



Microstructure and tribological performance of self-lubricating diamond/tetrahedral amorphous carbon composite film

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ABSTRACT

In order to smooth the rough surface and further improve the wear-resistance of coarse chemical vapor deposition diamond films, diamond/tetrahedral amorphous carbon composite films were synthesized by a two-step preparation technique including hot-filament chemical vapor deposition for polycrystalline diamond (PCD) and subsequent filtered cathodic vacuum arc growth for tetrahedral amorphous carbon (ta-C). The microstructure and tribological performance of the composite films were investigated by means of various characterization techniques. The results indicated that the composite films consisted of a thick well-grained diamond base layer with a thickness up to 150 μm and a thin covering ta-C layer with a thickness of about 0.3 μm , and sp^3 -C fraction up to 73.93%. Deposition of a smooth ta-C film on coarse polycrystalline diamond films was proved to be an effective tool to lower the surface roughness of the polycrystalline diamond film. The wear-resistance of the diamond film was also enhanced by the self-lubricating effect of the covering ta-C film due to graphitic phase transformation. Under dry pin-on-disk wear test against Si_3N_4 ball, the friction coefficients of the composite films were much lower than that of the single PCD film. An extremely low friction coefficient (~ 0.05) was achieved for the PCD/ta-C composite film. Moreover, the addition of Ti interlayer between the ta-C and the PCD layers can further reduce the surface roughness of the composite film. The main wear mechanism of the composite films was abrasive wear.

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1. Introduction

The unique properties of diamond are promising for extensive applications in many fields of industry. In particular, in cutting tool protection [1–3], diamond films have been used as anti-abrasion components because of their high hardness, excellent wear resistance and chemical inertness. However, traditional chemical vapor deposition (CVD) polycrystalline diamond (PCD) films usually have coarse surface (surface roughness R_a up to a few microns), with crystallinity, crystal size and grain orientation varied with deposition parameters [4]. High surface roughness will lead to higher friction coefficient and wear rates, deteriorating the tribological performance of diamond films. It is then of great interest to smooth the surface and further enhance the wear resistance property of diamond films.

In recent years, various methods for smoothing the surface of diamond films have been developed. Nanocrystalline diamond (NCD) film [5–7], diamond polishing [8], activated hydrogen etching on surface [9], substrate pretreatment [10], addition of

interlayers [11], growth parameters control [12], alternate deposition of poly/nanocrystalline diamond multilayer [13–15], and solid lubrication composite coating on diamond films [16], have been proved to be effective tools to lower the surface roughness and improve the tribological behavior of the diamond film.

Diamond-like carbon (DLC) films [17], a group of metastable amorphous carbon films rich in sp^3 bonds, have also shown excellent mechanical properties and tribological performance (low friction coefficient ~ 0.1). Especially tetrahedral amorphous carbon (ta-C), possessing high sp^3 -bond carbon fraction (up to 90%) and smooth surface ($R_a \sim 0.1 \text{ nm}$) [18], has extremely low friction coefficient (lower to 0.02) and wear rate ($\sim 10^{-9} \text{ mm}^3/\text{Nm}$) [19]. During friction and wear process, the ta-C film transforms by stress-induced transformation to graphitic over-layer, which can act as a solid wear-reducing lubricant [19]. In dry solid–solid friction, Nosonovsky and Bhushan [20] have reviewed that various types of inhomogeneities between the contacting bodies, including surface roughness, material deformation and contamination, lead to friction. Surface roughness is one of the major factors in determining frictional behavior. Every nominally flat surface is not ideally smooth and has roughness due to small asperities. A contact between the two bodies during friction occurs only at the summits of the asperities. The friction coefficient is not only a material property but also an indication of contact mechanics between surfaces

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Table 1

The deposition parameters of two-step procedure for diamond/ta-C composite films.

HFCVD (first step for diamond deposition)		FCVA (second step for ta-C deposition)	
Parameters		Parameters	
CH ₄ /H ₂ flow (sccm)	3/150	Graphite cathode purity	99.99%
Reaction pressure (kPa)	5.5	Working pressure (Pa)	4.0×10^{-4}
Deposition temperature (K)	1073	Arc current (A)	55
Filament temperature (K)	2273 $\pm$ 50	Substrate temperature (K)	300
Bias current (A)	3.5	Bias voltage (V)	–100
Duration (h)	78	Deposition time (min)	60

with roughness. Thus, a very smooth surface is highly desirable to achieve excellent friction performance for most tribological applications. From this point of view, deposition of a lubricating ta-C layer (maintaining high hardness but with very smooth surface comparing to CVD diamond film) onto rough CVD polycrystalline diamond layer to form diamond/ta-C composite film may be an effective technique to lower the surface roughness of diamond film and achieve exceptional surface friction performance.

In the present work, a new set of self-lubricating diamond/ta-C composite films were prepared by a two-step procedure including first deposition of rough polycrystalline diamond layer by hot-filament chemical vapor deposition (HFCVD) and then tetrahedral amorphous carbon layer by filtered cathodic vacuum arc (FCVA). The microstructure and tribological performance of the as-deposited diamond/ta-C composite films were investigated.

2. Experimental details

2.1. Material preparation

Commercially available silicon carbide (SiC) wafers were selected as substrates. The SiC wafers were received with a mirror-like polished surface ($R_a = 0.025 \mu\text{m}$) and then were machined to the dimension of $20 \text{ mm} \times 20 \text{ mm} \times 9 \text{ mm}$ by laser cutting. The specimens were dipped in HF acid solution for 5 min to dissolve the oxides on the surfaces, and ultrasonically cleaned in acetone for 10 min to remove the surface contamination, followed by ultrasonically bathing in de-ionized water and drying by flowing air before deposition. The diamond/ta-C composite films were synthesized by a two-step procedure. Firstly, the polycrystalline diamond films were deposited in a HFCVD chamber. A gas mixture of methane and hydrogen was activated by four tungsten filaments (0.8 mm in diameter and 7.5 mm apart from each other) arranged in a 6 mm distance from the substrate. Then, the diamond-coated SiC specimens were enclosed in FCVA chamber for ta-C deposition. The carbon plasma was produced from the arc spot on a graphite cathode of 99.99% purity. The resulting plasma beam was then passed through an off-plane 90° bend solenoid to filter out particulates and other neutral species. To further lower the surface roughness of the diamond film, a $0.4 \mu\text{m}$ thick metal Ti interlayer was deposited onto the diamond surface prior to ta-C deposition. Thus, three groups of diamond films including single PCD diamond film, PCD/ta-C composite film, PCD/Ti/ta-C composite film were synthesized for comparison. The details of growth parameters were summarized in Table 1.

2.2. Sample characterization

The surface roughnesses of films were measured by a three dimensional white-light interfering surface profiler (Micro XAM-3D). FEI Quanta 200 FEG field emission scanning electron microscope (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS) was used to investigate the microstructure and elemental composition of the as-deposited diamond films. Raman

measurements were performed with a Renishaw 2000 Raman spectrometer using a 514.5 nm line of Ar⁺ laser as the excitation wavelength. The X-ray diffraction (XRD) patterns were recorded by a Philips PW 1710 automated diffractometer, using glancing angle technique with an incidence angle of 2° . The carbon bonding of the diamond film was investigated by X-ray photoelectron spectroscopy (XPS, PHI Quantera SXM), using a single Al-K α X-ray source, operated at 250 W. A ball-on-disk tester (MS-T3000) was used to evaluate the tribological properties of the samples. The coated SiC specimens were fixed on a rotary platform with a sliding velocity of 0.125 m/s under a normal load of 5 N using a Si₃N₄ ball of 4 mm in diameter as the counterpart. The produced initial Hertzian contact pressure under this applied normal load was about 2.5 GPa. The duration was 8 h, equating to a sliding distance of 3600 m. The relative humidity during testing was $43 \pm 2\%$, and the temperature was $25 \pm 2^\circ\text{C}$. After the wear test, SEM observation and Raman measurements were carried out on the wear track to study the friction behavior and wear mechanism of the diamond films.

3. Results and discussion

3.1. Surface and fracture microstructure

The SEM surface morphologies of the three as-prepared samples are shown in Fig. 1 under different magnifications. For the single PCD film shown in Fig. 1a, it can be observed that an almost continuous coarse polycrystalline diamond film was obtained by HFCVD method. The crystal facets of the diamond film mainly exhibit (1 1 1) and (2 2 0) preferential orientation, with grain size in the range of 20–35 μm . The well-faceted diamond crystallites are surrounded by tiny ball-like diamond grains. These small-sized diamond crystallites may be formed due to the second re-nucleation during deposition. Moreover, the inset micrograph at high magnification clearly shows that some deep grooves existed between the well-faceted diamond crystallites, resulting in a relatively rough surface. It was measured by white-light interfering surface profiler that the surface roughness of the PCD film was up to $1.55 \mu\text{m}$. In comparison with PCD film, the PCD/ta-C composite film shown in Fig. 1b presents somewhat smoother surface. The diamond crystal facets and inter-crystal grooves are covered by the subsequently deposited ta-C film by FCVA, obtaining a surface roughness of $1.04 \mu\text{m}$. Especially for the PCD/Ti/ta-C composite film shown in Fig. 1c, the addition of Ti interlayer before ta-C deposition further smoothes the surface. The grooves are nearly filled in by Ti/ta-C composite layers as shown in the inset. The surface roughness R_a decreased to $0.73 \mu\text{m}$. Therefore deposition of covering material with superior smoothness (for example, ta-C, nanocrystalline diamond, and so on) onto rough polycrystalline diamond to form composite film is an effective tool to lower the surface roughness.

The SEM image of the cross-sectional fracture of typical PCD/Ti/ta-C composite film is presented in Fig. 2. It is clearly observed that the interface between SiC substrate and diamond film is relatively sharp and smooth. The polycrystalline diamond

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