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Humidity dependence on the friction and wear behavior of diamond-like carbon film in air and nitrogen environments

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Abstract

Diamond-like carbon (DLC) films were deposited on Si (100) wafers by a plasma enhanced chemical vapor deposition (PECVD) technique using CH₄ plus Ar as the feedstock. The friction and wear behaviors of the resulting film sliding against Si₃N₄ balls were investigated on a ball-on-disk test rig in air and nitrogen environments at a relative humidity from 5% to 100%. The worn surface morphologies of the DLC film and the Si₃N₄ counterpart were observed on a scanning electron microscope (SEM), while the chemical states of some typical elements thereon were investigated by means of X-ray photoelectron spectroscopy (XPS). It was found that the DLC film recorded continuously increased friction coefficient and wear rate with increasing relative humidity in air. It showed linearly increased friction coefficient with increasing relative humidity in nitrogen, in this case the wear rate sharply decreased and reached the minimum at a relative humidity of 40%, which was followed by an increase with further increase of the relative humidity. The interruption of the transferred carbon-rich layers on the Si₃N₄ balls, and the friction-induced oxidation of the films in higher relative humidity were proposed to be the main reasons for the increases of the friction coefficient and wear rate. Moreover, the oxidation and hydrolysis of the Si₃N₄ ball in higher relative humidity, leading to the formation of a tribochemical film that mainly consists of silica gel on the wearing surface, were also thought to have effects on the friction and wear behaviors of the DLC films. © 2006 Published by Elsevier B.V.

Keywords: PECVD; DLC film; Friction and wear behavior; Relative humidity

1. Introduction

Diamond-like carbon (DLC) films exhibit attractive mechanical and tribological properties (e.g. high hardness, low friction, high wear resistance, etc.), which makes them suitable for numerous potential tribological applications such as magnetic hard disks, gears, bearings, cutting tools and other moving mechanical assemblies [1–3]. However, previous studies have shown that the tribological properties of the DLC films are strongly dependent on the nature of the films and on the deposition methods and conditions, and are very sensitive to the testing environment, especially to the relative humidity (RH) [4–14]. Depending on the above-mentioned factors, the friction coefficients for the DLC films have been reported to broadly range from 0.003 to more than 0.5 [7,9,13–19].

Various theories have been proposed to explain the friction and wear behaviors of DLC films. The build-up of a friction-induced transfer film on the counterpart, followed by easy shear within the interfacial material is the most frequently observed friction-controlling mechanism for DLC films [3,10,20,21]. Liu et al. said that the steady-state low friction of DLC films was due to the wear-induced graphitization, i.e., the formation of a low friction graphitized tribolayer [7,22]. Erdemir et al. investigated the tribological properties of different DLC films in open air and dry nitrogen, and argued that the general low friction coefficient of hydrogenated DLC films was mainly attributed to the high chemical inertness of the DLC films by passivating the surface dangling bonds by hydrogen [17,23]. Many other researchers proposed that the tribochemical reactions in the tribo-system played an important role in determining the friction and wear behaviors of DLC films [2,3,13,20]. However, these reported friction and wear mechanisms for the DLC films are the subjects to controversy and the role of

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the environments on the friction and wear behaviors of DLC films is still not well understood.

With that perspective in mind, the effect of relative humidity (RH) on the friction and wear behavior of DLC film in air and nitrogen environments was investigated, in a hope to reveal the humidity dependence of the friction and wear behavior of the DLC film in air and nitrogen and to clarify the friction and wear mechanism of the DLC film as well.

2. Experimental details

2.1. Deposition and characterization of the DLC film

DLC film was deposited on Si (100) wafers making use of PECVD technique, using CH₄ plus Ar as the feedstock. The details about the deposition equipment and process were described elsewhere [5,24]. Prior to deposition, the substrates were cleaned with Ar plasma sputtering at a bias voltage of -400 V for 15 min so as to remove the native oxide on the Si surface. The deposition conditions and some properties of the resulting DLC film are summarized in Table 1.

The deposited DLC films are dark brown in color and are extremely smooth and featureless when viewed with unaided eyes. The film thickness measured on a profilometer is about 640 nm using. The hardness and Young's modulus of the film are determined to be about 15.2 GPa and 120 GPa, respectively, using a nano-indenter.

2.2. Friction and wear test

The friction and wear behavior of the DLC film sliding against a Si₃N₄ ball was evaluated on a ball-on-disk test rig equipped with an environmental chamber with which the relative humidity and gaseous environment could be controlled. The friction and wear tests were performed in air and nitrogen environments, respectively, with the relative humidity ranging from 5% to 100%, at a normal load of 2 N, a sliding velocity of about 125 m/min, to a maximum sliding duration of 60 min.

The wear rate of the DLC film was calculated from the wear track profiles measured on a surface profilometer. The worn surfaces of the DLC film and the counterpart Si₃N₄ ball were analyzed on a JSM-5600LV scanning electron microscope (SEM) equipped with a KEVEX SIGMA energy dispersive X-ray spectrometer (EDS). The chemical composi-

tions and chemical states of the worn surfaces were analyzed on a PHI-5702 X-ray photoelectron spectroscope (XPS) operating with monochromated Al-K α irradiation at a pass energy of 29.4 eV.

3. Results and discussion

3.1. Friction and wear behaviors

Fig. 1 shows friction behaviors of the DLC film at different relative humidity in air and nitrogen environments. It is seen that the friction coefficients continuously increase with increasing relative humidity in both air (Fig. 1(a)) and nitrogen (Fig. 1(b)) environments. Moreover, the friction coefficient in nitrogen is always lower than that in air under the same relative humidity (Fig. 1(c)). Particularly, in dry nitrogen (RH-5% and RH-20%), the DLC film fails very quickly, though it records a much low friction coefficient.

Fig. 2 shows the variation of the wear rates of the DLC film with the relative humidity in air and nitrogen. It is seen that the wear rate of the DLC film is closely dependent on the relative humidity and environment. Namely, the wear rate of the DLC film continuously increases with increasing relative humidity in air, while it decreases sharply with increasing relative humidity in nitrogen and reaches the minimum at a relative humidity of 40%. In the latter case, the friction coefficient assumes an increase with further increase of the relative humidity above 40%. At a relative humidity below 40%, the DLC film has a much higher wear rate in nitrogen than that in air, while above a relative humidity of 40% it shows a smaller wear rate in nitrogen than in air. In particular, the DLC film failed quickly in dry nitrogen (relative humidity about 5%) and recorded a specific wear rate of approximately 6.5×10^{-7} mm³/N m, which was larger than that in dry air (7.1×10^{-9} mm³/N m) by nearly two magnitude orders.

3.2. SEM analyses of the worn surfaces

In order to gain more insights into the friction and wear mechanisms of the DLC film sliding against the Si₃N₄ ball under different relative humidity in air and nitrogen environments, SEM and EDS analyses of the worn DLC film and the counterpart Si₃N₄ surfaces were performed. Figs. 3 and 4 show the SEM pictures of the worn surfaces of the DLC film and the counterpart Si₃N₄ ball at different relative humidity in air and nitrogen environments. It is seen that the worn surface of the DLC film in air is very much smooth and shows only signs of slight scuffing (Fig. 3(a)). The worn surface of the counterpart Si₃N₄ ball in this case is covered by transferred materials, with the transferred layer mainly composed of carbon to be compact and homogeneous (Fig. 3(b)). Contrary to the above, the DLC film sliding against the Si₃N₄ ball in dry nitrogen is characterized by obvious flake-like peeling off (Fig. 3(c)), though the counterpart Si₃N₄ ball is still covered by a transferred carbon-rich layer in this case (Fig. 3(d)). The accumulation of a large number of wear debris around the contact region of the Si₃N₄ ball indicates that the DLC film

Table 1
Summary of the deposition conditions and some properties of the DLC film

Item	Parameters
CH ₄ gas flow rate	11.6 sccm
Ar gas flow rate	11 sccm
Deposition pressure	5 Pa
RF power	300 W
DC bias	200 V
Deposition time	3 h
Thickness	640 nm
Hardness	15.2 GPa
Young's modulus	120 GPa

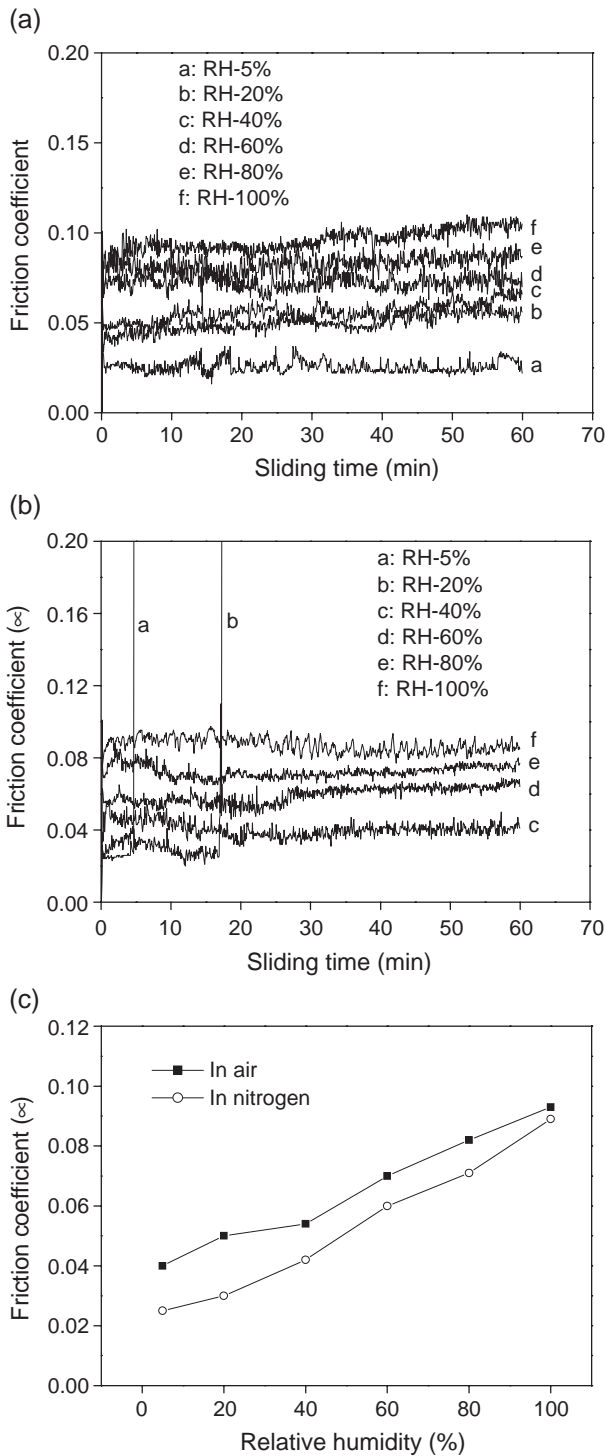


Fig. 1. Friction behaviors of the DLC film at different relative humidity: (a) friction coefficient as a function of sliding time in air; (b) friction coefficient as a function of sliding time in nitrogen; (c) average friction coefficient as a function of relative humidity.

experiences a mass-transfer to Si_3N_4 ball during the friction process.

As the relative humidity in nitrogen increases to 40%, the worn surface of the DLC film is smooth and shows many granular wear debris scattered at the edge of the wear track (Fig. 4(a)). The corresponding contact area of the Si_3N_4 ball is

covered by a compact and dense transferred carbon layer aside from the surrounding loose wear debris (Fig. 4(b)). However, when the relative humidity in nitrogen increases to 100%, obvious differences will be seen on the worn surfaces of the DLC film and the counterpart Si_3N_4 as compared with those at a relative humidity of 40% in nitrogen. As shown in Fig. 4(c), at a relative humidity as high as 100% in nitrogen, a discontinuous layer is formed in the central wear track of the DLC film, and more wear debris is gathered on the wear track edge. The corresponding worn surface of the Si_3N_4 ball is larger and much rougher, indicating a severe wear of the ball (see Fig. 4(d)). The similar changes of the worn surfaces of the DLC film and the counterpart Si_3N_4 ball are also observed with increasing relative humidity in air.

3.3. XPS analysis of the worn surface

Table 2 summarizes the changes in the concentrations (at.%) of C, Ar, O, and Si, determined by XPS analysis (the XPS data are estimated to have an error of 10%), on the original and worn surfaces of the DLC film at varied testing conditions. The presence of Ar in the film is attributed to the CH_4 plus Ar as the feedstock. The Ar concentration on the worn surface of the DLC film decreases owing to the removal of the Ar atoms during the friction process. The concentration of O on the worn film surfaces tested at a relative humidity of 5%, 40% and 100%, respectively, is 24.7 at.%, 27.7 at.%, and 28.3 at.%, which is much larger than 5.3 at.%, that of the original DLC film. Moreover, as the relative humidity exceeds 40% in air, the concentration of Si increases as well. Similar variation trends are also observed for the O and Si concentrations on the worn surfaces of the DLC film with increasing relative humidity in nitrogen environment. It should be noted that in dry nitrogen (relative humidity about 5%), the O concentration on the wear track of the DLC film is 6.4 at.%, which is higher than that of the original film (5.3 at.%) but much smaller than 24.7 at.%, that on the wear track of the DLC film tested in dry air, due to the protection action of nitrogen. Thus, it can be inferred that the

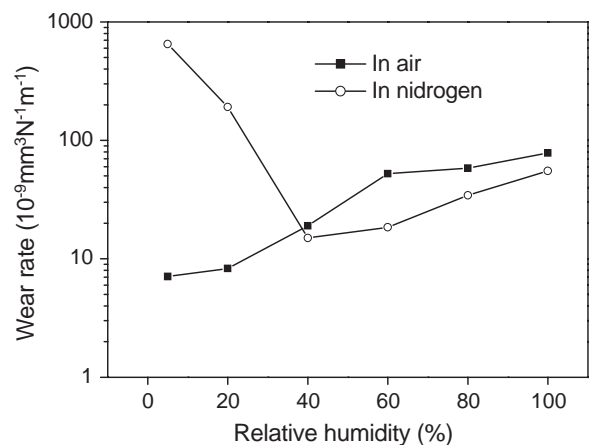


Fig. 2. Wear rate of the DLC film as a function of relative humidity in air and nitrogen environments.

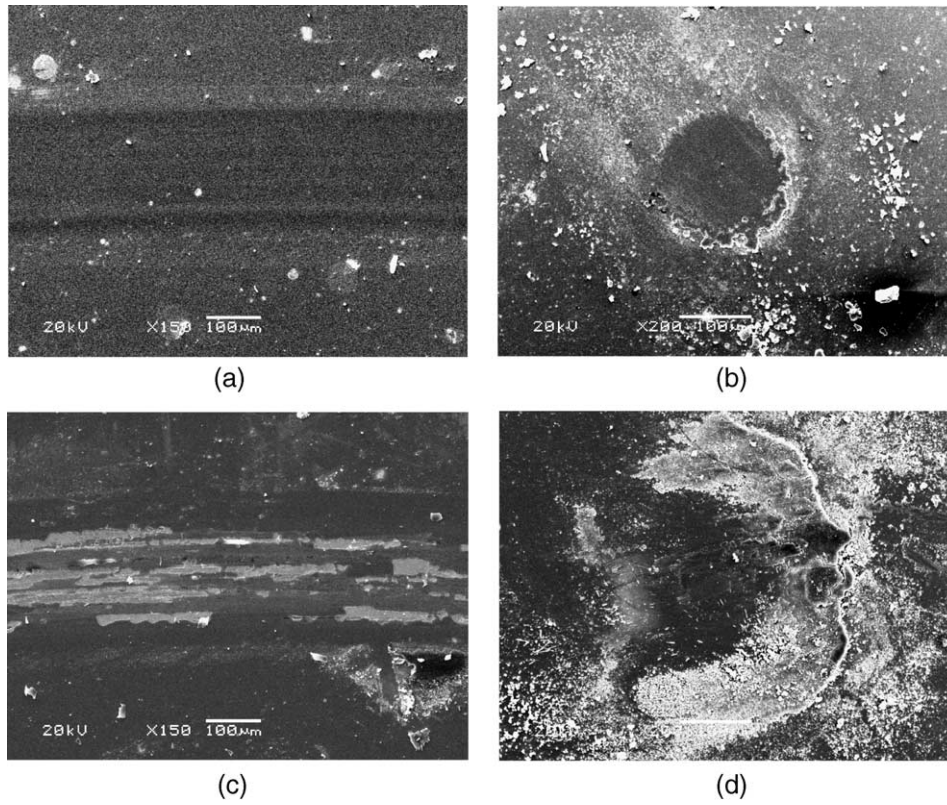


Fig. 3. SEM morphologies of the worn surfaces of the DLC film (a, c) and counterpart Si₃N₄ ball (b, d) in dry air and nitrogen (relative humidity about 5%): (a) and (b) in dry air; (c) and (d) in dry nitrogen.

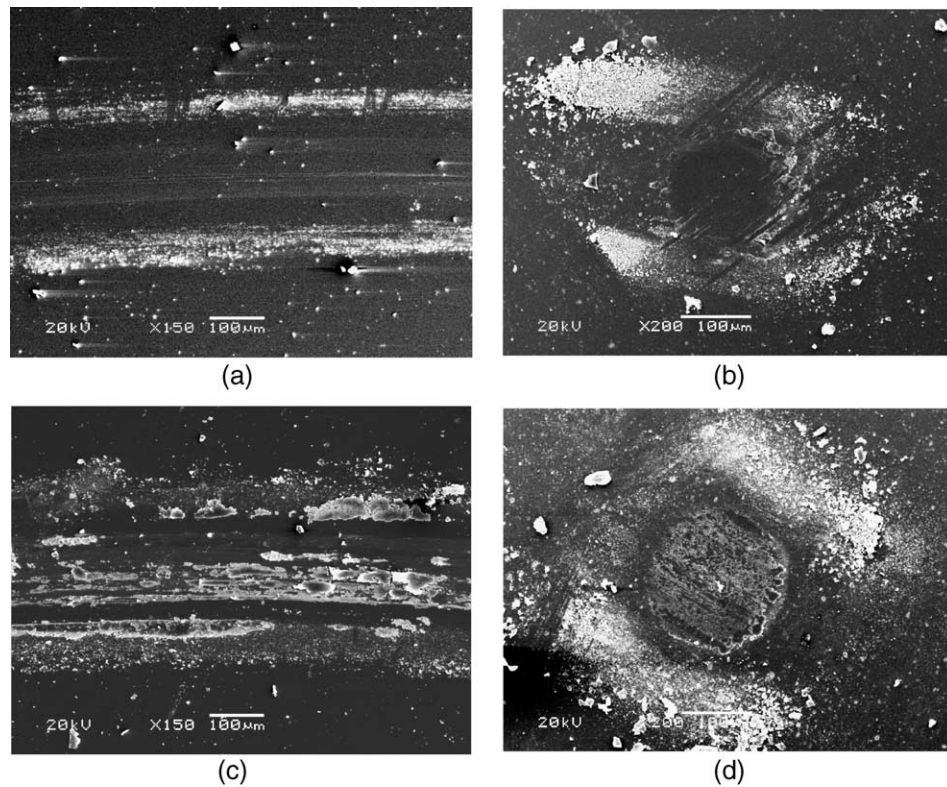


Fig. 4. SEM morphologies of the worn surfaces of the DLC film (a, c) and counterpart Si₃N₄ ball (b, d) in humid nitrogen: (a) and (b) 40%RH; (c) and (d) 100%RH.

Table 2
Changes of concentrations (at.%) of C, Ar, O, and Si on the original and worn surfaces of the DLC film

Element	As-deposited film	Worn surfaces of the DLC films tested in different environments					
		In air (RH%)			In nitrogen (RH%)		
		5%	40%	100%	5%	40%	100%
C	89.8	73.2	69.5	62.6	92.3	82.0	68.1
Ar	4.9	2.1	2.6	2.3	1.3	1.2	1.9
O	5.3	24.7	27.7	28.3	6.4	16.3	22.5
Si	0.0	0.0	0.2	6.8	0.0	0.5	7.5

DLC film experiences structure change and tribochemical reactions during the friction process.

Fig. 5 shows the XPS spectra of C1s and O1s on the original surface of the DLC film. The C1s spectra can be fitted into three components around 284.8 eV, 286.3 eV, and 288.7 eV, respectively (Fig. 5(a)). The one around 284.8 eV corresponds to C–C or C–H bonds, and that around 286.3 eV corresponds to C–O bonds [25,26]. The third peak of a much smaller intensity

near 288.7 eV is assigned to C=O bonds [25,26]. At the same time, the symmetrical O1s peak at 532.6 eV is attributed to C–O bonds caused by the absorbed oxygen on the film surface due to air exposure (Fig. 5(b)).

Figs. 6 and 7 show the XPS spectra of C1s on the wear tracks of the DLC films tested at different relative humidity in air and

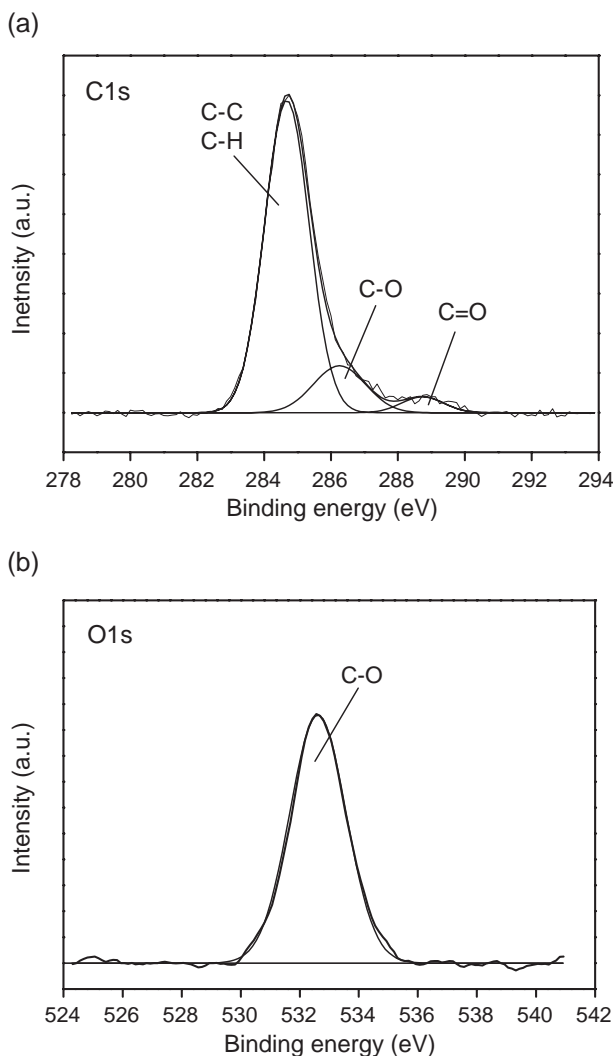


Fig. 5. XPS spectra of C1s (a) and O1s (b) on the surface of the as-deposited DLC film.

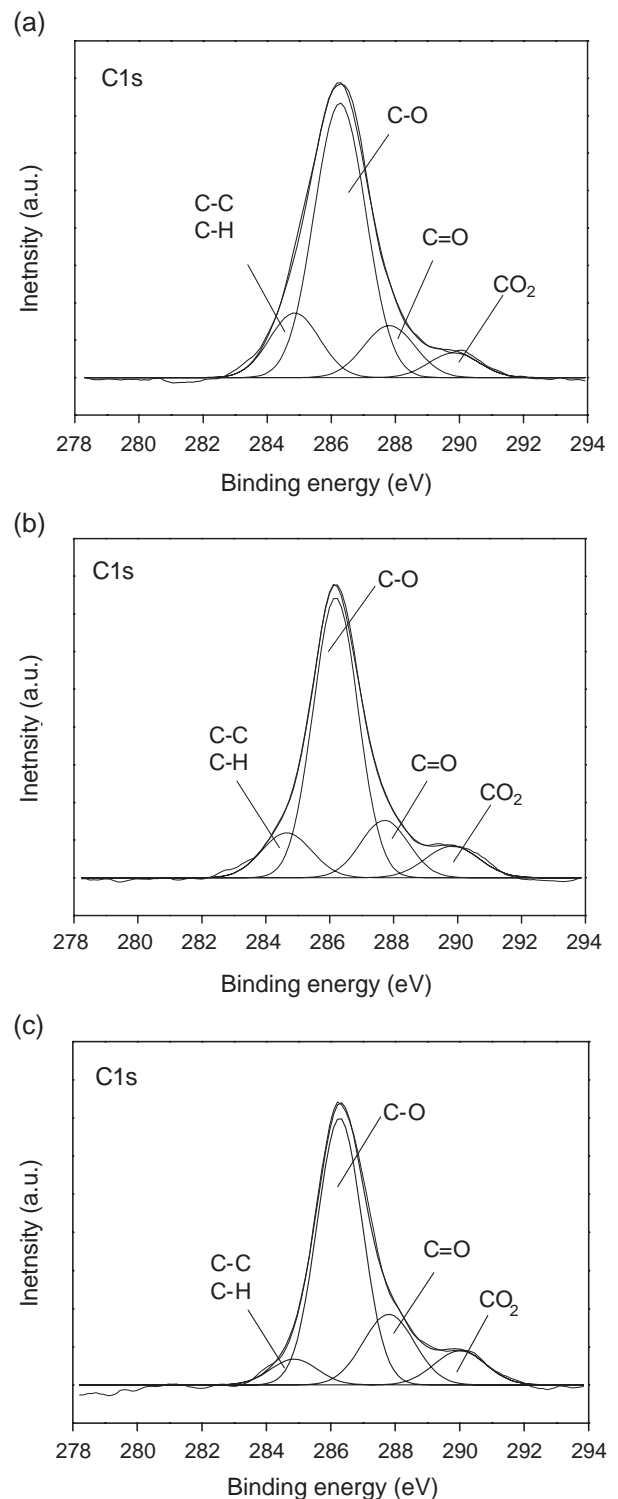


Fig. 6. XPS spectra of C1s on the wear tracks of the DLC film tested under different relative humidity in air: (a) 5%RH; (b) 40%RH; (c) 100%RH.

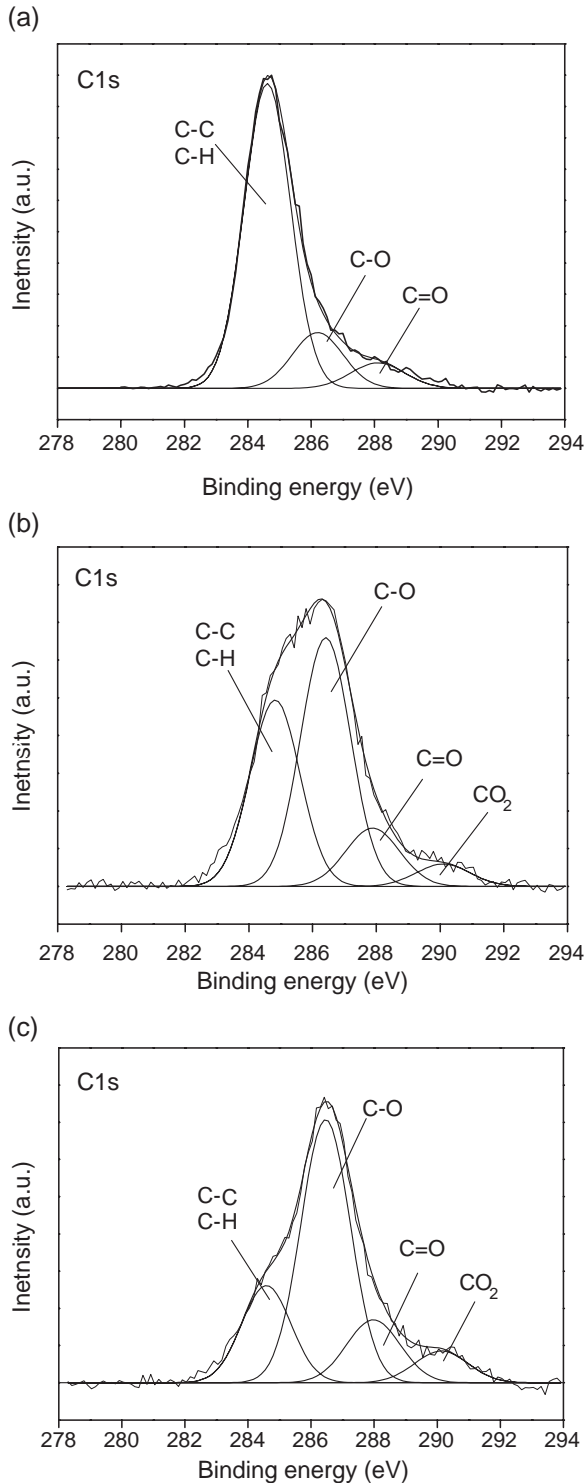


Fig. 7. XPS spectra of C1s on the wear tracks of the DLC film tested under different relative humidity in nitrogen: (a) 5%RH; (b) 40%RH; (c) 100%RH.

nitrogen, respectively. It is seen that the C1s spectra of the wear tracks tested at different relative humidity in air (Fig. 6) are much different from that of the original film (Fig. 5). With the increase of the relative humidity in air, the C1s core level spectrum shifts to a higher binding energy, the intensity of C–C (or C–H) peak decreases, while the intensities of C–O and C=O peaks significantly increase. A new C1s peak near 290.1 eV on

the wear track is ascribed to the adsorbed CO₂ groups [27]. Similar variation trends are also observed for the C1s states on the wear tracks of the DLC films with increasing relative humidity in nitrogen (see Fig. 7). It is interesting to note that the XPS C1s states on the wear track of the DLC film tested in dry nitrogen (relative humidity about 5%) are similar to that on the original surface of the film. Moreover, the C–C (or C–H) bond intensity on the wear tracks of the DLC films tested in nitrogen is higher than that in air under the same relative humidity. This could also be attributed to the protection action of nitrogen.

The XPS Si2p spectra on the wear tracks of the DLC film and the counterpart Si₃N₄ ball tested at a relative humidity of 100% in nitrogen are shown in Fig. 8. It is seen that the Si2p spectrum on the wear track of the DLC film is centered at 103.4 eV (see Fig. 8(a)), which is assigned to gel-type amorphous SiO₂ [28]. The asymmetrically broad Si2p spectrum on the worn surface of the Si₃N₄ ball can be fitted into two components around 101.8 eV and 103.4 eV, respectively (see Fig. 8(b)). The one around 101.8 eV corresponds to the Si–N bonds of the Si₃N₄ ball, and that around 103.4 eV to the Si–O bonds of gel-type amorphous SiO₂ [28].

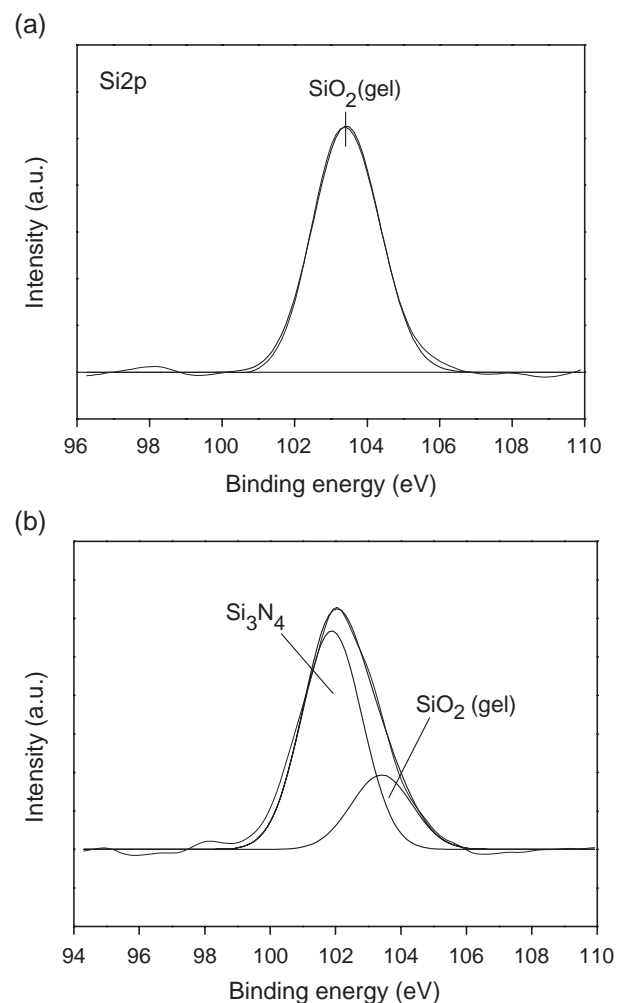
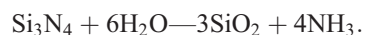
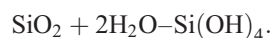


Fig. 8. XPS spectra of Si2p on the worn surfaces of the DLC film (a) and counterpart Si₃N₄ ball (b) tested at a relative humidity of 100% in nitrogen.

From the above-mentioned XPS analyses of the worn surfaces of the DLC film and the counterpart ceramic ball, it is deduced that two kinds of tribochemical reactions are involved during the friction testing under a higher relative humidity. One is the oxidation of the DLC film, and the other is the oxidation and hydrolysis of the Si_3N_4 ball. As reported by Kim et al. [20], the oxidation of the DLC film was expected to be similar to that of hydrocarbon polymers described by Scott [29]. Namely, the oxidation of the DLC film during the friction process was divided into three steps. First, the C–H bonds on the DLC film surface were mechanically broken by friction force and the water molecules (and O_2 molecules in air) were chemisorbed to form the C–OH or C–O groups. Secondly, the chemisorbed peroxides were transformed to COOH or remained as active radicals such as COO–. Thirdly, further shearing of the C–H bonds in the neighbors donated electrons to form C=O. Therefore, the DLC film experienced a change in the surface chemical states from C–H bonds to oxygen-containing groups during the friction testing. On the basis of a simple model for asperity temperature rise due to friction [30], Liu et al. had proposed that the temperature on the local asperity contact could reach about 1200 °C for the ceramic ball sliding against DLC film [22]. The temperature was so high that the contact points of the Si_3N_4 ball could react with water to form SiO_2 by way of the reaction below:



The resultant SiO_2 would further react with water to form silicic acid [28]:



The silicic acid would accumulate on the friction surface and form a thin hydrated film, therefore to reduce friction and wear.

3.4. General discussion

Erdemir et al. said that the low friction coefficient (0.025–0.093 in our work) of DLC films was mainly attributed to the high chemical inertness of the films [17,23]. Namely, in a highly hydrogenated plasma, constant hydrogen bombardment on the DLC films during growth process increases the hydrogen content of these films, and most of the hydrogen atoms are expected to be paired with the covalent or free σ -bonds, leading to the elimination of dangling bonds on the sliding DLC surfaces. Furthermore, the intense hydrogen ion bombardment also prevents cross-linking or C=C double bonding in growing DLC films and etches out the graphitic phases. Thus, the residual π – π^* interactions that can result from graphitic phases are also minimized when a highly hydrogenated plasma is used. Finally, some of the carbon atoms (at least those on the surface) can be di-hydrated (i.e. two hydrogen atoms bonded to one carbon atom). The presence of di-hydrated carbon atoms on DLC surfaces is expected to provide better shielding or a higher degree of chemical passivation. Subsequently, the dramatically lowered friction coefficients are recorded for the DLC films, owing to the elimination of the possibility of strong covalent

and π – π^* interactions at sliding DLC interfaces, plus better shielding and higher chemical passivation by di-hydration [23].

However, the significant effect of the relative humidity on the friction and wear behavior of the DLC film could never be underestimated (see Figs. 1 and 2). In other words, water had a specific effect on the formation of a DLC transferred layer on the counterpart surface and on the tribochemical reactions of the film itself and the counterpart ceramic as well (see Figs. 3–8).

As shown in Figs. 3 and 4, the formation of the transferred layer on the Si_3N_4 ball is strongly influenced by H_2O and O_2 . In dry air, the worn surface of the Si_3N_4 ball is covered by a thick and compact transferred carbon-rich layer (see Fig. 3(b)). This transferred carbon-rich layer helps to prevent the direct contact between the ceramic ball and the DLC film and acts as a “lubricant” of a low-shear-strength at the interface to result in a much small friction coefficient (0.03) [2,20,21]. With the increase of the relative humidity, water vapor can be condensed on the contact zone to prevent the formation of the transferred carbon-rich layer on the Si_3N_4 ball [7] (see Fig. 4(d)) and hence to result in a continuously direct contact between the DLC film and the ceramic ball, thus higher friction coefficient and wear rate of the DLC film are observed.

The oxidation of the DLC film in the presence of H_2O and O_2 during friction testing also has effect on the friction and wear behavior of the DLC film. With the increase of the relative humidity in air, the oxidation of the DLC film became increasingly severe and the DLC film experienced a surface-chemical-state change from C–H bonds to oxygen-containing groups (see Figs. 6 and 7). Subsequently, during the friction process, the bonding strength increased from about 0.08 eV per bond for the Van der Waals bonding of hydrocarbons to about 0.21 eV per bond for the hydrogen bonding at C–O and C=O sites in the presence of H_2O molecules, which resulted in a continuous increase in the friction coefficient and wear rate of the DLC film [3,18].

The thin tribochemical film mainly composed of silica gel and formed by way of the oxidation and hydrolysis of the Si_3N_4 ball due to friction-induced temperature-rise on the sliding surface (see Fig. 8) has also an effect on the friction and wear behavior of DLC film. On one hand, the tribochemical reactions of the Si_3N_4 ball acted to prevent the formation of the transferred carbon-rich layer thereon, which led to increased friction coefficient of the DLC film and wear of the Si_3N_4 ball. On the other hand, a thin hydrated film of silicic acid formed on the sliding surfaces of both DLC film and ceramic ball could act as a “lubricant” of a low shear strength and hence resulted in a friction coefficient 0.090 at a higher relative humidity, which was much smaller than 0.2, that reported elsewhere under a high relative humidity [19,31].

In nitrogen environment, the friction coefficient is always smaller than that in air under the same relative humidity. The nitrogen molecules are expected to hinder the access of active species like water and oxygen molecules to the counterface and prevent or decelerate the friction-induced tribochemical oxidation of the DLC films, thus to decrease the friction coefficient [32,33]. The DLC film experienced mass-transfer of carbon to the Si_3N_4 ball during the sliding in nitrogen (see Fig. 3(d)),

which caused a much larger wear rate and hence the early failure of the DLC film. Yet it has not been known why the DLC film is liable to mass-transfer carbon to the Si_3N_4 ball in a dry nitrogen environment. As the relative humidity increases to an intermediate value (about 40%) in nitrogen, water molecules may passivate the freshly formed dangling bonds on the sliding surface [14] and prevent the mass-transfer of carbon from the DLC film to the Si_3N_4 ball. Therefore, the wear rate of the DLC film decreases sharply to a minimum value and the wear track of the film retains smooth after tested at a lower relative humidity (RH ~ 40%) in nitrogen.

Andersson et al. [34] investigated the friction behavior of a DLC film under varying water vapor pressure and pointed out that water molecules adsorbed to the friction surface could cause the dipole-like interaction, increasing the adhesive forces and hence friction coefficient. In addition, more water would initiate a greater viscous drag and capillary forces. This could account for the linear increase of the friction coefficient with increasing relative humidity in our work in some sense.

4. Conclusions

The friction and wear behavior of the DLC film deposited on a Si (100) wafer, making use of PECVD technique with CH_4 plus Ar as the feedstock, is highly dependent on the relative humidity. The DLC film sliding against Si_3N_4 in a ball-on-disk configuration registers continuously increased friction coefficient and wear rate with increasing relative humidity in air. It records linearly increased friction coefficient with increasing relative humidity in nitrogen, while its wear rate sharply decreases and reaches the minimum at a relative humidity of 40%, followed by an increase with further increase of the relative humidity above 40% in this case. A transferred carbon-rich layer is formed on the counterpart Si_3N_4 ball surface, along with the tribochemical reactions involving the frictional pair materials and the environmental H_2O and O_2 . The oxidation and hydrolysis of the Si_3N_4 ball in higher relative humidity leads to the formation of a tribochemical film mainly composed of silica gel on the sliding surface, which, together with the transferred carbon-rich layer and the friction-induced oxidation of the DLC film at an increased relative humidity, accounts for the environment dependence of the tribological behavior of the DLC film and its variation therewith.

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