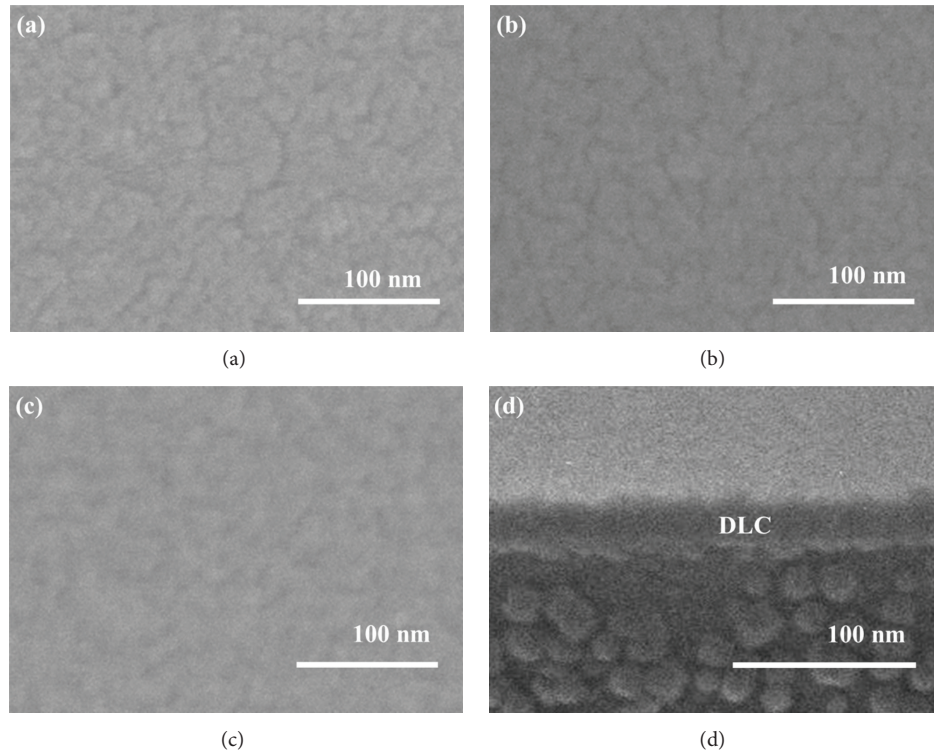


FIGURE 3: SEM images of DLC film are deposited on D263T glass substrate at room temperature by using RF magnetron sputtering technique. (a) SEM images of DLC film deposited referred to the specimen GD 9. (b) SEM images of DLC film deposited referred to the specimen GD 6. (c) SEM images of DLC film deposited referred to the specimen GD 3, and (d) the cross-section view shows the corresponding for image (c), which is 30 nm thick DLC film.

atoms to lose most of their kinetic energies before reaching the substrate at higher plasma pressures. This is expected to lead to the formation of thin film with high surface roughness [30].

3.3. Hydrophobicity Analysis of DLC Films. Figure 6 shows a response graph of four factors versus contact angle. The ranking order of the magnitude of contact angle at various levels indicates that the effects of various factors on the contact angle are ranked in the following order: RF power, deposition time, working distance, and sputtering pressure. A number of researchers have reported that the contact angle depends on the sp^3 content of the film. It may be that hydrophobicity significantly decreases with the increase of sp^2 bonded carbon in the films. The findings of this study may demonstrate that the surface energy varies at different carbon hybridized states, which modify the surface properties and associated hydrophobic/hydrophilic behavior. The surface energy of the sp^3 terminated surface is very high because of its strong covalent character compared with that of the sp^2 terminated surface as sp^2 bonded graphite exhibits weak polarity due to its dangling bonds [31–33]. In this study, the DLC films under the RF power of 175 W show higher hydrophobicity than those under the RF power of 150 W and 200 W. It is clear that those films have high sp^3 carbon content. Thus, the wetting contact angle of DLC films can be

modified and controlled by the variation of sp^3 content to obtain a hydrophobic or hydrophilic surface property.

3.4. Optical Properties Analysis of DLC Films. Figure 7 illustrates the response graph of four factors versus transmittance. The ranking order of the magnitude of transmittance at various levels indicates that the effects of various factors on the transmittance are ranked in the following order: RF power, deposition time, working distance, and sputtering pressure. The UV/Vis spectrophotometer shows that the optical properties of the films deposited with a single DLC layer are obtained from the five-point position of the films onto D263T glass. Figure 8 shows the refraction spectra for the D263T glass substrates coated with and without DLC films prepared under the sputtering process parameters, and the thickness of the as-deposited amorphous DLC films ranges from 17 to 66 nm. From the previous results, the values of the RMS roughness surface of the thin films are lower than 1.9 nm. It can be concluded that the effect of light scattering from the thin films was negligible. Thus, the reflectance of the thin films was decreased significantly with increasing transmittance. The reflectance in the visible wavelengths (at $\lambda = 400\text{--}700\text{ nm}$) significantly increases with increasing RF power. This is associated with the larger thickness. The total reflection changes with the refraction indices of the films and substrate, n and ns , respectively. As the refraction index of the substrate, ns , is larger than that of thin films, n , the resultant

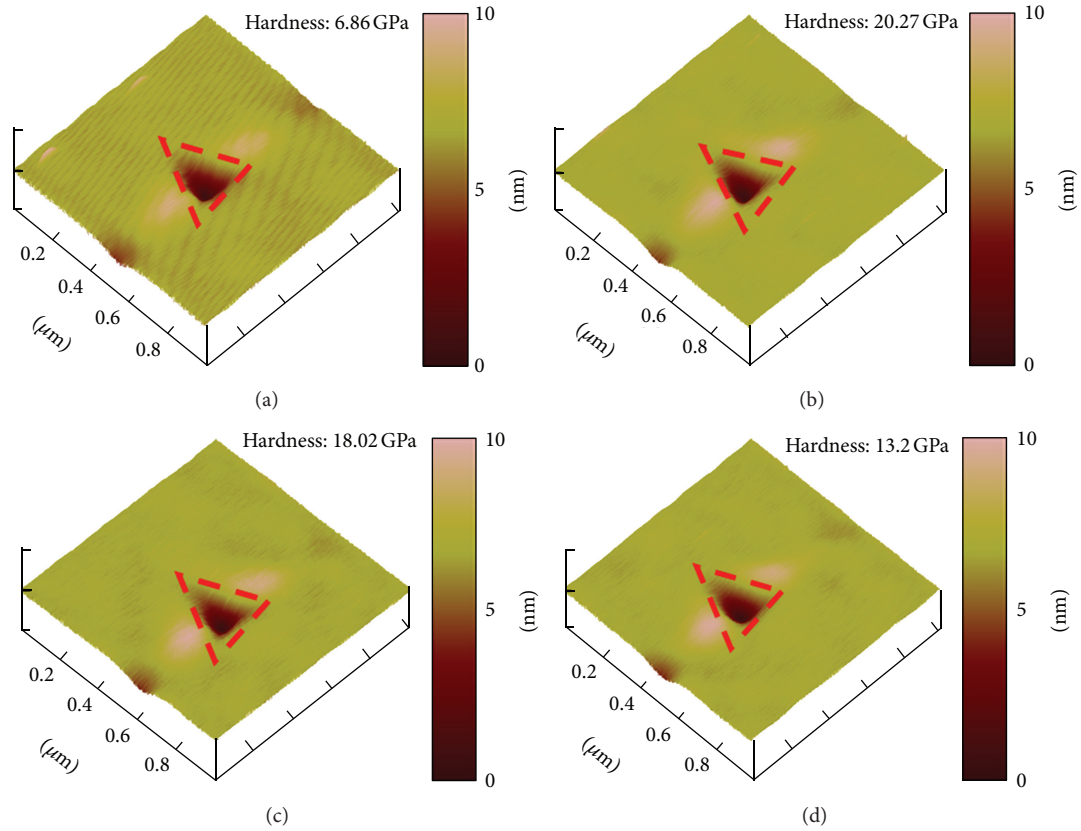


FIGURE 4: AFM topography after the nanoindentation test for the D263T glass substrates coated with and without DLC films prepared under a large load of 500 μ N: (a) D263T glass substrates, (b) specimen GD 9, (c) specimen GD 6, and (d) specimen GD 3.

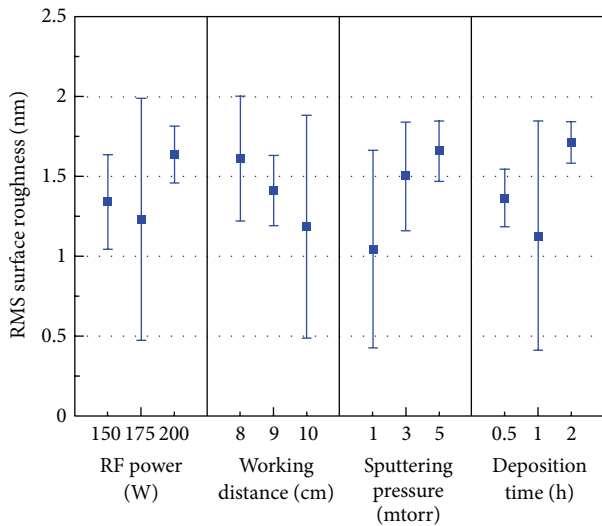


FIGURE 5: Response graph of four factors versus RMS surface roughness. The ranking order of the magnitude of RMS surface roughness at various levels indicates that the effects of various factors on the RMS surface roughness are ranked in the following order: sputtering pressure, deposition time, working distance, and RF power.

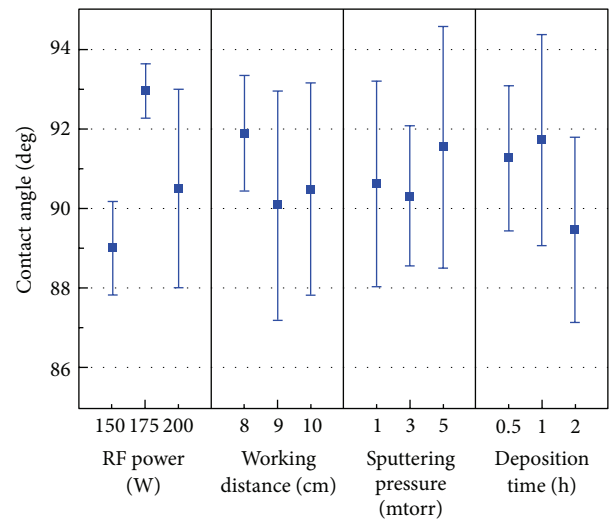


FIGURE 6: Response graph of four factors versus contact angle. The ranking of the order of the magnitude of the contact angle differs at various levels, indicating that the effects of various factors on the contact angle increasing in accordance with RF power, deposition time, working distance, and sputtering pressure.

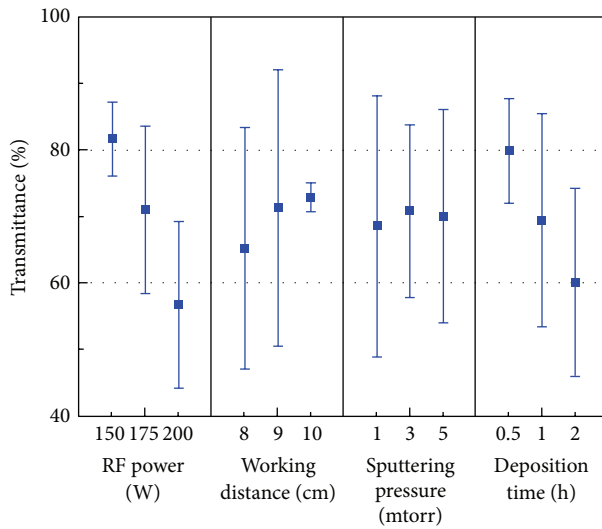


FIGURE 7: Response graph of the four factors versus transmittance. The ranking of the order of magnitude of transmittance for various levels indicates that the effects of various factors on the transmittance are ranked in the following order: RF power, deposition time, working distance, and sputtering pressure.

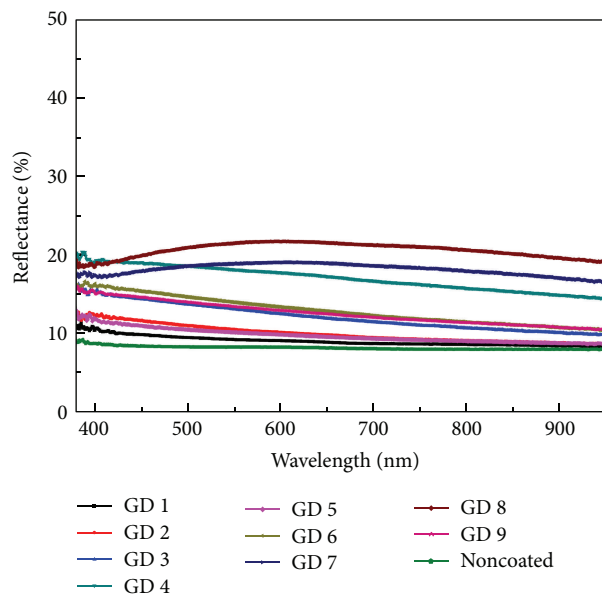


FIGURE 8: The various average reflectance of DLC films deposited by RF magnetron sputtering onto D263T glass.

reflection will be reduced. This is referred to as constructive interference or broken interference [10]. In this study, the reflectance of the as-deposited DLC films is higher than that of the noncoated one. This indicates that the refractive index of thin films is larger than that of the substrate one. The refractive index (n) of D263T glass substrate is 1.52. The UV/Vis spectrophotometer measurements show that the refractive index (n) of all as-deposited DLC films ranges from 1.95 to 2.1 at $\lambda = 600$ nm. These results demonstrate that the thickness was increased correspondingly with the increasing

reflectance. On the other hand, under an RF power of 150 W and a deposition time of 0.5 h, the specimen GD 1 had high transmissive ability ($>81\%$) and low average reflectance ability ($<9.5\%$) in the visible wavelengths (at $\lambda = 400\text{--}700$ nm). This result could make an antireflection coating for solar cell devices available, which would improve efficiency [9].

4. Conclusions

The present work shows that DLC films have been successfully deposited onto D263T glass substrate at room temperature via a diamond powder target using the radio frequency (RF) magnetron sputtering technique and Taguchi method. Experimental results show that the content of sp^3 bonded carbon decreases due to the effect of substrate temperature. The NIP test results showed that the hardness of the prepared DLC films ranges from 13.2 to 22.5 GPa. Moreover, the water repellence of the deposited DLC films improved significantly with increasing sp^3 content, and its contact angle is larger than that of the noncoated one by 1.45 times. The reflectance in the visible wavelengths (at $\lambda = 400\text{--}700$ nm) significantly increases with increasing RF power, which is considered to be associated with the larger thickness. In addition, the refractive index (n) of all as-deposited DLC films ranges from 1.95 to 2.1 at $\lambda = 600$ nm. Furthermore, when the RF power was set to 150 W with the deposition time of 0.5 h, the prepared DLC films possessed high transmissive ability ($>81\%$) and low average reflectance ability ($<9.5\%$) in the visible wavelengths (at $\lambda = 400\text{--}700$ nm).


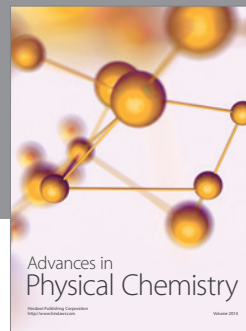
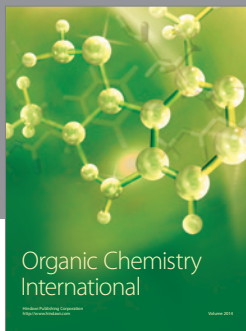
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