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# Tribological behavior of diamond-like carbon film with different tribo-pairs: A size effect study

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ABSTRACT

A friction force microscope (FFM) with different probes and a ball-on-disk (BOD) tribo-meter were used to investigate the tribological properties of diamond-like carbon (DLC) films. DLC films were prepared by chemical vapor deposition (CVD) method by altering the deposition parameters, and their morphologies and structural information were examined with an atomic force microscope (AFM) and the Raman spectrum. The wear traces of the DLC films after frictional tests were analyzed by an optical microscope. It is found that surface roughness and adhesion play important roles in characterizing the tribological properties of DLC films using FFM. Moreover, the debris accumulation is another significant factor affecting the frictional behavior of DLC films, especially for the sharp tip. The difference in coefficients of friction (COFs) obtained by the BOD method among different DLC films under water lubrication is much smaller than the case without water lubrication. The variation trends in COF for the flat tip and the BOD test are similar in comparison with the result obtained with the sharp tip. The wear traces after frictional tests suggest that DLC films under water lubrication are prone to be damaged more readily.



## 1. Introduction

Amorphous carbon film due to its particular diamond-like performance has been attracting much attention. As the deposition process is gradually perfected, diamond-like carbon (DLC) film, with high mechanical hardness, chemical inertness, optical transparency, and wide band gap, is becoming the focus of thin film area [1]. At present, DLC film shows its great potential in applications for its peculiarities of decoration and function, such as optical windows, magnetic storage disks, biomedical coatings, micro-electromechanical system (MEMS) devices, and field emission materials [2,3].

Many previous studies on DLC films have been carried out mostly for DLC deposition technology [4–9] or the mechanical and macro-tribological characteristics of DLC coating itself [4,6–8,10–15]. The surface mechanical behavior and micro-tribological properties with the thickness decreasing in order to satisfy the desire for ultra high storage capacity and minimizing devices are also focused on. Atomic force/friction force microscope (AFM/FFM) appears to be an ideal instrument for evaluating these properties. Extensive studies have been conducted on the micro-tribological characterization [16–18] and wear resistance [19] of different kinds of DLC films using the AFM/FFM and AFM-based nanowear testing techniques. Also, the tribological properties of DLC films in aqueous environment suggest that DLC film is a promising tribo-material for rubbing components in water hydraulic machinery in spite of some limitations [11,20–22].

Despite these progresses, a comparative study of the tribological properties of different DLC films prepared by various deposition parameters using different tribo-pairs with different sizes has been rarely studied. In the present work, AFM/FFM with different probes and a ball-on-disk (BOD) tribo-meter were used to do such work in ambient air and water environment, and then the underlying tribological mechanisms will be put forward.

## 2. Experimental

### 2.1. Test sample

All DLC films were deposited on the silicon wafers by an electron cyclotron resonance (ECR) microwave plasma chemical

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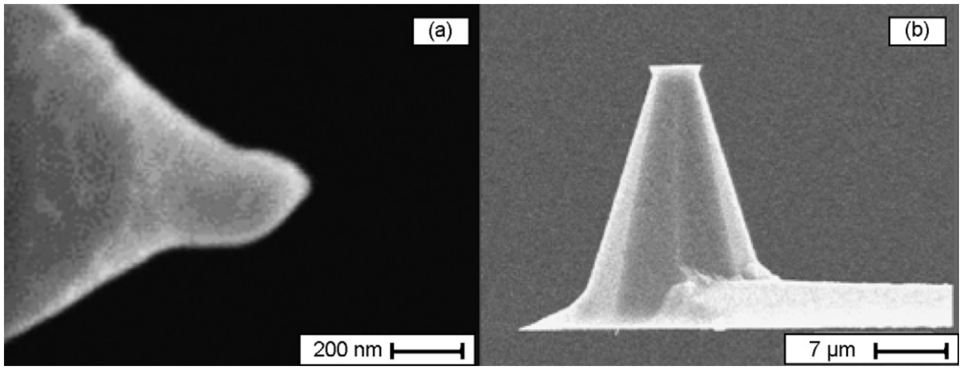


**Fig. 1.** SEM micrographs of the tips used in the experiment: (a) standard sharp tip and (b) customized flat tip.

vapor deposition technique. Prior to deposition, the wafers were ultrasonically pre-cleaned with acetone and then ethanol, rinsed in deionized water, dried with nitrogen before being loaded into the deposition chamber. Subsequently, the chamber was evacuated to a base vacuum with a pressure of $2.0 \times 10^{-3}$ Pa. Methane ($CH_4$) and hydrogen ($H_2$) gas mixtures were used as the working gas, and the overall gas flow was 50 sccm ($CH_4$ 40 sccm and $H_2$ 10 sccm) with a working pressure of 0.17 Pa. The experimental system consists of microwave hardware capable of delivering microwave power at 2.45 GHz, 1.5 kW. Magnetic coils arranged around the peripheries of discharge chambers generate magnetic field of 875 G necessary for ECR condition. Three samples were obtained by changing the dc substrate bias voltage of 300, 350 and 400 V, and numbered as DLC 1, DLC 2, and DLC 3 for convenience. The substrate temperature was less than 60 °C, and the depositing time was 120 min with a film thickness of approximately 600 nm.

### 2.2. Test method

DI Nanoscope III scanning probe microscope (SPM) with a tapping mode was employed to analyze the surface topography of the prepared films, and their roughness values were subsequently evaluated with the off-line analysis software. A microscopic confocal Raman spectrometer (Renishaw Corporation) was used to analyze the chemical state of carbon and structural information of the DLC films utilizing Argon laser with a wavelength of 514.5 nm as the source of excitation. A micro-XAM (Nikon Corporation) three-dimension profiler was used to observe the wear trace after frictional tests. The water contact angle of the surface was measured with the DSA100-e (KRÜSS, Germany) contact angle system. The hardness was measured by a nanoindentor from Hysitron Company.

Adhesion and micro-friction experiments were conducted with the friction force mode of the SPM, and a laser beam deflection technique was used to detect the distortion of a flexible cantilever. For the adhesion measurement, it was evaluated by measuring the vertical deflection of the cantilever when the tip approached and left the sample surface. For the micro-friction measurement, the cantilever scanned the surface laterally, resulting in its torsion, which was proportional to the frictional force between the tip and sample surface. The measured frictional value was denoted with the voltage signal directly, which would not affect the qualitative illustration, due to the uncertainties of the lateral sensitivity of the cantilever calibration [23]. Two tips with different shapes were employed in the experiment. One was a typical silicon nitride NP-S probe with a spring constant of 0.58 N/m, which was consisted of a cantilever of 100-μm long and a sharp tip at the end (as illustrated in Fig. 1(a)), and its nominal tip radius was 20–50 nm. The other one was a flat silicon tip with a flat size of approximately 4.5 μm and a nominal spring constant of 3.1 N/m, as shown in Fig. 1(b). The adhesion measurement was conducted with the NP-S probe, and at least five such measurements were performed on each sample to obtain the average value. All the experiments were carried out in ambient environment (RT = 25 °C, RH = 60 ± 5% relative humidity). The frictional properties were also evaluated using a reciprocating BOD tribo-meter (UMT-2MT, CETR Company). The reciprocating amplitude in sliding was 5 mm. A GCr15 ball with a diameter of 4 mm and a surface roughness $R_a$ of about 40 nm was used as the counterpart. The average sliding speed was 0.015 m/s for a fixed sliding time of 10 min. The normal load applied was 0.5 N, and the corresponding contact pressure was 398.1 MPa.

## 3. Results and discussion

### 3.1. Material properties

The topographical images of the prepared DLC films are shown in Fig. 2, and their measured roughness and adhesion values are also shown in Table 1. It can be seen that with the increase of the bias voltage, the specimen tends to go flat, however, the adhesion between the tip and sample presents an increase trend, implying that the surface free energy increases. The result of water contact angle in Table 1 also shows a similar trend in surface free energy to the adhesion result. The observed difference of the film texture might be caused by an increase in the areal density of the nucleation sites and the mobility of adatoms with more high-energy ions arriving at the substrate with increasing the substrate bias. The variation in adhesion and water contact angle should be

**Table 1**
The measured technical data of different DLC films

| Sample | Substrate bias voltage (V) | Roughness RMS (nm) | Hardness (GPa) | Elastic modulus (GPa) | Adhesion (nN) | Contact angle (°) | Friction coefficient (mV/nN) | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | Sharp tip | Flat tip |
| DLC 1 | 300 | 0.56 ± 0.05 | 16.09 ± 2.93 | 134.50 ± 21.26 | 13.13 ± 0.30 | 87.67 ± 0.22 | 0.50 ± 0.03 | 0.91 ± 0.04 |
| DLC 2 | 350 | 0.21 ± 0.03 | 19.15 ± 0.95 | 146.51 ± 2.33 | 23.87 ± 0.50 | 79.83 ± 2.22 | 0.63 ± 0.03 | 0.43 ± 0.02 |
| DLC 3 | 400 | 0.19 ± 0.02 | 19.53 ± 0.39 | 130.29 ± 4.15 | 27.32 ± 0.60 | 70 ± 1 | 0.58 ± 0.02 | 0.33 ± 0.01 |