Microstructure, erosion and tribological behaviour of thick DLC coatings by the filtered cathodic vacuum arc combined with a high-voltage pulse power

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Abstract

Hydrogen-free diamond-like carbon (DLC) films with thickness of 36 µm and 50 µm have been prepared on Si and AISI 304L stainless steel substrates, respectively, by filtered cathodic vacuum arc (FCVA) together with a high-voltage pulse power. The structure, chemical bonding state, mechanical properties and wear, corrosion resistance and anti-erosion performance were evaluated. The thick DLC films with compressive stress of 0.71 GPa and hardness of 3160 HV were obtained. The preparation of the low stress but high hardness coating mainly due to the appropriate high negative bias and short pulse duration, which could result in an alternate ion bombardment and deposition. The 50 µm-thick coating showed corrosion resistance with high polarization resistance of \(4.69 \times 10^8\) \(\Omega\) cm\(^2\) and low corrosion current density of \(5.47 \times 10^{-8}\) A cm\(^{-2}\). Thick coating exhibited outstanding sand particle erosion resistance with low average mass loss rate of 0.035 mg g\(^{-1}\). Under high-speed sand erosion conditions, the coating remains unbroken, without peeling or cracking were observed on the erosion center. In addition, DLC coatings showed good tribological performance under dry, water- and oil-lubricated condition.

1. Introduction

Diamond-like carbon films have been widely used in industry and other fields due to their outstanding properties, such as good corrosion resistance, self-lubricating property, anti-abrasion, biocompatibility, optical properties, etc [1–6]. It is well known that thick DLC films have more advantages than thin-films in longer service life and severe working circumstances. In some applications such as oil and gas production including pipes and valves, according to Liskiewicz [7], thick DLC coating are needed because of the presence of degradation mechanisms of wear and wear-corrosion. In addition, Liu et al [8] proposed that the thick DLC films can improve the service life of tubings that work in sand environment, and it can be considered as potential coatings for severe high-temperature applications [9]. However, the problems in delamination, cracks and high stress limit the thickness of the diamond-like carbon films [8, 9]. An amount of work has been done to solve those issues, such as adding doping elements [10], annealing [11, 12] and surface texturing [13]. Yatsuzuka et al [14] prepared a superthick a-C:H film with the nano-hardness of approximately 13 GPa using the plasma ion immersion deposition (PIID) technique. Lin et al [15] deposited 20 µm-thick diamond-like carbon coatings with a mean hardness of 15 GPa by deep oscillation magnetron sputtering. Liao [16] prepared a multilayer 30 µm-thick TiC-DLC film with an optimized Vickers hardness (> 1800 HV) by ion beam technique. So far, the thick DLC coatings have been prepared by doping elements, but the coating hardness is compromised (< 20 GPa). The low-hardness films cannot provide sufficient protection in some extreme environments. Therefore,
the research on thick DLC coatings with excellent hardness are indispensable. In addition, a few thick films (e.g., > 5 μm) are deposited on a brittle substrate e.g., silicon, because of its brittleness, which is prone to ductile fractures and even overall fragmentation. Brittle substrates can meet the needs of a wider range of applications and provide a promising method for depositing thick films on other substrates.

High-voltage pulse power, due to its convenient adjustment of voltage and pulse frequency which correspond to different ion bombardment energies, has a large effect on the interface characteristics and film properties [17, 18]. The influence of construction and properties of DLC films under different substrate bias voltages have been extensively studied. Negative bias voltage has a direct influence on the ion kinetic energy, which could induce implantation, collision and re-sputtering. The researchers [19–21] proposed that the pulse bias could reduce the high stress in diamond-like carbon through atomic relaxation, thereby forming hard/soft films and improving the adhesion property of the films. They also summarized that substrate temperature rise was associated with high internal stress during film growth, and the pulse power could supply the pause of pulsed ion bombardment to reduce substrate temperature. Therefore, high-voltage pulse power with low pulse frequency and pulse width can avoid excessive heating of substrate, which is a good choice to prepare thick films.

The filtered cathodic vacuum arc (FCVA) is one of the most promising methods to deposit films because of its high ionization rate and filtration of neutral atoms and macro ions, and combined with high-voltage pulse power, this method has been considered to prepare low stress and good adhesion amorphous carbon films [18].

In this article, the filtered cathodic vacuum arc with bias voltage of ~7 KV was used to fabricate a thick DLC films on Si and widely used stainless steel substrates. Peeling off and cracking are common issues in application of thick coating, in order to ensure the use stability and reliability, corrosion resistance, anti-sand erosion performance and tribological properties in different lubricated condition of thick DLC coating were evaluated. This study proposed a possibility for depositing thick and hard DLC coating by using the FCVA method together with a high-voltage pulse power and do research on its properties for wider application.

2. Experimental details

2.1. The preparation of the DLC films

The filtered cathodic vacuum arc (FCVA) system was used to deposit diamond-like carbon films on monocrystalline silicon (100) substrates and AISI 304L austenitic stainless steels (SS) samples. The substrates were mechanically ground and polished with an eventual roughness of 10 ± 2 nm. Before deposition, substrates were cleaned ultrasonically with acetone and alcohol for 15 min each. The schematic of the equipment is given elsewhere [22]. A graphite cathode with the purity of 99.999% was used to generate a carbon plasma. The chamber with 180° bent duct was pumped to 3 × 10⁻³ Pa before the experiment and a steady arc was maintained at a current of 100 A. A homemade pulsed power provides a ~7 KV bias on the substrate with pulse frequency of 50 Hz and pulse width of 5 μs. A transition layer deposited under a negative bias (~300 V—700 V), which was supplied by a different power source, was used to improve the adhesion strength between films and substrate.

2.2. The analysis method of the films

The surface morphology was obtained by a Scanning Electron Microscope (SEM, Hitachi SM4800). The thickness and curvature radius of DLC films deposited on silicon wafer were measured by a surface morphology instrument (Talysurf 5P-120). Raman spectra were acquired at a wavenumber range of 800–2000 cm⁻¹ by the Raman spectrometer (Jobin-Yvon HR800) with a laser wavelength of 532 nm. Using an x-ray photoelectron spectroscopy (VG ESCALABMK II) to analyze chemical bonding state of the DLC films. A microhardness tester (Taiming Optical Instrument Ltd.) was applied to measure hardness.

The corrosion behavior of DLC films were performed using the CS300 Electrochemical Station with the 3.5 wt% NaCl aqueous solution as an electrolyte. The potentiodynamic tests were conducted by a conventional three electrodes method, applying the DLC films as working electrode, a platinum coil as auxiliary electrode and saturated calomel (SCE) as the reference electrode. The exposed surface area of samples was 0.5 cm². Besides, the potential range was −0.8 ~ 0.8 V (versus SCE) and scan rate was 1 mV/s. The electrochemical impedance spectroscopy (EIS) measurements were acquired with a frequency range of 1 MHz–10⁻² Hz.

The friction performance was assessed by using a reciprocating friction tribometer (MFT-5000) against a 100Cr6 bearing steel with a diameter of 6.5 mm. During the tests, the sliding speed was 50 mm s⁻¹, test time is 60 min, the lengths of wear tracks were 5 mm and the normal load of 10 N. The experiments were assessed under different lubricated conditions, namely, dry, oil and water. The polyalphaolefin (PAO) base oil was used in oil lubricated condition. To ensure the reliability of the experimental data, each specimen was subjected to at least three sliding tests.
Sand erosion tests were conducted by a gas-blast erosion system which is designed following the ASTM-G76. The sharp and angular sand particles which were collected from Taklamakan with an average particle size of 150 μm were chosen for erosion tests. The sand feed rate of 2 g min$^{-1}$, particle velocity of 100 m s$^{-1}$, impingent angle of 45° and erosion time of 10 min were used for erosion testing.

3. Results and discussion

3.1. Morphology and composition structure of DLC coatings

The SEM micrographs of cross-sectional of DLC coatings deposited on Si (100) substrate and AISI 304L stainless steels (SS) substrate are given in figure 1. It shows that the thickness of DLC coating on the Si substrate and AISI 304L SS substrate are 36 μm and 50 μm, respectively. Some large particles are observed on the surface of the coating, which can be decreased by adjust the bent duct angle and other parameters, and this should be further studied.

The residual stress of thick DLC film testing is 0.71 GPa, which is much lower compare with the DLC films in other articles [1, 12, 13, 15] (table 1). In addition, the thick DLC films with low residual stress still maintain high hardness of approximately 3160 HV. For Si substrate, the silicon wafer are brittle and prone to form ductile fractures even overall fragmentation, which makes it very difficult to deposit thick films on it, especially thick and hard DLC films [23]. The preparation of such thick DLC films on Si substrate in our experiment could be explained as follows: on the one hand, the thin and hard transition layer could prevent the Si substrate from being directly influenced by the stress formed during the deposition process; on the other hand, the high negative bias and short pulse duration could result in alternate ion bombardment and deposition on the substrate.

According to the subplantation model [1], at pulse-off period, part of the ions with its own ion energy could penetrate the surface and stop in the subsurface, the increase in density lead to the formation of sp$^3$ bonding and high compressive stress in subsurface. Moreover, at pulse-on period, the ions with high energy were penetrate through surface layer and the incident process produce a lot of knock-on atoms. The energy of ion penetrate surface is only a little part, and a large portion energy dissipate as phonons. A huge energy in instantaneous time, ‘thermal spike’ would occur, which leads to the relaxation of the around atoms which cause the transform of sp$^3$ bonding to sp$^2$ bonding and relax the stress and density. Though the specific explanation of deposition process is still under debate, the appropriate ratio of ion bombardment and deposition and the value of bias voltage might be responsible for such thick films with low stress and high hardness.

![Figure 1. SEM micrographs of cross-sectional of DLC coatings on different substrates. Si (100) substrate (a), AISI 304L substrate (b).](image-url)

| Author     | Films | Stress   | Hardness |
|------------|-------|---------|----------|
| J. Robertson [1] | DLC   | 4.2–10 GPa | /        |
| J.P. Sullivan [12] | DLC   | 6.4 GPa  | /        |
| S. Kondo [13]  | DLC   | 8.5 GPa  | 36 GPa   |
| J.L. Lin [15]  | DLC   | 1.8 GPa  | 14 GPa   |

Table 1. Residual stress of DLC films in other literatures.
To explore the bonding structure of coating, the Raman and XPS spectroscopy was measured. Raman spectra of DLC films were characterized by two peaks that are fitted by Gaussian curves: D peak and G peak at about 1350 cm\(^{-1}\) and 1560 cm\(^{-1}\), respectively [24]. The fitting data are shown in figure 2(a), the result shows that G peak at 1541 cm\(^{-1}\), D peak at 1358 cm\(^{-1}\) and \(I_D/I_G\) value of DLC films is 1.57. Typical XPS spectra of DLC films are shown in figure 2(b). Prior to measurement, the surface of the films was subjected to 30 s Ar\(^+\) etching. The C1 peaks, which are used to identify chemical states of diamond-like carbon films, can be fitted by 3 peaks:

![Raman Spectrum](image1)

**Figure 2.** Typical Raman spectrum (a) and XPS C1s spectra (b) of the DLC films.

![XPS Spectra](image2)

![EIS Plots](image3)

**Figure 4.** EIS plots of AISI 304L and DLC films: (a) Nyquist plots, (b) Bode plots.

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the peak at approximately 284.6, 285.3 and 286.6 eV are marked as C=C bond, C–C bond and C–O bond, respectively [25]. According to the split peaks, the estimated sp³ ratio is 43%.

3.2. Electrochemical corrosion behaviors of DLC films

Figure 3 displays the curve of potentiodynamic polarization of thick DLC coatings and AISI 304L stainless steel (SS) in 3.5 wt% NaCl aqueous solution. Polarization resistance (R_p) and corrosion current density (I_corr) were selected to assess the anticorrosion ability. A low I_corr and high R_p value represent good anticorrosion ability of a material [26]. The measured values of corrosion voltage (E_corr), I_corr, R_p, anodic or cathodic Tafel slopes (b_a or b_c) and corrosion rate are listed in table 2. Using Stern–Geary equation 27 to calculate R_p:

\[
R_p = \frac{b_a \times b_c}{2.303 \times I_{corr}(b_a + b_c)}
\]  

Table 2. Fitting results of potentiodynamic polarization curves for AISI 304L and DLC films.

| Sample | E_corr (V) | I_corr (A·cm⁻²) | b_a (mV) | b_c (mV) | R_p (Ω·cm²) |
|--------|------------|-----------------|----------|----------|-------------|
| 304L   | −0.28      | 1.69 × 10⁻⁶     | 323.47   | 117.75   | 2.21 × 10⁷  |
| DLC    | −0.157     | 5.47 × 10⁻⁸     | 107.78   | 130.65   | 4.69 × 10⁸  |

Figure 5. The coefficient of friction (a) and wear rate (b) with different conditions.

Figure 6. The SEM images of wear tracks of thick DLC coating under different friction condition. (a)–(b) dry, (c)–(d) water-lubricated and (e)–(f) oil-lubricated condition.
The $E_{\text{corr}}$ and $I_{\text{corr}}$ of uncoated AISI 304L stainless steel substrate is $-0.28 \text{ V}$ and $1.69 \times 10^{-6} \text{ A cm}^{-2}$, respectively, as given in table 1. Nevertheless, the DLC coating displays a higher $E_{\text{corr}}$ of $-0.157 \text{ V}$ and a lower $I_{\text{corr}}$ of $5.47 \times 10^{-8} \text{ A cm}^{-2}$, which reveals that the anti-corrosion performance of AISI 304L stainless steel is prominently enhanced by DLC coating. The $p$-potential ($E_{\text{bd}}$) represents the potential at which the pitting corrosion is initiated and propagated. The $E_{\text{bd}}$ of AISI 304L substrate is 157 mV, however, the $p$-potential of thick DLC coating was not detected and exhibit a process from self-passivation to transpassivation of passive films. The oscillations of $I_{\text{corr}}$ in the passivation zone reveal that the passive films is being repaired [28]. Compared to AISI 304 L stainless steel substrate, the value of $I_{\text{corr}}$ of DLC coatings decrease by about two orders of magnitude.

The Nyquist and Bode plots of uncoated AISI 304L SS and DLC films are demonstrated in figure 4. As shown in figure 4, the diameter of capacitive arcs of DLC coatings on AISI 304L SS is larger compare with that of uncoated AISI 304L, which implies that the DLC films can significantly improve the corrosion resistance of the AISI 304L SS substrate.

3.3. Tribological properties

The coefficient of friction (COF) of the thick DLC film against 100Cr6 steel balls (HRC 61–64) under different friction condition at a load of 10 N are given in figure 5(a). The coating shows lowest average COF of 0.073 under water lubricated condition. Under oil-lubricated condition, the mean COF is about 0.091, which is lower than that under dry condition. In the steady stage of the friction test, the mean COF is 0.106 under dry friction condition. Figure 5(b) shows the wear rate of thick DLC coating under variation lubricated condition. As shown, under dry, water- and oil-lubricated condition, the wear rates of coating are $8.76, 6.06$ and $5.12 \times 10^{-8}$ mm$^3$/N·m, respectively.

Figure 6 exhibits SEM morphology of the wear tracks of the thick DLC film, the enlarged part shown in figures 6(b), (d), (f) and the EDS result of debris under dry friction condition. As shown in figure 6, the wear track width of coating under oil condition is narrowest. A lot of abrasive particles on the wear track of coating are observed under dry friction condition. The elemental composition analysis of the debris was conducted, there...
are mainly consist of Fe and C elements, together with a small quantity of O element. The generation of the amount of debris under dry friction condition is mainly attributing to its rough surface, the high contact pressure on the sharp asperities result in the particles and debris, which as abrasive cause severe abrasive wear. The wear mechanism is mainly abrasive wear. Due to the water or an oil lubricated film exist between the partial solid–solid contact faces [29, 30], the particles are less on the wear track, part of them are dissipate in lubricated solution and other debris are between the worn asperities under oil- and water-lubricated. Schematic illustrations of the wear mechanism of DLC samples under different lubrication condition show in figure 7, the contact is solid–solid contact faces. But the water and oil lubrication condition, due to the existence of the lubrication layer decrease the direct contact between ball and coating, and the debris could accumulation on the gap of asperities in solution and together form lubrication layer, decrease the abrasive wear. The high wear rate of thick DLC under dry friction condition mainly due to the asperities on the surface, which should be removed for further application. These results suggest that the thick DLC films are potential protective surface materials under dry, water and oil lubrication conditions.

3.4. Anti-sand erosion performance
As given in figure 8(a), the dimension of the polygonal sand particles are distributes from 100 to 200 μm. The average mass loss rate are used to evaluate the anti-sand erosion performance of materials. Figure 8(b) exhibit the average mass loss rates of stainless steel substrate and the thick DLC coating. The substrate showed much poor sand resistance with larger average mass loss rates of 1.37 mg g⁻¹. The average mass loss rates of thick DLC coating of 0.035 mg g⁻¹ is far smaller than that of bare stainless steel substrate. Accordingly, the 50 μm-thick DLC coating on stainless steel can prominently improve its anti-sand erosion performance.

Figure 9 shows the SEM images of the eroded surface of the AISI 304L stainless steel substrate and 50 μm-thick DLC coating. As shown in figure 6, the ploughing action and erosion pit can be observed on the erosion surface of substrate. The erosion surface of thick DLC coating shows squashed asperities and some sand erosion lead to some defects. The coating remain whole, no obviously crack and delamination occur on the erosion surface. The damage mainly caused by the defects on the surface of coating. Research has proved that excessive residual stress was one of the main factors of ceramic films leading to fracture and delamination, decreasing the sand particle erosion resistance [32, 33]. Thereby, the low stress and high hardness are mainly responsible for the great anti-sand erosion performance. The results indicating that the 50 μm-thick DLC coating shows outstanding sand erosion resistance, the thick DLC coating is a potential coatings for severe sandy environment applications in sand environment.
4. Conclusion

Thick DLC coating are potential in longer service life and severe working circumstances. DLC films with thickness of 36 and 50 μm are successfully prepared on Si and AISI 304L SS substrates by filtered cathodic vacuum arc combined with high-voltage pulse power. The thick DLC film with high hardness of 3160 HV and low internal stress of 0.71 GPa is achieved. The 50 μm-thick coatings show low coefficient of friction of 0.112, 0.073, 0.091 and wear rate of 8.76, 6.06, 5.12 × 10⁻⁶ mm³/N·m under dry, water- and oil-lubricated condition, respectively. The wear mechanism is mainly abrasive wear under dry friction condition. Under high speed sand erosion, thick DLC coating shows small average mass loss rates 0.035 mg g⁻¹, which is far smaller than that of bare stainless steel substrate. Moreover, coating still whole, without fracture and delamination are observed on the erosion surface after erosion test. The results suggest that filtered cathodic vacuum arc method combined with high-voltage pulse power can be an effective strategy to fabricate thick DLC coating with great properties, and the coating is promising used in severe working circumstances.

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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).

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