



Microstructure changes of self-mated fullerene-like hydrogenated carbon films from low friction to super-low friction with the increasing normal load

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ABSTRACT

Self-mated fullerene-like hydrogenated carbon films (FL-C:H films) realized the conversion from low friction (~ 0.012) to super-low friction (~ 0.006) state due to the increasing normal load.

The structure changes in the conversion and the effect of environments (N_2 and humid air) on the structure changes were discussed by the combination of SEM, TEM, Raman and XPS spectra. With the increasing normal load, smaller wear scar area (or contact area) and thicker tribofilm could be observed. At smaller scale (such as nanoscale), not only more perfect graphitic structure can be formed, but newly formed longer graphitic sheets also tended to curve.

1. Introduction

Driven by the discovery of nanocarbons such as fullerenes, graphene and nanodiamonds, scientists are trying to introduce nanocarbon clusters into diamond-like carbon (DLC) films to significantly improve macro properties, such as graphene [1,2], nanodiamond [3,4], fullerene-like (FL) [5–10] and graphite-like nanostructures [10,11]. Hydrogenated DLC films with FL nanostructures (FL-C:H) always exhibit ultra-low friction and wear in humid air (Relative humidity $\leq 35\%$) under high contact pressures [12,13]. In general, FL-C:H films have been often deposited on one surface at the sliding interface, and their frictional behaviors have been discussed with the choice of counterface materials and environments [14,15]. Recently, we have realized the deposition of FL-C:H films on steel substrates by adding plasma nitriding pre-process in the plasma enhanced chemical vapor deposition (PECVD) which can form the films. In the literatures, we have found that self-mated FL-C:H films can attain super-low friction at the contact pressures lower than previous reported values. Later, we have answered how self-mated sp^2 -rich carbon films (namely, FLC and graphite-like carbon) evolve in N_2 gas with the increasing sliding cycles and normal load. However, the tribological behaviors of DLC films are not only sensitive to working conditions (sliding speed and normal load) but also depend on and environments [16,17]. Here, the frictional behaviors and structure evolution of self-mated FL-C:H films were discussed in humid air from low to super-low friction due to increasing normal load.

The films' friction coefficients decreased from ~ 0.012 , to 0.009, to 0.007, to 0.006, when the normal loads increased from 8, to 10, to 12, to 14 N. With the conversion from low to super-low friction state, smaller wear scar area (or contact area) and thicker tribofilms were observed. With the combination of TEM, Raman and XPS spectra, the conversion should originate from the formation of more perfect graphitic structure on the worn surfaces at smaller scale (such as nanoscale), and especially the curvature of newly formed longer graphitic sheets at higher load. The effects of environments (N_2 and humid air) on the structure changes under super-low contacts were also discussed together with the study [10]. The self-mated method confirms the potential of FL-C:H films which can realize super-low friction at lower contact pressures in N_2 gas and humid air. This will widen the scope of realizing super-low friction and promote the development and application of super-low friction carbon films, which is helpful to reduce energy dissipation and material loss in moving mechanical systems due to friction.

2. Experimental details

2.1. Materials

In this study, FL-C:H films were deposited on Si (100) substrates and steel balls (diameter 3 mm) by using PECVD (Fig. 1). The diameters of circular upper electrode and sample holder were 300 and 200 mm,

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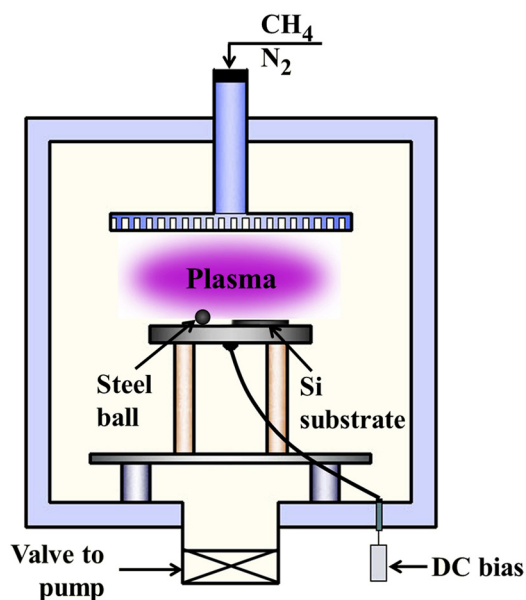


Fig. 1. Schematic diagram of depositing PECVD.

respectively. The distance between them was set as 50 mm. Prior to the film deposition, these substrates and balls were ultrasonically cleaned in the mixed solution of acetone and alcohol for about 15 min and dried in N_2 gas. After the samples were put into the deposition chamber, the chamber was pumped down to 10^{-4} Pa. All the substrates and balls were treated with Ar^+ in order to remove the surface native oxides ((1) gas flow rate: 320 SCCM; (2) gas pressure: 5 Pa; (3) pulsed frequency: 80 kHz; (4) duty cycle: 0.8; (5) bias voltage: -1000 V; (6) deposition time: 30 min). Later, the in-situ nitriding process was used with nitrogen gas as the feedstock. The process was supplied by a negative bias of 1200 V ((1) gas flow rate: 90 SCCM; (2) gas pressure: 55 Pa; (3) pulsed frequency: 80 kHz; (4) duty cycle: 0.8; (5) depositing time: 1.5 h). The process could form a FeN intermediate layer to enhance the interaction of FL-C:H films to underlying steel balls. Finally, FL-C:H films with the thickness of about 500 nm were deposited at a negative bias of 1100 V using methane as the feedstock. They had the deposition conditions: (1) gas flow rate: 15 SCCM; (2) gas pressure: 20 Pa; (3) depositing time: 2.5 h.

2.2. Characterization methods

The friction tests of self-mated FL-C:H films (on Si substrates and steel balls) in Fig. 2 were performed by a UMT-3 tribometer (UMT-3,

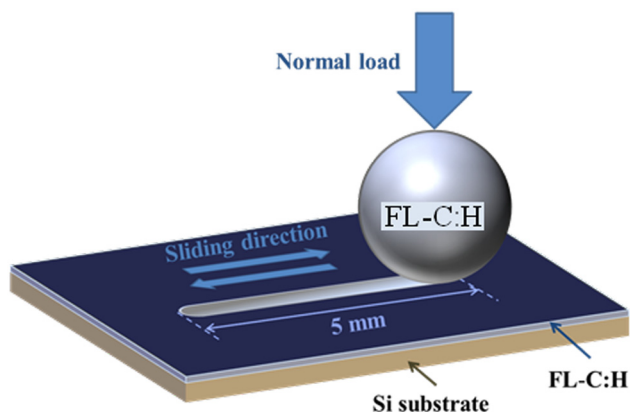


Fig. 2. The schematic diagram of UMT-3 tribometer in the reciprocating sliding (ball-on-disk) mode.

Bruker-CETR, USA) in open air (relative humidity of 30%) under different normal loads (8 N, 10 N, 12 N and 14 N). These loads created initial maximum contact pressures ~ 0.71 , to 0.88, to 1.06, to 1.24 GPa according to the equation. The amplitude and frequency were set as 5 mm and 10 Hz with the sliding speed of 10 cm/s, respectively. At the beginning of each test, the zero calibration of the machine was performed automatically. The tests were repeatedly carried out three times. After the friction experiments, the structures of the films and their wear debris were obtained by a transmission electron microscope (FEI Tecnai F30, USA) with 0.14 nm resolution using an accelerating voltage of 300 kV. The TEM samples of the wear debris were prepared by floating off the worn surfaces in distilled water and dipping the debris on holey Cu grids. The chemical states of the worn surfaces were revealed by a micro-Raman spectrometer (532 nm, Jobin-Yvon HR-800, Horiba/Jobin Yvon, Longjumeau, France) and a x-ray photoelectron spectroscope (XPS, PHI-5702, Physical Electronics Inc., USA) operating with Al-K α radiation and the chamber pressure below 10^{-6} Pa. The worn surfaces' morphologies were characterized with a field emission scanning electron microscopy (FESEM, TESCAN MIRA3 XM, Czech) at 7 kV and a 3D surface profilometry (ZYGO Nexview, USA).

3. Results and discussion

3.1. Microstructure changes of FL-C:H films in humid air

FL-C:H films were prepared on Si substrates by PECVD and the other films were deposited on steel balls ($\Phi 3$) by combining the PECVD process with plasma nitriding. Fig. 3 shows the TEM, Raman and FTIR results of the films. The films contain many curved and cross-linked graphite layers with the layer spacing of about 0.35 nm, which is in good agreement with that of the graphite face (0002) [18]. The Raman spectrum was fitted by symmetric multipeak methods [10,19] involving four peaks at ~ 1200 , ~ 1360 , ~ 1470 and ~ 1560 cm^{-1} . As shown in Fig. 3, a much better fit quality can be obtained. The peak at 1200 cm^{-1} originates from heptagons distributed randomly throughout a hexagonal network, and the 1470 cm^{-1} peak comes from pentagons in the network, respectively [10,20]. From the Fig. 3, a strong signature from the odd rings is observed, which induces the curvature of graphite sheets to form FL structure. FTIR spectrum shows a strong absorption peak centered at ~ 1600 cm^{-1} , apart from the two weak C–H absorption peaks at ~ 1450 and ~ 2950 cm^{-1} . For carbon, the ~ 1600 cm^{-1} peak is respectively assigned to vibrations due to sp^2 carbon bonding in aromatic rings and olefinic chains [21–23]. Thus, the structures of as-prepared films on the Si flakes and steel balls, characterized by TEM, Raman and FTIR spectra, are considered as FL structures.

The coefficient of friction (COF) curves of self-mated FL-C:H films in humid air are shown in Fig. 4. The COF curves show a rapid drop at running-in stage under different loads, indicating the occurrence of distinct shear weakening at the contact interface. The steady COF has been presented as the average friction values after the running-in stage. Accordingly, the COF at the stable sliding period drops into super-low state with increasing normal load. For example, the COF at normal load of 8 N is about 0.012, and approaches 0.009 when the normal load reaches 10 N.

The corresponding SEM and 3D surface profiles of the wear scars on the coated steel balls were shown in Fig. 5. A great deal of loose particle-like wear debris are scattered around the worn surface of 8 N, while there is little wear debris at 10 N and 12 N (Fig. 5a, c and e). Meanwhile, the wear scar area of 8 N is largest, and later it becomes smaller with increasing normal load. Since the COF depends on the true contact area, the decrease of true contact area will mean lower friction [24]. In addition, a tribofilm can be observed at all the loads, but the quantity of the 8 N tribofilm seems like to be lower than the higher load. This is supported by the 3D surface profiles of the wear scars. The thickness of the 8 N tribofilm is smaller than that of another higher load (Fig. 5b, d and f). The wear difference can be responsible for the drop

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