



Effect of tribochemistry on friction behavior of fluorinated amorphous carbon films against aluminum



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ABSTRACT

Fluorinated amorphous carbon (a-C:H:F) films synthesized from C₂H₂ and CF₄ were examined by Raman spectra and X-ray photoelectron spectroscopy (XPS). Their tribological properties were tested against aluminum balls in dry sliding. The a-C:H:F films with low F content showed lower friction coefficient than hydrogenated amorphous carbon (a-C:H) film, whereas high F content in films resulted in a significant friction increase. Contact surfaces were analyzed in detail to elucidate the possible sliding mechanism. Results indicated that the friction behavior was closely related to the nature of a composite-like tribo-layer consisting of Al compounds and carbon components formed on Al ball, relying on the tribochemical processes of contact interface. The accumulated F atoms on tribo-layer reduced the adhesion across sliding interface because of strong repulsion between F atoms, and thereby lowered the friction of a-C:H:F films. However, with increasing F contents in films, the enhanced tribochemical reaction between Al and F caused crack, delamination and fragmentation of the tribo-layer, and then a marked abrasive process at the sliding interface. Consequently, the increased shearing and abrasive actions strongly opposed the contribution of the reduced adhesion to friction, and result in a rather high friction of highly fluorinated a-C:H:F films.

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1. Introduction

The use of lubricating coatings greatly alleviates aluminum adhesion and transfer to tools, a very pressing problem in Al alloys dry machining [1]. Diamond-like carbon (DLC) films, with excellent tribological properties, have shown better anti-adhesive effect and lower friction coefficient than other conventional TiN, ZrN, TiAlN and TiCN coatings when tested against Al [2–5]. This has greatly stimulated interest in using them as promising tool coatings for Al dry machining. However, to improve the performance of DLC films, lower friction in DLC/Al contact is a continual drive for optimizing and designing DLC films. Thus, it is quite necessary to probe the detailed friction mechanism of various DLC films against Al.

Many studies have investigated the friction behavior and mechanism of DLC films and their modifications against Al under different conditions. Non-hydrogenated DLC films exhibit high friction coefficient ($\mu = 0.5\text{--}0.8$) under high vacuum and inert (He, Ar and N₂) environments, where the adhesive transfer of Al to DLC surface results in an undesirable Al–Al contact [6,7]. On the contrary, low friction ($\mu < 0.2$) is obtained as the introduction of H₂ and H₂O that passivate the

unoccupied C— σ bonds on carbon surface [6–8]. Oxygen results in a high friction ($\mu = 0.28\text{--}0.44$) in DLC/Al contact due to the oxidation of Al ball and carbon surface, but no Al adhesion transfer was observed. [6,9,10]. Hydrogenated DLC films exhibited lower friction coefficient than H-free DLC at evaluated temperature and showed lower cutting forces and less adhesion, attributed to higher chemical inertness of H-containing DLC network. [11,12]. The a-C:H/a-Si:O coating showed low steady-state friction coefficient up to 400 °C, where a Si-containing carbon-based transfer layer terminated by H, O and OH was identified on Al surface [13]. Similarly, a nano-structural carbon tribo-layer terminated by H, HO and F on Al surface accounted for the low friction behavior of a-C:H:Si:O:F coating in both vacuum and ambient atmosphere [14]. In addition, the formation of WO₃ on Al surface dominated the low friction behavior of W-DLC/Al at 400 °C [15]. Briefly, the passivation of free C— σ bonds and the formation of tribo-layer on Al surface are fundamental to the achievement of low friction in DLC/Al contact.

Fluorinated amorphous carbon (a-C:H:F) films have been investigated as anti-sticking coatings in nanoimprint lithography and biomedical applications because of their low surface energy property [16–18]. Theoretical calculations reveal that fluorinated carbon surfaces display lower adhesion tendency when facing each other [19–21]. Meanwhile, Donnet et al. [22] found that a-C:H:F films showed comparable tribological properties to non-fluorinated ones when F content is <20 at.%. Interestingly, when tested against Al counterparts, Sen et al. [23] recently found that FDLC film showed lower friction coefficient than HDLC film.

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They pointed out that a fluorinated transfer layer on Al surface may reduce the friction level of FDLC/Al contact. This suggests a potential use of a-C:H:F films in Al anti-adhesion. However, this meaningful result was only observed and discussed for a certain FDLC film (3 at.% F, 25 at.% H) in ambient air, many issues are still open. Most importantly, considering the large electro-negativity of F and fluorinated tribo-layer, a-C:H:F films perhaps signify more complicated tribochemical processes, especially with increasing F contents, which possibly markedly influence the friction behavior of a-C:H:F against Al. So, it is of great significance to know the relevant details, which can offer more complete information to further consider possible use of a-C:H:F films, e.g. in Al dry machining.

The present work, accordingly, focuses on the influence of F content and tribochemistry on the friction behavior of a-C:H:F against Al. The a-C:H:F films were produced from the glow plasma decomposition of C_2H_2 and CF_4 mixture. The fundamental nature of films was characterized and understood. The friction behavior of films was assessed under dry contact conditions. The contact surfaces were examined to identify the factors that influenced the friction behavior of a-C:H:F films against Al.

2. Experimental procedure

2.1. Materials

The a-C:H:F films were deposited on silicon (110) and mirror-polished stainless steel plates (roughness: $R_a < 20$ nm) by plasma enhanced chemical vapor deposition (PECVD) system, using acetylene (C_2H_2) and carbon tetrafluoride (CF_4) as precursor gases. The base pressure of deposition chamber was better than 2×10^{-3} Pa. Solvent cleaning and Ar^+ etching procedures were performed to remove contaminants on substrate surfaces before deposition process. A thin a-Si:H interlayer (~ 80 nm) and a-C:Si:H gradient layer (~ 500 nm) were produced to improve the adhesion of film to substrates. The a-C:H:F layer was synthesized from C_2H_2 and CF_4 at a pressure of 6.5 Pa. A pulse power source was used (frequency, 1.5 kHz; duty ratio, 30%). The gas flow ratio (C_2H_2/CF_4) and the corresponding sample are listed in Table 1. The a-C:H (about 10 at.% H) and a-C:H:F-6 films were deposited at substrate voltage -0.8 kV and -1.0 kV, respectively, and the other films were produced at -0.9 kV. The total thickness of films was about $2.0 \mu m$ (measured by SEM).

2.2. Friction and wear tests

The sliding experiments were performed using a CSM tribometer and a ball-on-disk contact geometry with a reciprocating motion under a load of 3.0 N, a speed of 0.05 m/s (frequency: 5.0 Hz, stroke length: 2.5 mm). The counterpart was commercial purity ($>99\%$) aluminum ball with a diameter of 4.0 mm, Vickers hardness of about 270 MPa and the surface roughness, R_a , of ~ 60 nm. Aluminum balls were tested against films deposited on steel substrates in ambient air with a relative humidity (RH) of $30 \pm 2.5\%$ and halted after 20,000 laps, corresponding to a sliding distance of 200 m. The cross-sectional area of the track was determined by a profile-meter (KLA-Tencor, D-100). Furthermore,

Table 1
Source gas ratio, F content, hardness (H), elastic modulus (E), internal stress (σ) and water contact angle (θ) of as-deposited films.

Sample	$C_2H_2/CF_4/sccm$	F/at.%	H/GPa	E/GPa	σ /GPa	$\theta/^\circ$
a-C:H	120/0	0	14.8	127.8	-0.85	71.8 ± 0.7
a-C:H:F-1	60/20	1.7	15.4	131.5	-0.70	78.3 ± 0.7
a-C:H:F-2	60/40	3.4	14.6	134.4	-0.61	77.2 ± 1.5
a-C:H:F-3	60/60	6.5	13.4	122.7	-0.43	78.0 ± 0.7
a-C:H:F-4	50/70	9.6	12.9	116.3	-0.58	74.7 ± 0.8
a-C:H:F-5	40/80	16.7	11.8	99.1	-0.41	81.7 ± 1.3
a-C:H:F-6	30/90	18.6	10.3	89.4	-0.41	81.6 ± 2.4

friction tests were also carried out under dry N_2 (RH: 1.0%), dry air (RH: 5.0%) and humid air (RH: 60.0%) to consider environmental effect.

2.3. Characterization methods

Micro-Raman spectra were recorded by a LabRam HR800 Jobin-Yvon spectrometer with an excitation wavelength of 532 nm. The hardness (H) and elastic modulus (E) of films were determined by a nanoindenter Hysitron TI 950 USA. The residual stress of films was determined through the deformation of Si wafer caused by the deposition of film (SuPro Instruments, FST-1000). Surface compositions and chemical states of samples were analyzed by an ESCALAB 250Xi (Thermo Fisher, USA) X-ray photoelectron spectroscopy (XPS) system with an $Al K\alpha$ (1486.7 eV) X-ray source. A DSA 100 goniometer was employed to measure water contact angles. Surface morphology of wear tracks was acquired by 3D Laser Scanning Microscope (Keyence VK-X100/X200, Japan). The transfer layers were observed using a JSM-5600LV scanning electron microscope (SEM) equipped with an energy-dispersive X-ray spectroscopy (EDS).

3. Results and discussion

3.1. Characterization of films

The micro-structure of films was investigated by Raman spectroscopy. As shown in Fig. 1, the broad, asymmetrical Raman peaks between 1000 and 1700 cm^{-1} suggested disordered carbon structures. According to recent publications by Neuville [24,25], the spectra can be resolved into two sub-bands: the so-called D band at $1340/1398\text{ cm}^{-1}$ and C5/C7 odd ring band at $1534/1552\text{ cm}^{-1}$. The so-called D band in fact corresponds to either disordered diamond (1330 cm^{-1}) or to the G Aedge vibration modes of sp^2 clusters (1350 cm^{-1}). For a-C:H film, the D band at 1340 cm^{-1} mainly corresponded to disordered $C_{sp^3}-C_{sp^3}$ clusters. The C5/C7 band at 1534 cm^{-1} indicated that there were no significant hexagonal cyclic sp^2 rings within film, which further signified that the D band at 1340 cm^{-1} could only encapsulate weak G Aedge

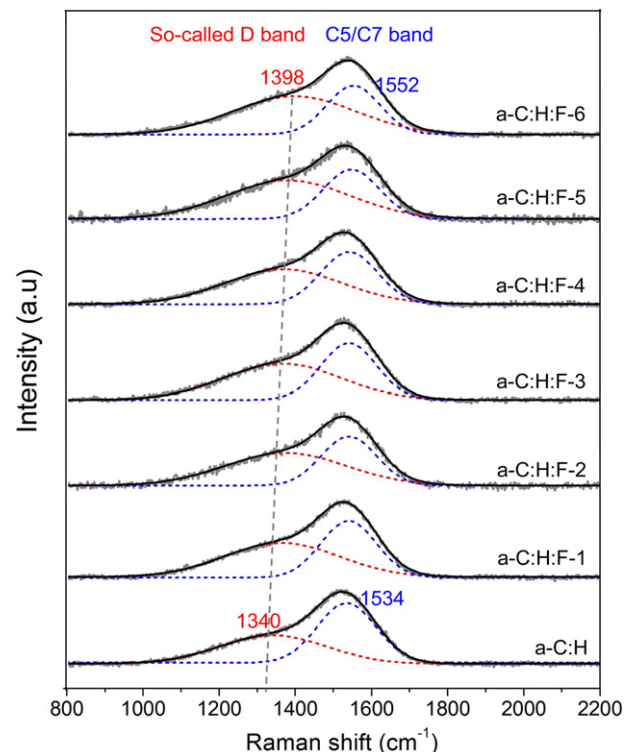


Fig. 1. Raman spectra of a-C:H and a-C:H:F films.

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