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Further improving the mechanical and tribological properties of low content Ti-doped DLC film by W incorporating

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ABSTRACT

W/Ti-doped diamond-like carbon (DLC) films were fabricated on Si substrates by co-sputtering W and Ti targets in methane and argon mixture atmosphere. The composition and the microstructure, internal stress, mechanical and tribological properties of the films were measured by X-ray photoelectron spectroscopy (XPS), high-resolution transmission electron microscopy (HRTEM), Raman spectra, BGS 6341 type film stress tester, nano-indentor and reciprocating ball-on-disc tester, respectively. The results indicated all films showed the amorphous structural characteristics and a constant thickness value of ~600 nm (195 ± 15 nm Ti interlayer was designed in order to minimize the influence of Ti interlayer), and the Ti content increased from 0 to 34.5% as the W target current increased from 0 A to 14 A. As the W concentration increased from 0 to 2.6 at.%, the hardness increased to 12.7 GPa and the internal stress maintained at a low value and almost no change. Meanwhile, the friction coefficient and wear rate showed the lowest value (0.023 and 1.2×10^{-8} mm^3/N m, respectively). With the W content increased to 34.5 at.% further, the internal stress, friction coefficient and wear rate of film are dramatically increased.

1. Introduction

Diamond-like carbon (DLC) film as an effective protected coating, due to its excellent physicochemical properties like high hardness, low friction coefficient and wear, is promising candidate for reducing the wear loss during manufacturing operations including machining and tribological systems such as the plungers running against the cylinder in fuel injection system of automotives [1–5]. Unfortunately, poor adhesive strength with metallic substrates is the major drawback, which severely limits the successful application of DLC films. Overall, two main reasons are expected to explain it. On the one hand, high residual stress ranging from 1 to 10 GPa lead to spall off from metallic substrates, at the same time, limit the film thickness [6–8]. This one could be effectively solved by the doping elements such as Ti, Cr, W and so on [9–11]; On the other hand, weak adhesive strength at the boundary between the film and substrate, which could be solved by the introduction of the addition Me, Me_XC, Me_XN interlayer [12–14]. Above mentioned these attempts to enhance the adhesion have proved to be very successful, but most works are based on the cost of sacrificing the mechanical properties [15,16]. Fortunately, our previous studies indicated that the introduction of low content Ti not only significantly reduced the residual stress but also retained the high hardness [17,18].

However, our works has only shown that the internal stress has been effectively reduced without changing the hardness by incorporating low content Ti atoms. On the basis of it, if other elements were also introduced into the low content Ti-doped DLC film, so that it can effectively enhance the hardness. Namely, if the incorporated elements could be introduced successfully into the DLC films together and carried out “their duties”, so more excellent properties could be obtained. Silva et al. [19] found that the addition of W atom led to a significant hardening of the DLC coating from 10 GPa to 18 GPa. Yue and co-workers [20] investigated the W-doped DLC film, and found that the W content increased to 10.73 at.%, the hardness is increased to ~20 GPa, they all suggested that the enhancement of the hardness is probably due to the formation of hard phase WC disperses in the carbon matrix. Besides, many other researches also indicated that W is considered an effective doping element especially in improving the high temperature tribological properties of film [21,22].

In this study, W/Ti-doped DLC films with the different W content were deposited on the Si substrate by co-sputtering W and Ti targets in methane and argon mixture atmosphere. Our concept is W and Ti atom will not react to generate the intermetallic

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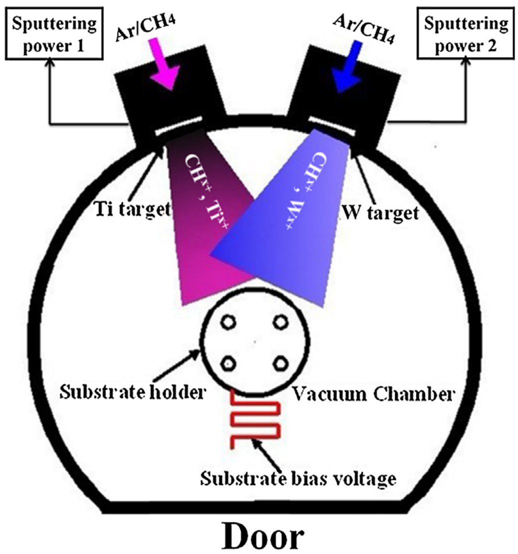

Fig. 1. The schematic diagram of the middle frequency sputtering system.

compounds, that may produce synergies between them in the film. The purpose of this work is to further improve the performance of low content Ti-doped DLC film by W incorporating, so that it can fully meet the needs of industrial applications. Although a lot of research works have been carried out about W-doped DLC films and Ti-doped DLC films, there is few works about the effect of W on the mechanical and tribological of low content Ti-doped DLC film. Therefore, mechanical properties and tribological performances of the W/Ti-doped films were investigated systematically based on the composition and microstructure analysis.

2. Experimental procedure

2.1. Films deposition

W/Ti-doped hydrogenated amorphous diamond like carbon (DLC) films were prepared on n-type silicon (1 0 0) wafers by middle frequency sputtering using Ar and CH_4 as the feedstock. Fig. 1 shows the schematic diagram of the middle frequency sputtering system for the present film deposition. The volume of the vacuum chamber was 0.8 m^3. Two rectangular targets fitted in an industrial chamber were used: titanium twin target (not shown in Fig. 1) with 100 mm × 672 mm to deposit adhesion improving interlayer; both of tungsten target and titanium target (100 mm × 672 mm) to deposit the W/Ti doped DLC film. Furthermore, it should be noted that two different sputtering powers were used with the W and Ti target in order to better control the W and Ti content. Sputtering power 1 and sputtering power 2 are supplied to the Ti target and W target, respectively. During the deposition, the Ti target current is set up a constant value by the pulse power 1 to obtain the constant Ti content (~0.3 at.%), while W target current is varied from 0 to 14 A using sputtering power 2. Before the deposition, all the substrates were ultrasonically pre-cleaned with acetone and hanging the chamber. Then, the vacuum chamber was evacuated up to 3.0×10^{-3} Pa, and 250 sccm argon was introduced into the chamber to sputter-clean by an argon discharge for 15–30 min at a bias voltage of −1000 V in order to remove surface oxide contamination, meanwhile, to clean the W/Ti target with 10 A. During the deposition, the whole process is divided into two parts: (1) a adhesion Ti interlayer was deposited on the Si substrate by sputtering the Ti target using Ar as the working gas, and the deposition time is about 20 min; (2) W/Ti film was deposited on the Ti interlayer, and the specific deposition parameters are as follows: CH_4 and Ar gas flow ratio were always kept at a fixed value of 200 sccm/90 sccm, and the sputtering power of Ti target was about 5.4 kW (~540 V, 10 A). While W content was controlled by varying the W target current from 0 to 14 A, the working pressure was about 0.7 Pa through a pressure valve. A negative pulsed bias voltage of 1000 V (the pulse length is about 7 μs and repetition rate is 90 kHz) was supplied to the substrate and the deposition time was adjusted to obtain the same film thickness in order to eliminate the influence of the film thickness. Furthermore, the low content Ti-doped DLC films with 0.3 at.% Ti content were prepared for comparison and analysis. In the further text, sometimes the terms "Ti-DLC, W/Ti-DLC1, W/Ti-DLC2, W/Ti-DLC3" were used to denote the films with 0, 2.6 at.%, 10.4 at.% and 34.5 at.% W content, respectively.

2.2. Film characterization

The elemental compositions (W and Ti content) were measured using X-ray photoelectron spectroscopy (XPS, VG ESCALAB 210 type). The thickness of the films was measured by a cross-section SEM images (Hitachi S-4800). The microstructure of the film was also analyzed by Raman spectroscopy, HRTEM and XPS. The HRTEM samples with the thickness of 50 nm were first grown on NaCl wafers, and then the NaCl wafer was dissolved into the distilled water followed by placing the film onto Cu grids. Hydrogen content has been measured by Elastic Recoil Detection Analysis (ERDA), which has been developed at the HI-13 tandem accelerator of CIAE (china institute of atomic energy). The hardness and elastic modulus of the film was evaluated by nanoindentator (Nano indenter II, MTS, US) with 40 nm indentation depth (1/10 of the film thickness). The internal stress was measured by BGS 6341 type film stress tester. The tribological behaviors of the films were carried out on the reciprocating ball-on-disk tester with a sliding velocity of 10 cm/s under a normal load of 10 N using a Al_2O_3 ball of 5 mm in diameter as the mating material, the sliding distance was 540 m with a test duration of 90 min under the air atmosphere at relative humidity (RH) of 40%. After friction testing, the total volume of wear scar was calculated from at least three measurements by a surface three-dimensional profiler, and then the specific wear rates of the films were defined as the wear volume at a unit load and in a unit sliding distance (mm^3/N m). The specific friction coefficient and wear rate were calculated by averaging the data of at least 3 individual operations. All mentioned friction coefficient and wear rate refers to the average friction coefficient and wear rate in this paper.

3. Results and discussion

3.1. SEM and HRTEM morphologies

Typical cross-sectional SEM morphologies of the as-deposited films are displays in Fig. 2. It can be seen that all films are consisted of two distinct phases corresponding to W/Ti-DLC top layer, Ti interlayer, respectively. The thickness of Ti interlayer is measured to be approximately 195 ± 15 nm, and the W/Ti-DLC top layer is 400 ± 15 nm, which is in accordance with the designed thickness. Obviously, the total thickness of all films were designed a constant value of ~600 nm in order to minimize the influence of the film thickness. Fig. 3 shows the TEM micrograph and corresponding selected area electron diffraction (SAED) pattern of the W/Ti-DLC1 film with 2.6 at.% W content and W/Ti-DLC3 film with 34.5 at.% W content. The smooth and dense granular contrasts could be observed, and the SAED pattern also presents a broad and diffuse halo without clear diffraction rings, which is a typical amorphous feature of the DLC film. Furthermore, other films also showed the same characteristics which is not shown here.

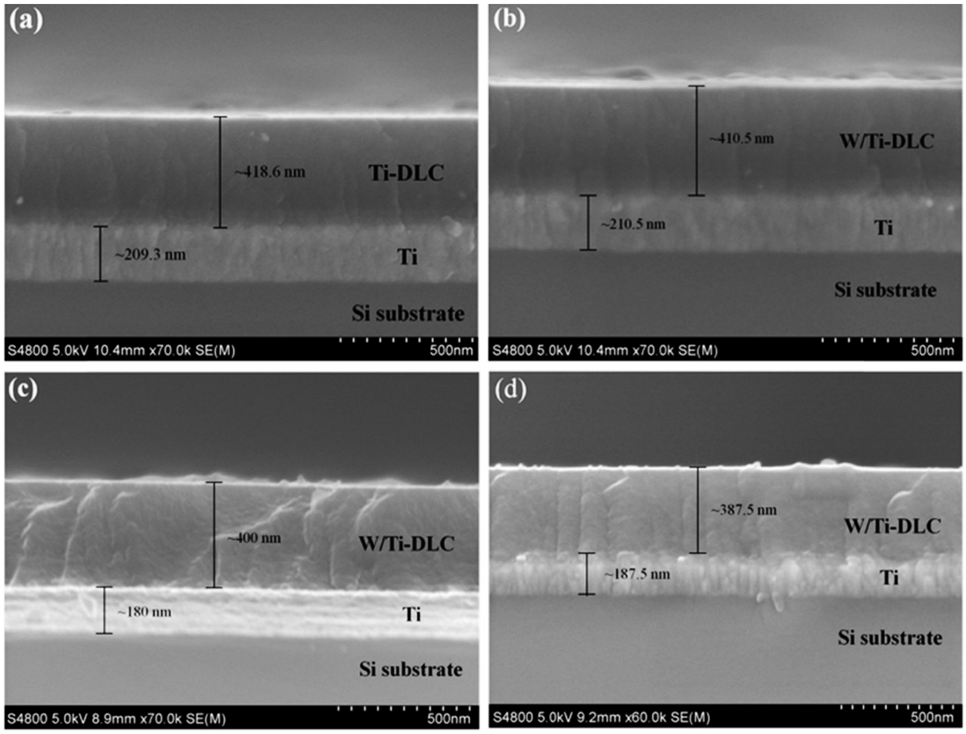

Fig. 2. Typical the cross-sectional SEM morphologies of the different films: (a) Ti-DLC film; (b) W/Ti-DLC1 film; (c) W/Ti-DLC2 film; (d) W/Ti-DLC3 film.

3.2. Raman analysis

Fig. 4 shows the Raman spectra in the wavenumber range of 800–2000 cm^{-1} for the films with the different W content. The results reveal an asymmetrical peak around 1560 cm^{-1} and a shoulder peak, which implies the typical feature of amorphous DLC films. The major Raman spectra could be deconvoluted into the two Gaussian peaks: *D* peak, due to the breathing modes of sp^2 atoms only in aromatic rings, is located at around 1380 cm^{-1}; *G* peak, attributed to the bond stretching of sp^2 atom in both aromatic rings and chains, is located at about 1550 cm^{-1} [23]. Normally, I_D/I_G ratio (the relative intensity ratio of the *D* peak and *G* peak) and the *G* peak position were used to qualitatively characterize the sp^2/sp^3 ratio, and the I_D/I_G ratio and the *G* peak position increase with the increase of sp^2/sp^3 ratio [9]. As shown in Fig. 4, the *G* peak position shifts from 1563.57 cm^{-1} to 1560.9 cm^{-1} with the increase of the W content from 0 to 2.6 at.%. As the W content increases further to 34.5 at.%, the G peak position shifts to the 1574.82 cm^{-1}. While the sp^2/sp^3 ratio decreases from 0.84 to 0.71 with the increase of the W content from 0 to 2.6 at.%, and then monotonously increases from 0.71 to 1.14 with the increase of the W content from 2.6 at.% to 34.5 at.%, which indicates the highest sp^3 content could be obtained for the film with 2.6 at.% W content.

3.3. XPS analysis

Fig. 5(a) and (b) display the Ti2p spectra of the W/Ti-DLC1 film and the W/Ti-DLC3 film, respectively. The Ti 2p spectra present two peaks at the binding energy about 458.8 eV and a shoulder peak at around 464.2 eV, which indicates Ti atoms have been successfully introduced into films. Fig. 5(c) and (e) presents a typical fitting profiles of W4f for the film with 2.6 at.% W content (W/Ti-DLC1) and 34.5 at.% W content (W/Ti-DLC3), respectively. Generally, the W4f peak could be deconvoluted into tungsten-bonding states including

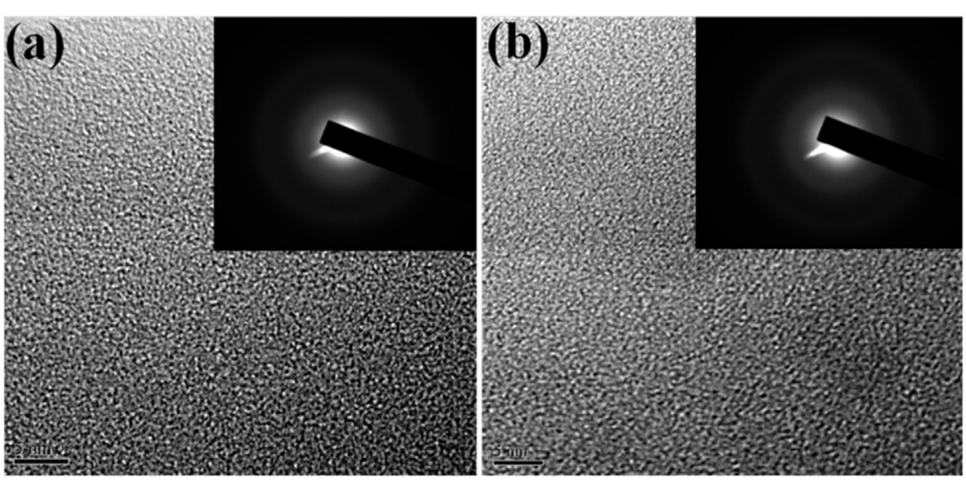

Fig. 3. The HRTEM micrograph and corresponding selected area electron diffraction (SAED) pattern of (a) the W/Ti-DLC1 film and (b) W/Ti-DLC3 film.

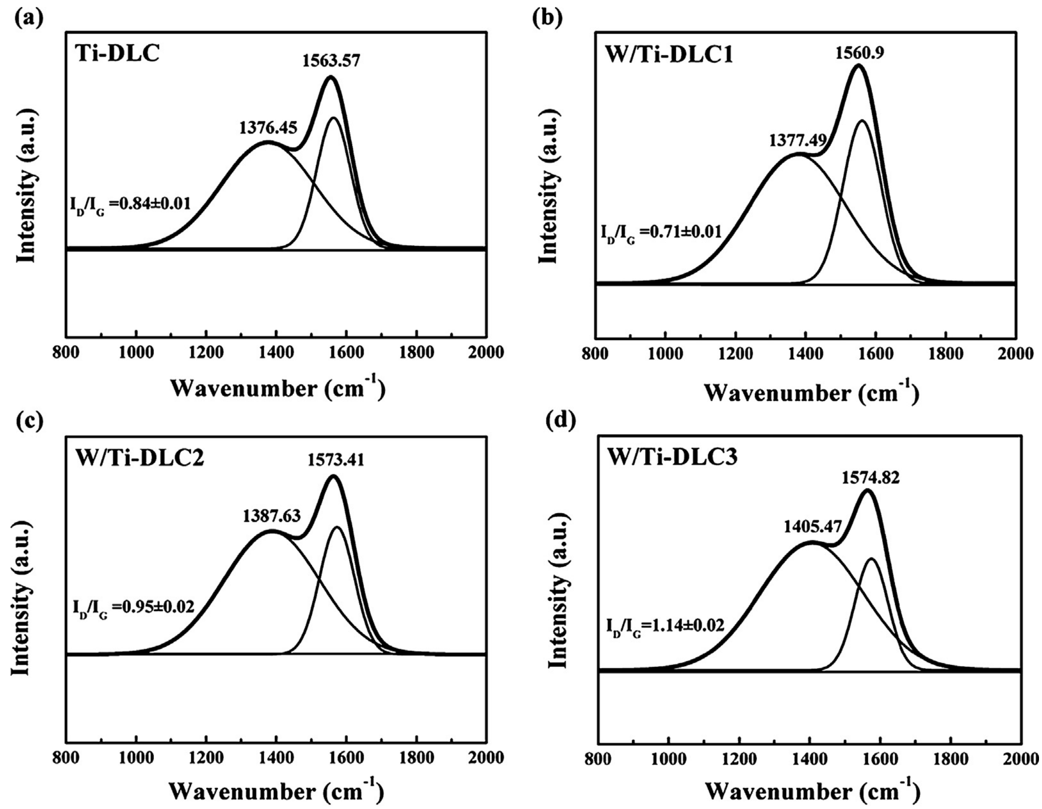

Fig. 4. Raman spectra for the films with the different W content.

W—C and W—O [24]. The whole discussion of the XPS spectra is based on the absence of oxygen in the bulk, so the W—O bond could be found. While the final level of oxygen in the chamber is uncertain, because the addition of the processing gases (argon and methane) whose purity is not specified, and desorption of oxygen from the chamber walls cannot be discarded. Thus, the existence of Oxygen atom might be attributed to the contamination, poor vacuum and other factors. The oxygen content is 2.6 at.% for the Ti-DLC film, 5.7 at.% for the W/Ti-DLC1 film, 6.2 at.% for the W/Ti-DLC2 film and 20.5 at.% for the W/Ti-DLC3 film, respectively. Fig. 5(g) shows the evolution of W4f XPS spectra from the film with the different W content. Clearly, except for W—O bonds, W—C bonds could only be found in all the films, which indirectly indicates W atom may only exist in the form of W—C states for all the films, the amount of W—C bonds mainly depends on the W content. Actually, W4f peak is unsuitable to differentiate the chemical bonds between metallic W and W—C bonds, because the binding energy of metallic W and W—C bonds is so close that it is hardly to be distinguished. Fig. 5(d) and (f) illustrates the C1s XPS spectra of the W/Ti-DLC1 film and the W/Ti-DLC3 film, respectively. A major peak located at 284.6 eV was observed, which represents the typical binding energy of the DLC film. The C1s peak could be fitted into five Gaussian peaks about 283.5 eV, 283.9 eV, 284.5 eV, 285.2 eV and 286.3 eV, corresponding to the WC, W_2C, sp^2—C, sp^3—C and C—O bonds, respectively [25]. Ti—C bonds, located at around 281.9 eV, could not be found, which means the Ti atoms incorporated into the film does not bond with the C atom. Fig. 5(h) shows the C1s spectra of films as a function of the W content. It can be seen that the C1s peak shifts toward the higher binding energy with the increase of W content from 0 to 2.6 at.%, and then shifts to the lower binding energy with the increase of W content from 2.6 at.% to 34.5 at.%, which indirectly demonstrates the highest sp^3 content could be obtained for the film with 2.6 at.% W content. Quantitatively, the sp^2/sp^3 ratio was calculated by the sp^2 peak area/sp^3 peak area ratio. Fig. 6 displays the sp^2/sp^3 ratio as a function of the W content. It is obvious that the sp^2/sp^3 ratio decreases from 1.91 to 1.73 with the increase of W content from 0 to 2.6 at.%, and then monotonously increases from 1.73 to 1.82 with the increase of W content from 2.6 at.% to 34.5 at.%, which further indicates the highest sp^3 content could be obtained for the film with 2.6 at.% W content in spite of the difference between these sp^2/sp^3 values is so small (about 2%), consistent with the Raman analysis.

3.4. *Hardness and internal stress*

The H content should be mentioned, because hydrogen is an important component about mechanical and tribological properties of the DLC film. Cavaleiro and co-workers [26] suggested that introduction of hydrogen atom is mainly led to the increasing ordering of the graphitic clusters and the formation of polymeric like features, and thereby affecting the properties of the films. In this paper, the related content is no longer too much discussion mainly based on two points: on the one hand, the measured H content has a constant range (10–15 at.%) for all films in this paper. Namely, the effect of H on the properties of the films are the same for all film; on the other hand, the effect of hydrogen on the properties of the films has a lot of the related research works [26]. The hardness and elastic modulus of films are given in Fig. 7(a). It's clear that there is an increase in the hardness at first with the increase of W content from 0 to 2.6 at.%, and then continuously decrease as the W content increased to 34.5 at.% further. First, it is well known that there is a close relationship between the hardness and the sp^2/sp^3 ratio. Combining the Raman and XPS analysis, the highest sp^3 content could be obtained for the film with 2.6 at.% W content, which finally also contributes to the highest hardness. Second, incorporated W atom in the film plays a critical role. On the one hand, the hard W carbides can form, because W atom can easily bond with the carbon atom; on the other hand, the formation of W carbides

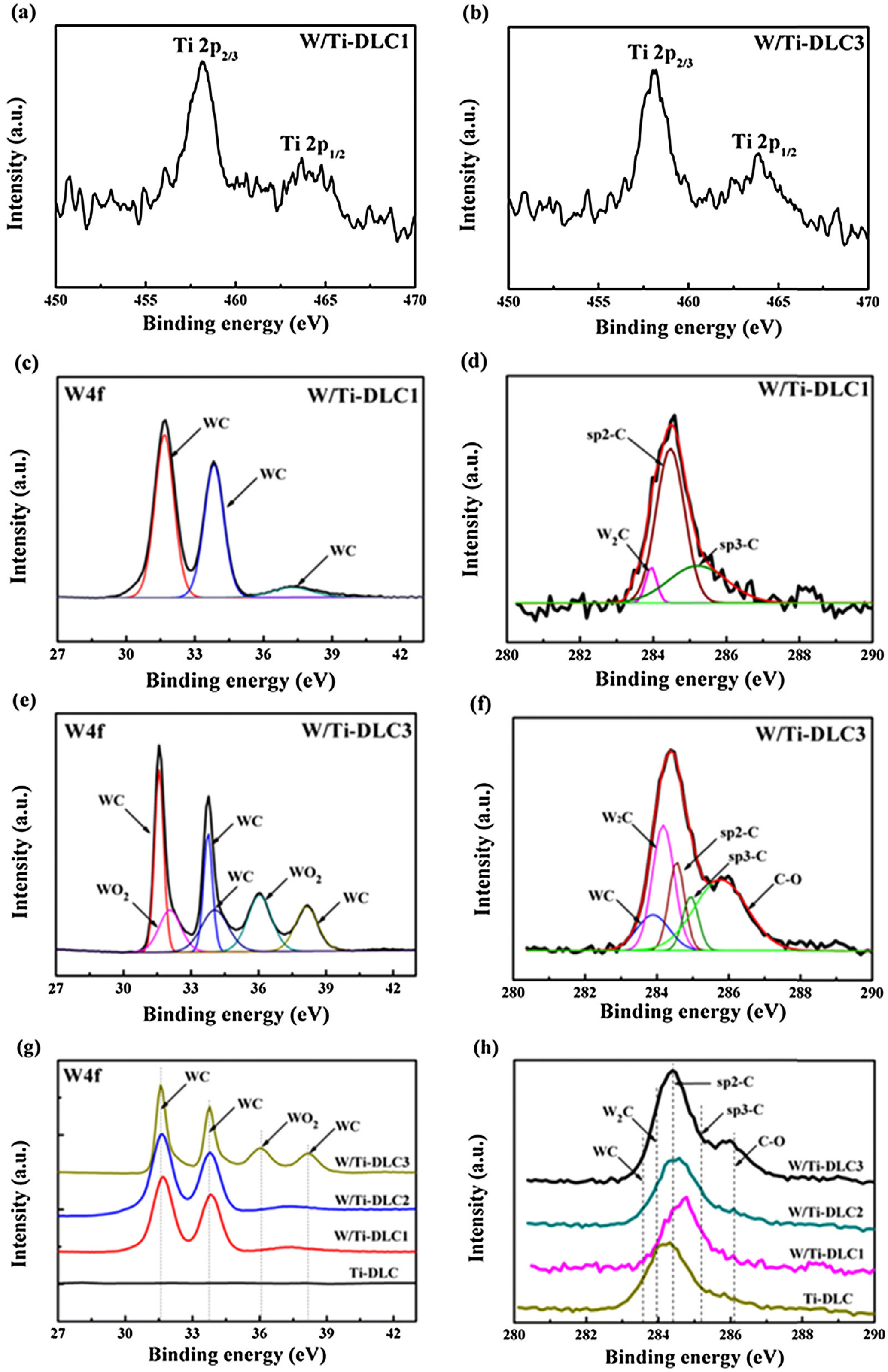

Fig. 5. The Ti2p XPS spectra of (a) the W/Ti-DLC1 film and (b) the W/Ti-DLC3 film, the W4f XPS spectra of (c) the W/Ti-DLC1 film and (e) W/Ti-DLC3 film, (g) the evolution of the W4f spectra for the films with the different W content, fitting C1s XPS peak of (d) the W/Ti-DLC1 film and (f) the W/Ti-DLC3 film, (h) the C1s spectra as a function of the W content.

can break the continuity of the carbon network. There is a certain competition between the above mentioned two processes. At the beginning, with increasing W content from 0 to 2.6 at.%, the formation of hard W carbides may be the major factor at this time, which will enhance the hardness of the films. However, as the W content increase further, a large amount of W carbides will break the continuity of the carbon network, and finally cause the hardness to decrease.

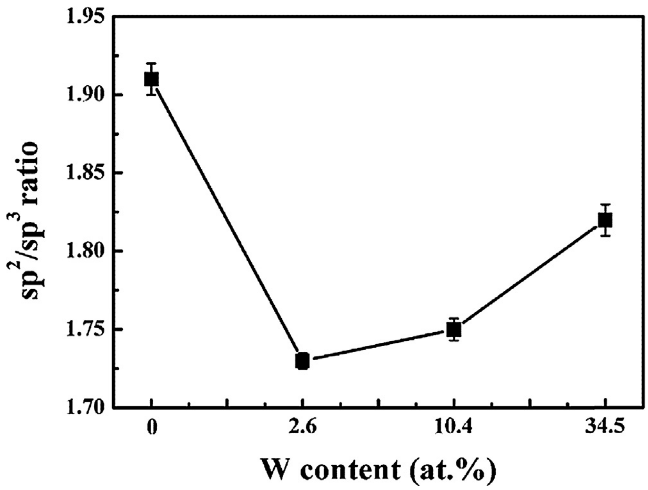

Fig. 6. The sp^2/sp^3 ratio as a function of the W content.

Wei and co-workers' [27,28] studies indicated that the interlayer had a significant impact on the stress of DLC film. Therefore, in order to minimize the influence of Ti interlayer on the film stress, the W/Ti-DLC3 films were deposited repeatedly for three times in the same condition and marked as W/Ti-DLC3-1, W/Ti-DLC3-2, W/Ti-DLC3-3, respectively. Meanwhile, the measurements of the corresponding stress are 0.90 GPa, 0.87 GPa and 0.95 GPa, respectively. Obviously, the stress of the film is almost no changes, which

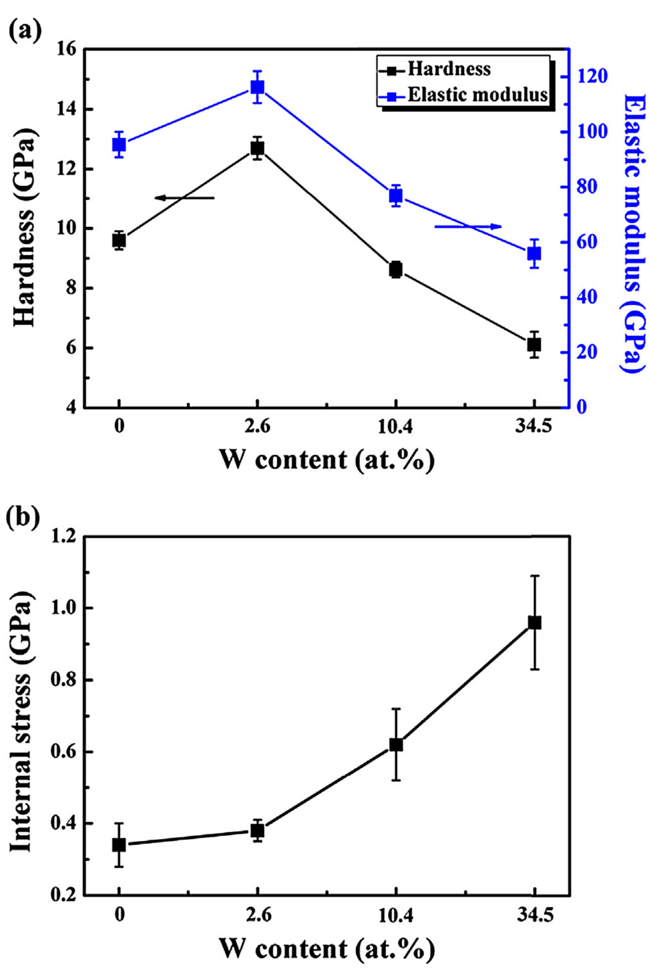

Fig. 7. (a) the hardness, elastic modulus and (b) internal stress as a function of the W content.

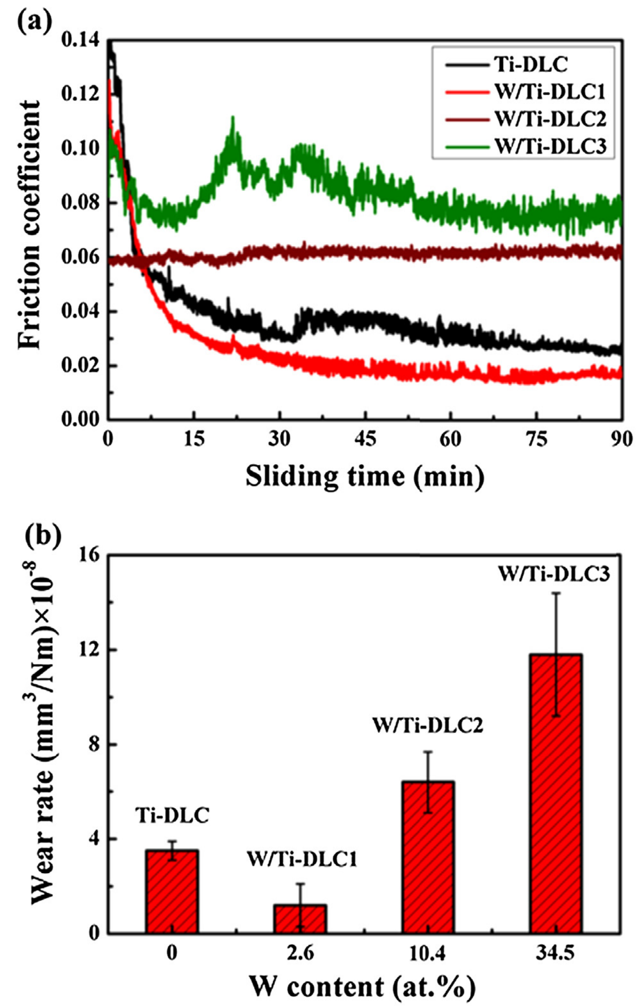

Fig. 8. (a) Friction coefficient curves as a function of the sliding time; (b) the specific wear rate of films as a function of the W content.

indicates components especially W content in this paper instead of Ti interlayer have a significantly effect on the internal stress. Fig. 7(b) presents the internal stress as a function of the W content. In the initial stage, the film internal stress is maintained at a relatively low level and showed a slight increase with the increase of W content from 0 to 2.6 at.%. As the W content increase to 34.5 at.%, the internal stress significantly increases to 0.97 GPa. Wang et al. [29] suggested that a small amount of W atom doped into the film will lead to the occurrence of the distortion of the atomic bond angles without a significant increase in the elastic energy, which eventually led to the release of the internal stress. At the same time, the increase of the sp^3 content from the Raman analysis, as the other important factor, will cause the increase of the internal stress. As a result, two counteracting factors finally lead to a relatively stable value of 0.37 GPa at 2.6 at.% W content. However, as doping of more W atoms, many W carbides were formed, because the W—C bond length is longer than the C—C bond length, which result in a significant increase in stress. Furthermore, it is worth noting that the mechanical hardness of low content Ti-doped DLC film has been further improved in the case of no increase in stress with the introduction of less W atoms.

3.5. Tribological properties

Fig. 8(a) shows the friction coefficient curves of the films as a function of the sliding time. Clearly, a low friction coefficient of

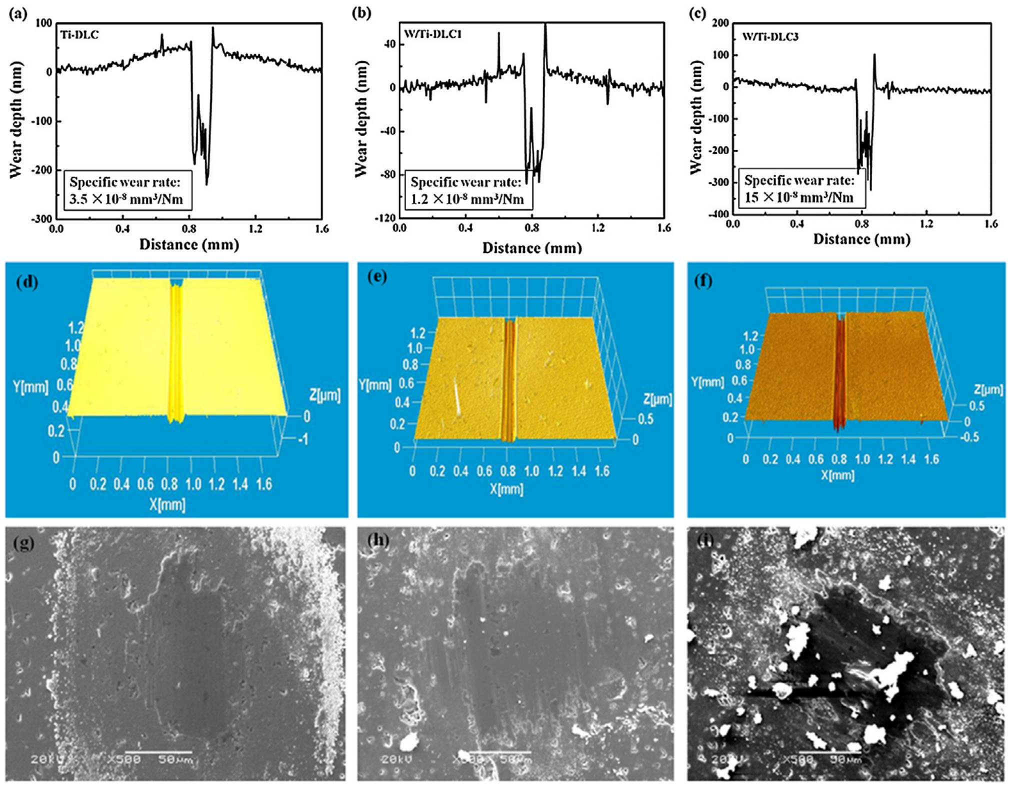

Fig. 9. The 2D cross-section surface profile (top row) and 3D scanning morphologies (mid) of wear tracks and optical micrographs (bottom) of the corresponding contact of films with the different W content.

0.036 and relatively stable friction behavior could be observed for the Ti-DLC film. With the W content increases from 0 to 2.6 at.%, the friction coefficient of the film drops obviously to 0.023. As the W content finally increases to 34.5 at.%, a high friction coefficient of about 0.084 and the severely fluctuating friction coefficient curve could be found. Fig. 8(b) presents the specific wear rate as a function of the W content. Fig. 9 shows the 2D cross-section surface profile (top row) and 3D scanning morphologies (mid) of wear tracks and optical micrographs (bottom) of the corresponding contact of films with the different W content. The film without W atom revealed a low wear depth of around 200 nm, the size of wear scar on the contact ball is about 100 μm, and a small amount of wear debris around the wear scar on the contact ball could be observed, and the specific wear rate is also relatively low (3.5×10^{-8} mm^3/N m). As the W content increases from 0 to 2.6 at.%, the wear depth sharply decreases to around 80 nm, the size of wear scar on the contact ball is almost no change and no wear debris could be found, and the specific wear rate decreases to 1.2×10^{-8} mm^3/N m. With the W content finally increases to 34.5 at.%, the wear depth monotonously increases from 80 nm to 300 nm, the width of wear tracks and wear scar on the contact ball are still almost no changes, while a large amount of wear debris around the wear scar on the contact ball could be observed, and the specific wear rate is also reaches up to 15×10^{-8} mm^3/N m. Generally, the friction and wear behaviors mainly correlate with the composition and mechanical properties of the films. At the low W content (2.6 at.%), the film exhibit the amorphous characteristic, and the W atom mainly exists in the form of less W—C phase. Therefore, the film with 2.6 at.% W content shows a lower friction coefficient and wear rate as the Ti-DLC film. While at the high W content (34.5 at.%), the formation of many W carbides have broke the continuity of the carbon network, leading to a drastically reduction in hardness, and finally causes a higher friction coefficient and wear rate. Anyway, the introduction of W atom (2.6 at.% W content) further successfully improved the mechanical and tribological performance of the low Ti-doped DLC film without increasing the internal stress.

4. Conclusions

W/Ti-doped DLC film with the different W content were prepared on the Si substrate by varying the W target current using the Ar and CH_4 as the working gas, the W atom was introduced into the low content Ti-doped DLC film in order to further improve its mechanical and tribological performance. All films with a stable thickness value of ~600 ± 15 nm showed the amorphous characteristics by the HRTEM analysis. The results from the XPS and Raman analysis indicated that the Ti contents were controlled in a stable value of ~0.3 at.% by fixing the Ti target current. While the W contents were controlled in a large changed range from 0 to 34.5 at.% by adjusting the W target current. At the low W content (2.6 at.%), the W atom mainly exists in the form of less W—C phases. At the high W content (34.5 at.%), the W atom mainly exists in the W—C and W—O phases. As a result, the hardness increased from 9.8 GPa to 12.7 GPa and the internal stress was kept at a constant value of ~0.37 GPa and almost no changes with the increase of the W content from 0 to 2.6 at.%, because the formation of less hard W—C phases can enhance the hardness, but not lead to a significant increase in stress. As the W content increases to 34.5 at.% further, the formation of a large amount of W carbides, due to break up the continuity of carbon network, cause a reduction in hardness. Meanwhile, because the bond length of W—C bond is longer than the bond length of C—C bond, which cause a significant increase in stress. Furthermore, the friction and wear behaviors mainly depend on the composition and mechanical hardness of film. Thus, the lowest friction coefficient (0.023) and wear rate (1.2×10^{-8} mm^3/N m) could be obtained for the film with 2.6 at.% W content.

Acknowledgments

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