

# Preparation and Tribological Studies of C<sub>60</sub> Thin Film Chemisorbed on a Functional Polymer Surface

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A novel self-assembled C<sub>60</sub> film was prepared by chemical adsorption of C<sub>60</sub> molecules onto an amino-group-containing polyethyleneimine-coated silicon substrate surface. The contact angle of distilled water on the C<sub>60</sub> film was measured, the thickness was determined by means of ellipsometric analysis, and the morphology was observed with an atomic force microscope. The tribological properties of the films were investigated as well. It was found that the C<sub>60</sub> thin film had a contact angle of about 72° and thickness of 1.8 nm and exhibited a surface domain microstructure composed of fullerene clusters. Due to the hydrophobicity and low surface energy, the C<sub>60</sub> film possessed good adhesive resistance and had an adhesive force of about 7.1 nN, which was about an order of magnitude lower than that of the silicon substrate surface. Moreover, the C<sub>60</sub> film showed good friction reduction, load-carrying capacity, and antiwear ability, which were attributed to the higher mechanical stiffness and elastic modulus of C<sub>60</sub> molecules. Besides, the friction coefficient decreased with increasing sliding velocity and normal loads, due to the rolling effect of the physisorbed C<sub>60</sub> molecules.

## 1. Introduction

With low surface energy, high chemical stability, spherical shape, weak intermolecular bonding, and high load bearing capacity, buckminsterfullerene (C<sub>60</sub>) has attracted much attention in the application of reducing friction and adhesion since its discovery in 1985.<sup>1,2</sup> It was speculated that C<sub>60</sub> particulates and ultrathin films could be super solid lubricants, since the C<sub>60</sub> molecules might act as mimic ball bearings. However, lots of tribological studies conducted on the C<sub>60</sub> films deposited on lattice substrates cast doubt on such a supposition.<sup>3–7</sup> Schewars and co-workers investigated the load dependence of the friction behavior of carbon compounds including graphite, diamond, amorphous carbon, and C<sub>60</sub><sup>8</sup> and found that graphite showed very low microscopic friction, amorphous carbon and diamond had medium friction, and the C<sub>60</sub> thin film showed the highest friction. This is limiting the tribological application of C<sub>60</sub> thin films. Fortunately, the tribological properties of C<sub>60</sub> film could be improved by surface modification such as ion implantation. For example, the friction coefficient was lowered while the wear resistance was increased when C<sub>60</sub> film was irradiated with Ar or N ions.<sup>8,9</sup> Moreover, C<sub>60</sub> might find promising application in tribology as a novel lubricant additive.<sup>10–14</sup>

The Langmuir–Blodgett (LB) technique<sup>15,16</sup> and self-assembly method<sup>17–21</sup> are known as the novel alternative approaches of preparing C<sub>60</sub> films with good tribological properties. Zhang and co-workers<sup>15</sup> investigated the microfrictional behavior of various kinds of C<sub>60</sub> LB films. They reported that the C<sub>60</sub> LB film, in which the C<sub>60</sub> molecules were combined with long-chain fatty acids by van der Waals forces, possessed lower friction force than the C<sub>60</sub>-immobilized one due to the “rolling effect” of C<sub>60</sub> molecules. Tsukruk and his colleagues compared the frictional behaviors of self-assembled monolayers (SAMs) of C<sub>60</sub> with other SAMs possessing different terminal groups and found that the friction coefficient of C<sub>60</sub> SAMs was a little bigger than that of azide- and methyl-terminated monolayers but was much lower than that of a silicon surface.<sup>19</sup> Similar results were reported by Lander and co-workers, and they further pointed out that the chemisorbed C<sub>60</sub> SAMs possessed higher wear stability than the physisorbed C<sub>60</sub> films.<sup>20</sup> Huang and his colleagues<sup>22–25</sup> studied the microtribological behaviors of

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self-assembled C<sub>60</sub>-containing polymer films and found that, by incorporating a soft polymer chain to form a ternary film, the composite C<sub>60</sub> film showed not only good load-bearing but also low friction properties.<sup>22</sup>

Due to the fact that the C<sub>60</sub> molecules could not directly chemisorb onto the silicon surface, the chemisorbed C<sub>60</sub> monolayer film was generally prepared on SAMs with -NH<sub>2</sub> or -N<sub>3</sub> terminal groups.<sup>19,20</sup> To our knowledge, there have been no reports about the C<sub>60</sub> monolayer film chemisorbed on a polymer surface yet. In the present studies, we report a novel C<sub>60</sub> film chemically anchored to the amino-groups of a polymer-coated silicon surface. The adhesion, friction, and wear behaviors of the film were investigated. It was found that the C<sub>60</sub> film exhibited good lubrication, adhesion resistance, and antiwear ability.

## 2. Experimental Section

**Materials.** Polished and cleaned single-crystal silicon (111) wafers (obtained from GRINM Semiconductor Materials Co., Ltd., Beijing, China) with a root-mean-square (rms) roughness of about 0.3 nm over a range of 1  $\mu\text{m} \times 1 \mu\text{m}$  were used as the substrate. An aqueous solution of polyethyleneimine (abbreviated as PEI, MW = 50 000–60 000, mass fraction = 50%) was obtained from ACROS (New Jersey). C<sub>60</sub> particulates (purity, 99.9%) were used as received. Toluene (purity, >99.5%) was used as the solvent after dehydrating.

**Preparation of the C<sub>60</sub>-PEI Film.** The cleaned silicon wafers were hydroxylated by immersing in a piranha solution (a mixture of 7:3 (v/v) 98% H<sub>2</sub>SO<sub>4</sub> and 30% H<sub>2</sub>O<sub>2</sub>) at 90 °C for 30 min. After being hydroxylated and washed with plenty of ultrapure water, the wafers were then placed into a dilute aqueous solution of PEI (0.2 wt %) and held for 15 min. A thin layer of PEI was thus formed on the hydroxylated silicon surface. After being rinsed with ultrapure water and flushed with N<sub>2</sub>, the PEI-coated silicon substrates were put into a dilute solution of C<sub>60</sub> in toluene (concentration,  $1.0 \times 10^{-3}$  M) and kept for 2 days. Finally, the target C<sub>60</sub> film specimens were obtained after ultrasonically cleaning in toluene to get rid of the physisorbates.

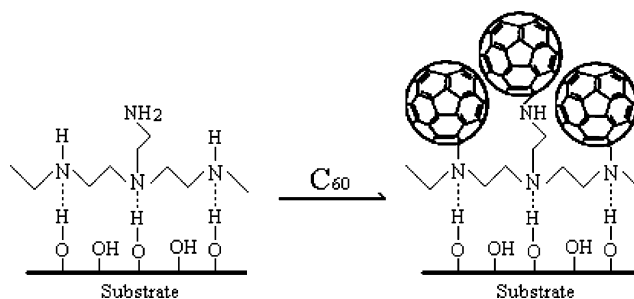
**Characterization of the Film.** Static contact angles for ultrapure water on the films were measured with a Kyowa contact-angle meter. At least five replicate measurements were carried out for each specimen, and the measurement error was  $\pm 1^\circ$ .

The thickness of the films was measured on a Gaertner L116-E ellipsometer, which was equipped with a He-Ne laser (632.8 nm) set at an incident angle of 70°. A real refractive index of 1.46 was set for the silica layer, and 1.45 was assumed for the PEI coating and C<sub>60</sub> monolayer. Five replicate measurements were carried out for each specimen, and the thickness was recorded to an accuracy of 0.3 nm.

A SPA400 (Seiko Instruments Inc.) atomic force microscope (AFM) was used to observe the film morphology, using "Tapping Scanning" mode.

The adhesion force of the film was measured with an AFM/friction force microscope (FFM) controlled by RHK electronics (RHK Technology, Rochester Hills, MI). Commercially available Si<sub>3</sub>N<sub>4</sub> cantilevers/tips with a nominal force constant of 0.5 N/m and tip radius of less than 50 nm (Park Instruments, Sunnyvale, CA) were employed. To obtain the adhesive force between the AFM tip and the film surface, the force-distance curve was recorded and the pull-off force was reckoned as the adhesive force. The tests were conducted at room temperature of about 21 °C and relative humidity (RH) of 65%.

The friction and wear behavior of the film was evaluated on a DF-PM unidirectional tribometer. The lower specimen slid against the upper counterpart ball made of Si<sub>3</sub>N<sub>4</sub> (diameter of 4 mm, roughness rms of about 8.6 nm) at a certain velocity for a distance of 7 mm. Loads of 0.5, 1, and 2 N were selected, and the corresponding initial Hertzian contact stresses were esti-



**Figure 1.** Schematic views of the formation of C<sub>60</sub> film on an amino-terminated PEI-coated silicon surface.

**Table 1. Contact Angle and Thickness for the Silica Layer of the Si Wafer and Two Kinds of Ultrathin Films Thereon**

film	contact angle (deg)	thickness (nm)
SiO <sub>2</sub> /Si	~0	1.9
PEI coating	9	0.7
C <sub>60</sub> thin film	72	1.8

mated to be about 0.7, 0.9, and 1.1 GPa, respectively. The friction coefficient and sliding cycles were recorded automatically. It was assumed that lubrication failure of the film occurred as the friction coefficient rose sharply to a higher and stable value similar to that of a cleaned silicon wafer against the same Si<sub>3</sub>N<sub>4</sub> ball (about 0.66). The number of sliding cycles at this point was recorded as the wear life of the film. All of the tests were conducted at room temperature (25 °C) and a relative humidity of 45%.

## 3. Results and Discussion

**3.1. Formation and Structure of the C<sub>60</sub> Film.** Branched polyethyleneimine  $\{ -[C_2H_5NHC_2H_5N(C_2H_5-NH_2)C_2H_5NH]_n - \}$ , with a distribution of primary, secondary, and tertiary amino-groups in the ratio of 1: 2: 1, can be easily adsorbed onto any hydroxylated solid surfaces through hydrogen bonds and van der Waals forces.<sup>26–29</sup> Since there could be abundant amino-groups exposed on the PEI film surface, the primary and secondary amino-groups could undergo N-H addition reactions across the C=C bonds in C<sub>60</sub> which fused two six-membered rings,<sup>30</sup> and thus amino-terminated PEI thin film was formed on the silicon substrate at the first stage, and then the C<sub>60</sub> film formed by the spontaneous reaction of the PEI film with the C<sub>60</sub> molecules, as shown in Figure 1.

Measurements of contact angle provide a sensitive probe of the chemical composition and order of organic thin films. The contact angles for water on the hydroxylated silicon surface and the target films thereon are shown in Table 1. Obviously, the hydroxylated silicon surface and the PEI coating with the water contact angles of about 0° and 9°, respectively, are hydrophilic. The strong hydrophilicity of the PEI film is attributed to the polar amino-groups. After the PEI-coated Si wafer is immersed in the C<sub>60</sub> solution for 2 days, the contact angle increases to 72°, which is similar to that of the C<sub>60</sub> monolayer film reported elsewhere<sup>17,18,30,31</sup> and reflects that the C<sub>60</sub> thin film has been produced on the PEI coating surface. The thicknesses of the films determined by ellipsometric measurement are also listed in Table 1. After hydroxylating in the piranha

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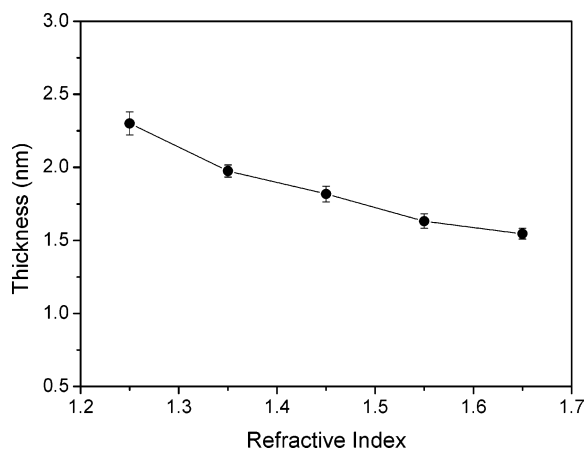
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**Figure 2.** The effect of real refractive index on the measured thickness of C<sub>60</sub> film.

solution at 90 °C for 30 min, a silica layer of about 1.9 nm thick is generated on the silicon wafer. The thickness for the PEI coating is about 0.7 nm, which indicates that the PEI film on the silicon substrate is a monolayer, with the polymer chains laterally sitting on the silicon surface. The thickness of the C<sub>60</sub> monolayer is about 1.8 nm, which is bigger than 1 nm, the reported diameter of C<sub>60</sub> molecules.<sup>3</sup> This implies that aside from the chemisorbed C<sub>60</sub> monolayer, some physisorbed C<sub>60</sub> molecules may also exist on the PEI film surface.

To estimate the accuracy to measure the thickness of the C<sub>60</sub> film with respect to the refractive index 1.45, we make a series of measurements within the refractive index range from 1.25 to 1.65. The results are shown in Figure 2. It can be seen that the thickness of the C<sub>60</sub> film assumes minor decreases with increasing refractive index. Generally, the real refractive index of organic materials is between 1.4 and 1.5. Thus, the refractive index 1.45 used in the ellipsometric measurement, which is also consistent with the datum used in the literature,<sup>17</sup> could well reflect the actual thickness of the C<sub>60</sub> film.

Figure 3 shows the AFM morphologies of the PEI surface and the C<sub>60</sub> film. It can be seen that the PEI surface is quite smooth and has a rms microroughness of about 0.2 nm over a scanning range of 1 μm × 1 μm, reflecting that the PEI molecules homogeneously cover the silicon surface (Figure 3a). Once the C<sub>60</sub> film is produced on the PEI coating, the surface domain microstructure composed of fullerene clusters is observed (Figure 3b). Nevertheless, the surface of the C<sub>60</sub> thin film is still flat on the micrometer scale, with the microroughness of about 0.4 nm, which is a little bit larger than that of the PEI surface. The appearance of the fullerene clusters on the C<sub>60</sub> surface also implies that some C<sub>60</sub> molecules are physisorbed in the C<sub>60</sub> thin film.

### 3.2. Tribological Behavior of the C<sub>60</sub>–PEI Film.

The adhesive forces between the AFM tip and the film surfaces are shown in Figure 4. Strong adhesion is observed on the hydroxylated silicon surface, on which the adhesive force is as high as about 70.4 nN. Once the PEI coating and the C<sub>60</sub> thin film thereon are generated, the adhesive forces are greatly decreased to 17.2 and 7.1 nN, respectively. This indicates that the C<sub>60</sub> thin film has good adhesion resistance. In combination with the contact angle data shown in Table 1, it is seen that the adhesion forces decrease with increasing contact angles, which agrees well with what was reported elsewhere.<sup>19,32,33</sup> On one hand, this is attributed to the effect of the capillary force which, as a main contribution to the adhesion, is closely related to the hydrophilicity of the surface and

would be greatly eliminated as the surface becomes hydrophobic.<sup>34</sup> On the other hand, the low surface energy of the C<sub>60</sub> film also accounts for the low adhesive force. The surface energy of solid crystalline C<sub>60</sub> was reported to be as low as 2.0 ± 0.5 mJ/m<sup>2</sup>, which was much lower than that of a hydrocarbon film (e.g., the surface energy of the film composed of –CH<sub>3</sub> groups was as large as 24 mJ/m<sup>2</sup>).<sup>3</sup>

The friction and wear behaviors of the silicon substrate, PEI coating, and C<sub>60</sub> film thereon are shown in Figure 5. The initial friction coefficient for the PEI film is about 0.22, and it sharply increases to about 0.65 in a few sliding cycles even under a load as low as 0.5 N, which is similar to that of the silicon substrate. This indicates that the PEI coating possesses poor load-carrying capacity and wear resistance. Once the C<sub>60</sub> thin film is formed on the PEI film, the C<sub>60</sub>–PEI film shows good friction-reducing behavior and considerably high wear resistance. It records friction coefficients of about 0.13–0.17, which are similar to what was reported,<sup>2,3,7,19,35</sup> and has antiwear lives above 10 000 cycles, around 3100 cycles, and around 2000 cycles, corresponding to the normal load of 0.5, 1, and 2 N, respectively. We found in our previous study that the SAMs of octadecyltrichlorosilane (OTS) showed a friction coefficient of 0.06 at a normal load of 0.5 N in the initial stage; however, it quickly increased to 0.13–0.14 after tens of sliding cycles, due to the wear of the monolayer. Moreover, the OTS monolayer failed at 1 N.<sup>36</sup> The better wear resistance of the C<sub>60</sub> film than the OTS SAMs might be attributed to the higher mechanical stiffness and elastic modulus of the C<sub>60</sub> molecules than the hydrocarbon chains. Such a supposition is rational if one notices that the C<sub>60</sub> molecule has a stiffness as high as 6 N/m,<sup>37</sup> and the C<sub>60</sub> films have elastic moduli as high as 10–20 GPa,<sup>37–40</sup> while the fluoroalkylsilane and alkylthiol SAMs have elastic moduli of only about 13 and 8 MPa, respectively.<sup>41</sup>

It has been reported that the physisorbed C<sub>60</sub> films prepared by sublimation in a vacuum are liable to wear and transfer onto the counterpart surface during the sliding,<sup>2,20</sup> while the stable C<sub>60</sub> monolayer prepared by the chemisorbing of C<sub>60</sub> molecules onto the SAM-modified surface possessed higher wear stability than the physisorbed C<sub>60</sub> films.<sup>3,19</sup> Since it is infeasible to directly compare the tribological properties of the C<sub>60</sub>–PEI film and the C<sub>60</sub> monolayer on SAMs using different experimental instruments, one could hardly tell which one possesses superior tribological characteristics with respect to the C<sub>60</sub>–PEI film dealt with in the present work and the C<sub>60</sub> monolayer on SAMs reported in the literature. We are now pursuing experiments to compare the tribological properties of C<sub>60</sub> monolayers on polymer surfaces with those on SAMs, and the results will be reported in the future. Although the PEI coating is only physisorbed onto the silicon surface, the PEI molecules containing a large amount of functional amino-groups are capable of forming

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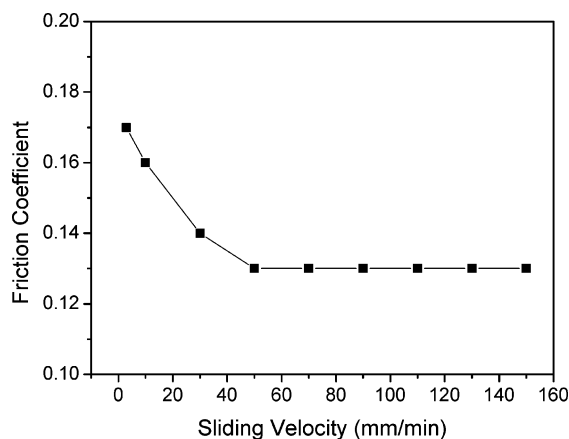
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**Figure 6.** Variation in the friction coefficients with sliding velocities for the C<sub>60</sub> film sliding against a Si<sub>3</sub>N<sub>4</sub> ball at a normal load of 1 N.

surface, the rolling effect of the desorbed physisorbed C<sub>60</sub> molecules may play a more important role in reducing the friction coefficient at larger sliding velocities or normal load. Noticing that the friction increased or initially increased and then decreased with increasing sliding velocities for the “normal” self-assembled films,<sup>19,33,42–44</sup> which was attributed to the growing dissipation of the accumulated energy or the phase transition of the film at higher shearing velocities, we supposed that it could be just the rolling effect of the desorbed physisorbed C<sub>60</sub> molecules that determined the abnormal velocity-dependent behavior of the C<sub>60</sub> film. Such a rolling effect of the C<sub>60</sub> molecules as molecular ball-bearings was also recently

confirmed by Kamiya and his colleagues.<sup>45</sup> They said that the system whose two graphite planes had a monolayer of C<sub>60</sub> sandwiched between them possessed negligible mean frictional forces due to the rolling effect of the C<sub>60</sub> molecules. This adds to our supposition that the C<sub>60</sub> films with properly controlled thickness and frictional mode could show good lubricity by way of the rolling effect of the C<sub>60</sub> molecules.

#### 4. Conclusion

In conclusion, a novel self-assembled C<sub>60</sub> film was prepared by chemical adsorption of C<sub>60</sub> molecules onto the amino-groups of the PEI film coated silicon substrate surface. Due to the hydrophobicity and low surface energy, the C<sub>60</sub> film possessed an adhesive force of about 7.1 nN, which was about an order of magnitude lower than that of the silicon substrate surface. Moreover, it had good friction reduction, load-carrying capacity, and antiwear ability, which were attributed to the higher mechanical stiffness and elastic modulus of C<sub>60</sub> molecules. Due to the rolling effect of the desorbed physisorbed C<sub>60</sub> molecules, the C<sub>60</sub> film showed a decreased friction coefficient at increasing sliding velocity or normal load.

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