Structural, mechanical and frictional properties of tetrahedral amorphous carbon film by filtered cathodic vacuum arc system

Xiang Yu a,⁎, Xu Zhang b, Cheng-biao Wang a, Fu-tian Liu c, Zhi-qiang Fu a

a School of Engineering and Technology, China University of Geosciences, Beijing 100083, China
b Key Laboratory of Beam Technology and Material Modification of Education, Institute of Low Energy Nuclear Physics, Beijing Normal University, Beijing 100875, China
c School of Material Science and Engineering, Jinan University, Jinan 250022, China

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Abstract

Tetrahedral amorphous carbon (ta-C) film was deposited on silicon coupons using a Filtered Cathodic Vacuum Arc system with a pre-implantation process, and then was investigated by some systematic analyses. The ta-C film shows a very smooth surface morphology and a dense cross-section texture, along with a gradient interlayer, about 0.5 μm between the substrate and ta-C film, also has a board distribution of asymmetric Raman intensity in the range of 1200–1700 cm⁻¹, centered at 1567 cm⁻¹. Microhardness and elastic modulus of ta-C film are 47.74 GPa and 298.46 GPa, respectively. Ta-C film also possesses a good adhesion, critical load (Lc) of 42 mN. Moreover, the ta-C film owns a low friction coefficient of about 0.15 in a long duration.

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1. Introduction

Tetrahedral amorphous carbon film (ta-C film) is a hydrogen-free carbon film with the remarkable properties comparable with those of diamond film, such as high hardness, optical transparency and chemical inertness [1,2]. Moreover, ta-C film can be synthesized through a relatively convenient method and has a much smoother surface, making the tribological performances of ta-C film better than those of the diamond film [3]. Among the various attempts used to prepare ta-C films, Filtered Cathodic Vacuum Arc (FCVA) system is advisable, and the performable properties make ta-C films suitable for wear-resistant applications e.g. precise movable component [4,5].

One key disadvantage of ta-C film, limiting the industrial applications, is the poor adhesion between the film and the substrate due to its high internal stress and poor consistency to the substrate. Many approaches have been used to improve the adhesion [6,7], e.g., sputter cleaning of the substrate surface, applying an interlayer or a transitional layer. Implanting the carbon ions before the deposition may benefit in-situ forming an interlayer and then improve the adhesion of ta-C film. So this paper focuses on investigating the microstructure and properties of ta-C film with a pre-implanted SiC interlayer.

2. Experimental details

The substrate employed some silicon coupons in a size of ∅45 mm × 5 mm, pre-polished to a surface roughness of Ra=0.1 μm, and then was cleaned in an ultrasonic bath and dried in an infrared light. MEVVA source and S-bend filter connecting two 90° bend together (whose schematic configuration is shown in Fig. 1) were used to produce carbon plasma from a graphite cathode target with a diameter of 10 mm and a purity of 99.99%. Pulsed mode with a pulsed length of 25 ms was employed in the arc source operation, and pulsed frequency 25 pulses/s. MEVVA source was operated at pulsed current of 100 A. The unwanted neutral and macro-particles were filtered through the S-bend filter. The base pressure was 2×10⁻⁵ Pa. Before deposition, the carbon ion was pre-implanted at 10 kV to a dose of 1×10¹⁷/cm² into the substrate to get a SiC interlayer.
Scanning Electron Microscope (SEM) equipped with energy-dispersive X-ray (EDX) analyzer was utilized to analyze the surface and cross-section morphologies of the ta-C film; Laser Raman Spectroscope was employed to investigate the bonding structure; Nano-indenter was adopted to measure the microhardness and elastic modulus; Micro-scratch tester was utilized to analyze the adhesion between ta-C film and Si coupons; Ball-on-disc tester was employed to investigate the frictional performance, under a load of 5 N (employing Si$_3$N$_4$ ball) and at a constant linear speed about 0.65 m/s.

3. Results and discussion

Fig. 2 shows the morphology comparison between ta-C film and amorphous carbon (a-C) film, deposited using the similar parameters to ta-C film by cathodic vacuum arc technique. Under the same enlargement, the a-C film, shown in Fig. 2a, has a relative rough surface with many arc-induced macro-particles in various sizes of micron-scale, whereas the ta-C film, shown in Fig. 2b, exhibits a very smooth surface without the visible macro-particles and other surface faults. Such optimal morphology of ta-C film is contributed to the effective filtrated effect of FCVA system.

Fig. 3 shows a cross-sectional image along with element distributions of ta-C film. Under the same enlargement, the a-C film, shown in Fig. 2a, has a relative rough surface with many arc-induced macro-particles in various sizes of micron-scale, whereas the ta-C film, shown in Fig. 2b, exhibits a very smooth surface without the visible macro-particles and other surface faults. Such optimal morphology of ta-C film is contributed to the effective filtrated effect of FCVA system.

Fig. 4 shows the comparison of Raman spectra of a-C film and ta-C film. Referring to C and Si elemental line distribution, the thickness of the ta-C film is about 0.3 μm, and a gradual interlayer is about 0.5 μm thick. In addition, C and Si elements...
exhibit a gradient change in the interlayer. The richness of C element shows a decreasing tendency from ta-C film to Si substrate inside, even exhibiting a certain penetration in Si substrate. Such C element penetration and gradient interlayer formation are favorable for improving the interfacial properties between ta-C film and Si substrate [10].

Fig. 4 shows Raman spectra of the ta-C film and a-C film. Comparing with a-C film spectrum in Fig. 4, ta-C film has one broad distribution of asymmetric Raman intensity in the range of 1200–1700 cm$^{-1}$, centered at 1567 cm$^{-1}$. This spectrogram can be fitted to two Gaussian peaks, one at approximately 1580 cm$^{-1}$ (“G” peak) and another at about 1360 cm$^{-1}$ (“D” peak). Some researchers have reported that the intensity ratio of these two peaks ($I_D/I_G$) can be used to measure the relative sp$^3$ content of DLC films [11]. The sp$^3$ content is about 75% in ta-C film.

Using the mathematical mean of three results as the measurement value, Fig. 5 shows microhardness and adhesion of ta-C film. In nano-indentation testing, the continuous measurement was carried out by operating the indenter at a constant displacement penetration depth of 50 nm. As shown in Fig. 5a, the hardness and elastic modulus of this ta-C film are 47.74 GPa and 298.46 GPa, respectively. Comparing with relatively low hardness of Si substrate (7 GPa), the hardness of ta-C film is much higher, and thus the hardness of Si substrate has been developed in evidence under action of ta-C film. Fig. 5b is a curve of scratch distance vs. penetration depth. After finding the critical change point of friction coefficient for C point in Fig. 5(b), the critical adhesion force (Lc) of ta-C film approaches 42 mN. Such good adhesion is very important to retain the film properties stable in a long service duration [12].

The ball-on-disc tester was employed to investigate the frictional performance and durability of ta-C film. As shown in Fig. 6, COF (coefficient of friction) of ta-C film possesses a low COF value of about 0.15, almost one sixth of that of Si wafer (about 0.82), which shows that ta-C film is a good friction-reducing coating. The low COF value is attributed to both the smooth surface and the mechanical properties of ta-C film. In addition, the COF of ta-C film is stable up to 12,000 revolutions without failure under the load of 5 N.

4. Summary

A ta-C film with a gradient interlayer was deposited on the silicon substrate using a FCVA system. The ta-C film possessed some good combined properties: a very smooth surface morphology and a dense cross-section texture, a gradual interlayer with thickness of about 0.5 μm, a high sp$^3$ content of 75%, a high microhardness of 47.74 GPa, a good adhesion of Lc 42 mN and a low friction coefficient of 0.15 kept in a long and stable duration of 12,000 revolutions.

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