

Highly wear resistant chemisorbed polar ultra-high-molecular-weight polyethylene thin film on Si surface for micro-system applications

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(Received 2 March 2009; accepted 12 May 2009)

We report deposition and tribological studies of a chemisorbed UHMWPE (ultra-high-molecular-weight polyethylene) film on an Si surface. UHMWPE molecules containing carboxyl and hydroxyl chemical groups were chemisorbed onto an Si surface using an intermediate GPTMS SAM (glycidoxypropyltrimethoxy silane self-assembled monolayer) layer. The carboxyl and hydroxyl groups of UHMWPE molecules react with the terminal epoxy groups of GPTMS SAM during chemisorption. The resultant film ($\sim 1.4 \mu\text{m}$ thick) has shown low coefficient of friction (~ 0.1) and high wear life (exceeding 100,000 cycles) in a sliding test against a 4 mm diameter Si_3N_4 ball at a normal load of 0.3 N and a sliding velocity of 0.042 m/s measured on a micro-tribometer. In contrast, bare Si or GPTMS SAM modified Si has shown a higher coefficient of friction and failed within a few tens of sliding cycles. The high wear durability of the chemisorbed polymer film is attributed to the excellent adhesion of the UHMWPE film with the substrate due to chemisorption and to the good lubrication properties of UHMWPE molecules. This wear resistant film has potential applications in micro-electro-mechanical systems made of Si.

I. INTRODUCTION

Improvements in the tribological properties of the Si surface are important for applications in micro-electro-mechanical systems (MEMS) or microsystems. Si, without any modification, shows high friction, high adhesive force, and high wear. Organic ultra-thin films [such as self-assembled monolayers (SAMs) or polymer films] have shown good improvement in reducing the coefficient of friction, adhesive forces, and wear.¹⁻⁵ Further developments in improving the lubrication properties of the Si surface (especially wear durability) are needed so that many novel and reliable microcomponents can be developed/produced from Si. In our recent studies, we have identified that thermoplastic polymers which have linear microstructure² and with the addition of nano-filler materials⁶ show excellent tribological properties (low friction and low wear) when they are coated onto Si. Further, UHMWPE films dip-coated onto a Si surface ($\sim 28 \mu\text{m}$ thick) have shown a wear life of $\sim 12,000$ cycles when tested against a 4 mm diameter Si_3N_4 ball at a normal load of 70 mN and at a sliding velocity of 0.042 m/s.⁷ However, two limitations have been identified from that study: (i) the thickness of the polymer film is high ($\sim 28 \mu\text{m}$; especially for some micro-system applications) and (ii) the wear life is still low. Chemisorption of the UHMWPE film is selected to overcome the above

two limitations because chemisorption improves adhesion between the Si surface and the UHMWPE film which eventually helps in improving wear durability. Chemisorption also helps in the control of film thickness as only a monolayer of the polymer is required to be chemically bonded with the substrate or the intermediate layer so that the subsequent physisorbed layers can be easily removed. Therefore, in the present study, we have used UHMWPE powder with the polymer molecules modified with chemical groups such as carboxyl and hydroxyl so that these polymer molecules could be chemisorbed onto Si using an intermediate layer of GPTMS SAM (glycidoxypropyltrimethoxysilane SAM). The chemical bonding between carboxyl groups or hydroxyl groups and epoxy groups has been extensively used in preparing polymer blends with high miscibility (compatibility) and improved mechanical properties (such a reaction between two immiscible polymers produces a copolymer by grafting).^{8,9} The tribological properties as well as other physical, chemical, and mechanical properties of the resultant dual-layer film are investigated. To summarize, in the present study, the effects of the intermediate layer of GPTMS SAM, the presence of the chemical groups in the UHMWPE molecules, and post-thermal treatment after chemisorption of UHMWPE film onto GPTMS SAM on the final tribological properties of the dual-layer film are investigated.

II. EXPERIMENTAL

Polished single crystal Si (100) wafers were used as the substrate. These Si wafers were cut into pieces of

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DOI: 10.1557/JMR.2009.0397

approximately 2 cm × 2 cm and then used for the surface modification. GPTMS [CH₂-O-CH-CH₂-O-(CH₂)₃-Si-(OCH₃)₃, obtained from Sigma Aldrich, Singapore] was used for the preparation of the SAM solution. Surface modified UHMWPE powder was purchased from Sigma Aldrich, Singapore. The bulk density of UHMWPE is 0.94 g/mL at 25 °C and the average particle size of the UHMWPE powder is 53–75 μm. Toluene (99.5%, anhydrous), methanol (99.8%), decahydronaphthalin (decalin), and distilled water were also used during sample preparation.

The cleaning and piranha treatment of Si have been reported elsewhere.¹ After the piranha treatment, GPTMS deposition was carried out according to the procedure reported in Satyanarayana et al.¹⁰ The GPTMS SAM modified Si samples are then dip-coated using a solution of UHMWPE in decalin. The polymer solution was prepared by dissolving the UHMWPE powder in decalin by heating first at 150 °C for ½ h followed by heating at 250 °C for another ½ h. A concentration of 1 wt%, dipping time of 1 min and a withdrawal speed of 2.1 mm/s were used for the formation of chemisorbed UHMWPE films. The UHMWPE film formed under the above conditions has a thickness of ~1.4 μm, which was measured using field emission scanning electron microscopy (FESEM) observation of cross sections of cut Si samples with the polymer film. At least five independent measurements were taken for each sample and the average of three samples is reported here. The variations in the thickness across the length were within ~0.10 μm.

The static contact angles for distilled water on the unmodified and modified surfaces were measured using the VCA Optima Contact Angle System (AST Products, Inc., Billerica, MA). A water droplet of 0.5 to 1 μL was used for contact angle measurement. At least 5 replicate measurements, for three different samples, were carried out and an average value was taken. The variation in water contact angle values at various locations of a sample was within ±2°. The measurement error was within ±1°.

The surface morphology of unmodified and modified Si substrates was investigated using a Dimension 3000 Atomic Force Microscope (AFM, Veeco Metrology Inc., Santa Barbara, CA). All images were collected in air using the tapping mode and a silicon tip. The set point voltage used was 1–2 V and the scan rate was 1 Hz.

Friction and wear tests were carried out on a ball-on-disk tribometer (Universal Micro Tribometer, CETR, Campbell, CA). An Si₃N₄ ball of 4 mm diameter was used as the counterface. Sliding tests were conducted at normal loads of 50 mN, 0.3 N, 0.5 N, 0.8 N, and 2 N, at sliding velocities of 0.021 m/s, 0.042 m/s, 0.084 m/s, and 0.168 m/s. The roughness of the ball used was 5 nm, as provided by the supplier. The ball was cleaned ultrasonically with acetone before each test. Every ball was viewed under an optical microscope before a test to

ensure that it was free from surface contaminants and manufacturing defects. For each test, a new ball was used. All experiments were performed in air at room temperature (23 °C) and at a relative humidity of approximately 70%. The wear life was defined as the number of cycles after which the coefficient of friction exceeded a value of 0.3 or a visible wear scar appeared on the substrate, whichever happened earlier. The wear life data were obtained on at least five different samples utilizing at least two different tracks on each sample and an average of the three best results is reported. The morphology of the worn surfaces was studied using a scanning electron microscope and AFM.

The elastic modulus and hardness of the thin films were measured by nanoindentation using an MTS Nano Indenter[®] XP (MTS Corporation, Nano Instruments Innovation Center, Eden Prairie, MN) with a continuous stiffness measurement (CSM) technique. CSM allows hardness and modulus to be determined as functions of indentation penetration depth with a single indentation load/unload cycle.^{11,12} A triangular pyramid (Berkovich) diamond indenter was used for all nanoindentation tests. The depth of indentation was set to 2000 nm. MTS Nano Indenter XP has load and displacement resolutions of 50 nN and <0.01 nm, respectively. In all CSM tests, a total of 10 indents on different random surface locations were performed and averaged to determine the mean hardness and elastic modulus values.

III. RESULTS AND DISCUSSION

Table I shows the water contact angles of various surfaces studied. The dual-layer film has a water contact angle of 109° whereas GPTMS SAM or Si alone have values of 52° and 12°, respectively. The water contact angle of GPTMS SAM corresponds to that reported in the literature.¹³ The pristine bulk UHMWPE surface usually shows a water contact angle of 87–90°. ^{14,15} Therefore, the water contact angle of the present chemisorbed UHMWPE film is approximately 10° higher than that corresponding to the pristine bulk UHMWPE surface. The hydrophobic nature of the chemisorbed UHMWPE films supports the absence of polar chemical groups on the film surface (the presence of polar chemical groups usually shows low water contact angle¹⁶) and further supports the good covalent bonding between the UHMWPE and GPTMS molecules. The large difference in water contact angles between the pristine bulk UHMWPE surface and the presently formed chemisorbed UHMWPE film is attributed to the differences in their surface condition (primarily surface roughness) and their grades (differences in density, etc.).

Figure 1 shows the SEM image and the AFM topography of the chemisorbed UHMWPE film. The SEM image shows a fibrous structure with plenty of protruding

TABLE I. Water contact angle, coefficient of friction, and wear life of all modifications studied in the present work.

Sample	Water contact angle (°)	Coefficient of friction	Wear life (number of cycles)
Bare Si	12	0.48	<100
Si/GPTMS	52	0.46	<100
Si/GPTMS/polar UHMWPE	109	0.1	>100,000
Si/polar UHMWPE	—	—	3,500
Si/GPTMS/UHMWPE	—	—	13,000
Si/GPTMS/polar UHMWPE; no post thermal treatment	—	—	6,000

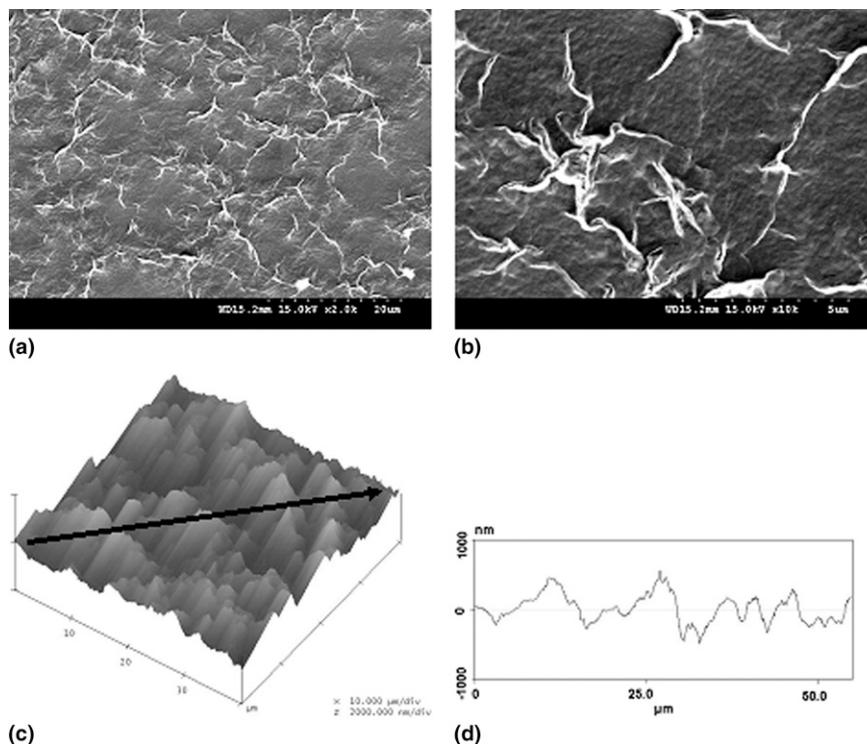


FIG. 1. SEM image of Si/GPTMS/polar UHMWPE at (a) low magnification (2,000 \times) and (b) high magnification (10,000 \times). (c) AFM topography of the Si/GPTMS/polar UHMWPE. The scan area for AFM imaging is 40 $\mu\text{m} \times$ 40 μm and the vertical scale is 2000 nm. RMS roughness over a scan area of 40 $\mu\text{m} \times$ 40 μm is \sim 216 nm. (d) Line profile obtained from AFM image in (c) (in the direction indicated by the arrow).

islands of the polymer distributed nonuniformly all over the surface which are also seen in the AFM image (over a scan area of 40 $\mu\text{m} \times$ 40 μm) with deep valleys. The depths of the valleys are in the range of 300–700 nm. The AFM image over a scan area of 40 $\mu\text{m} \times$ 40 μm showed an RMS roughness of \sim 216 nm. The textured features as shown by the dual-layer film in AFM imaging are expected to improve the tribological properties; especially in reducing the coefficient of friction as the textured features reduce the actual contact area during sliding.

CSM nanoindentation tests on chemisorbed UHMWPE film (\sim 1.4 μm thick) have an elastic modulus of \sim 3.78 GPa and a hardness value of \sim 0.07 GPa. The non-chemisorbed UHMWPE film on Si (\sim 28 μm thick) has an elastic modulus of \sim 3.7 GPa and a hardness of \sim 0.12 GPa.¹⁷ Bulk UHMWPE has elastic modulus and hardness values of 1–2 GPa and 0.04–0.06 GPa, respectively, measured using the nanoindentation technique.^{18,19}

Therefore, the present chemisorbed UHMWPE film on Si has shown similar nanomechanical properties when compared to the non-chemisorbed UHMWPE film on Si and bulk UHMWPE.

As shown in Table I, the dual layer film has a coefficient of friction of 0.1 (measured after 12 cycles of sliding) and a wear life of more than 100,000 cycles. Both bare Si and GPTMS SAM surfaces have coefficients of friction higher than 0.45 and wear lives of only a few tens of cycles (<100 cycles). Figure 2 shows the typical coefficient of friction versus the number of cycle curves for bare Si, GPTMS SAM, and the chemisorbed UHMWPE film. Figures 3(a), 3(b), and 3(c) show SEM images of the wear tracks for bare Si, Si/GPTMS, and Si/GPTMS/polar UHMWPE films, respectively, after an appropriate number of sliding cycles. The bare Si and GPTMS SAM surfaces had severe wear within a few tens of sliding cycles. The SEM images [Figs. 3(a) and

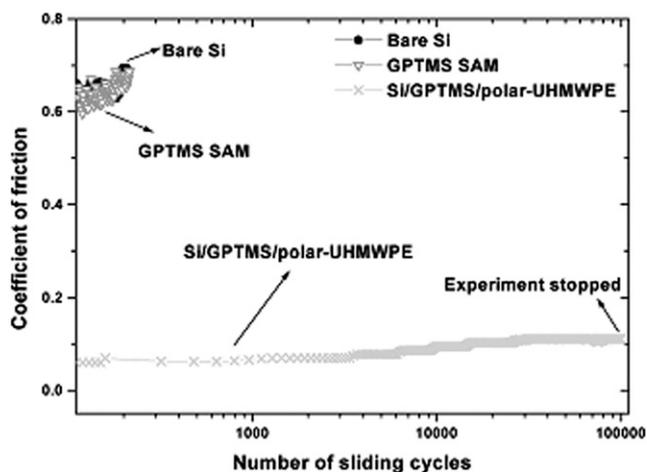


FIG. 2. Coefficient of friction versus number of cycles for bare Si, Si/GPTMS, and Si/GPTMS/polar UHMWPE samples, tested against a 4 mm diameter Si_3N_4 ball at a normal load of 0.3 N and a sliding velocity of 0.042 m/s.

3(b)) were taken after 500 cycles after running the test more than the failed life to see the extent of the damage. The Si/GPTMS/polar UHMWPE sample did not show any sign of wear or the generation of wear debris even after sliding for 100,000 cycles. EDS (energy dispersive spectroscopy) analysis in the regions outside and inside the wear track of Si/GPTMS/polar UHMWPE is shown in Figs. 3(d) and 3(e), respectively. The EDS spectra shown in Figs. 3(d) and 3(e) are almost identical except that the intensity of the C peak inside the wear track [Fig. 3(c)] is slightly less than that outside the wear track [Fig. 3(d)]. The slight reduction in the intensity of C peak inside the wear track must be because of the compression and spreading of all protruding molecules on the surface due to contact sliding. The Si peak appears even in the case of the coated and nontested region because of the small thickness of the polar UHMWPE film (eventually lower mass of polymer molecules) which cannot shield the appearance of the Si peak during EDS analysis. The EDS analyses of the regions inside and outside the wear track of Si/GPTMS/polar UHMWPE show no or negligible wear to the polymer film even after sliding for 100,000 cycles at a normal load of 0.3 N. The AFM image [Fig. 3(f)] inside the wear track of Si/GPTMS/polar UHMWPE after sliding for 100,000 cycles shows a smooth surface (with a surface roughness of ~ 20 nm in a scan area of $40 \mu\text{m} \times 40 \mu\text{m}$) without any debris.

To determine the effect of the presence of the intermediate SAM on the final tribological properties, tests were conducted by dip-coating the polar UHMWPE onto Si without the intermediate GPTMS SAM. The Si/polar UHMWPE sample has a wear life of only $\sim 3,500$ cycles whereas Si/GPTMS/polar UHMWPE has a wear life of more than 100,000 cycles when tested at a normal load of 0.3 N and a sliding velocity of 0.042 m/s. These

results support that the exceptionally high wear durability of the dual-layer film is because of the good adherence of UHMWPE layer through chemical bonding with the intermediate GPTMS SAM layer and indicates the importance of the chemisorption of the polymer film on the wear durability.

Further, the effect of post-heating on the final tribological properties was also studied. Si/GPTMS/polar UHMWPE post-heated at $\sim 110^\circ\text{C}$ has a wear life of more than 100,000 cycles whereas Si/GPTMS/polar UHMWPE dried at room temperature has a wear life of only $\sim 6,000$ cycles when tested at a normal load of 0.3 N and a sliding velocity of 0.042 m/s. A possible reason for this behavior could be that the strong covalent bonding between carboxyl or hydroxyl and epoxy groups requires thermal energy and hence the polymer film that was post-heated at 110°C has demonstrated good adhesion with the substrate and eventually high wear life; whereas the polymer film that was not subjected to post-heating failed earlier because of the weaker chemical interactions between the polymer film and the substrate. These results further demonstrate the importance of chemical bonding of the polymer molecules with the substrate through the interface layer (GPTMS) in enhancing wear life.

To understand the effect of the presence of chemical groups on the surface of UHMWPE powder on the tribological properties, a comparison was made between the UHMWPE films formed from the powders with and without the presence of chemical groups. UHMWPE film (physisorbed without any chemical groups) of $6 \pm 2 \mu\text{m}$ thickness was formed by dip-coating in a 3 wt% polymer solution in decalin and tribological tests were conducted under similar conditions. The sliding tests against a 4 mm diameter Si_3N_4 ball at a normal load of 0.3 N and at a sliding velocity of 0.042 m/s have shown that Si/GPTMS/polar UHMWPE (chemisorbed) has a wear life of more than 100,000 cycles, whereas Si/GPTMS/UHMWPE (physisorbed sample) has a wear life of only $\sim 13,000$ cycles. These data clearly support the importance of the presence of reactive chemical groups on the polymer molecules. It is evident that the application of an intermediate SAM layer with the polar UHMWPE greatly enhances adhesion and wear resistance.

Figure 4 shows the results of the effects of normal load and sliding velocity on the coefficient of friction and wear lives of Si/GPTMS/polar UHMWPE samples. Figure 4(a) shows that the coefficient of friction of the dual-layer film is nearly invariant with respect to the applied normal load, whereas Fig. 4(b) shows that the coefficient of friction gradually increases (coefficient of friction doubles from 0.07 to 0.14 with an increase in the sliding velocity from 0.021 m/s to 0.168 m/s) with respect to sliding velocity. The wear durability data [Figs. 4(c) and 4(d)] show that the presently formed film withstands a normal load of 0.5 N and a sliding

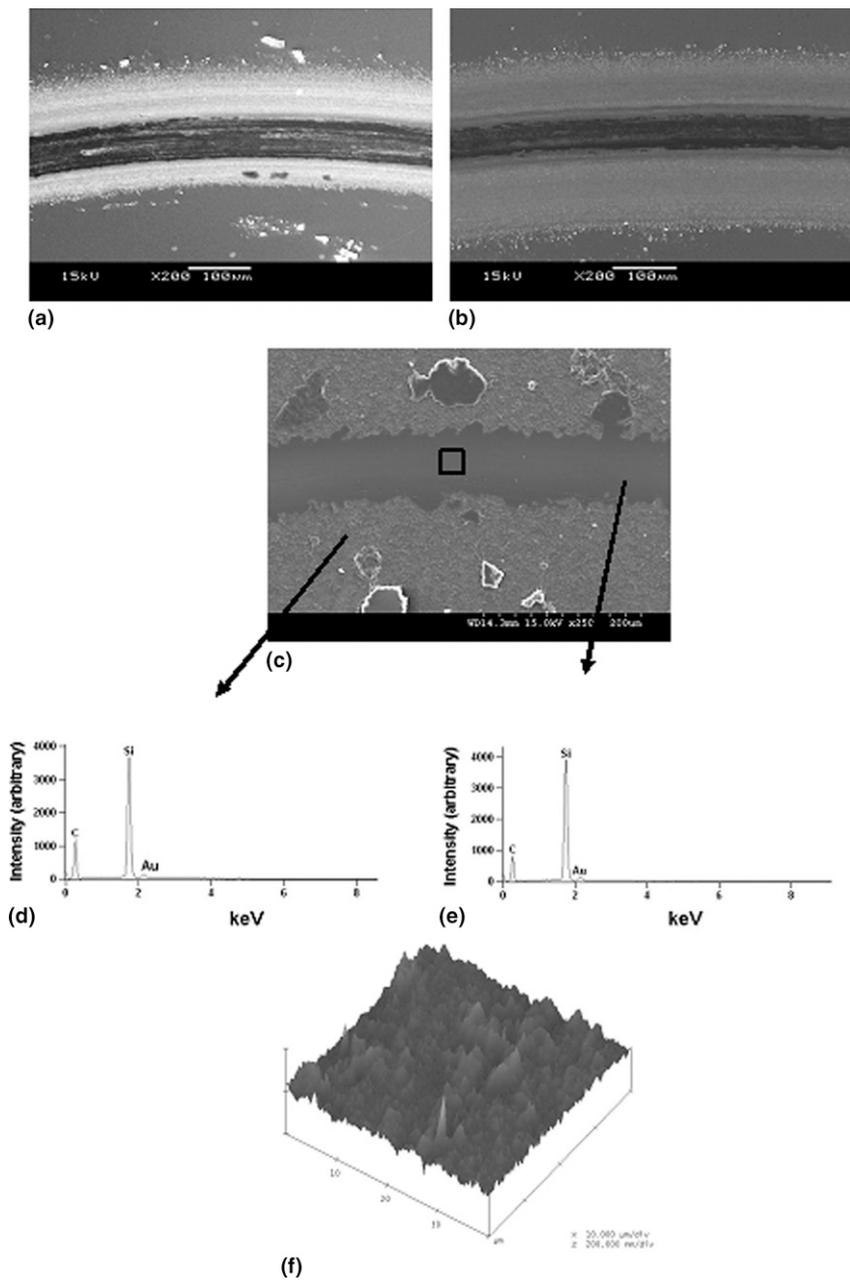


FIG. 3. SEM morphology of worn surfaces after an appropriate number of sliding cycles. (a) Bare Si, run for 500 cycles; (b) Si/GPTMS, run for 500 cycles; and (c) Si/GPTMS/polar UHMWPE, run for 100,000 cycles. Bare Si and Si/GPTMS were tested at a normal load of 50 mN and a sliding velocity of 0.021 m/s, whereas Si/GPTMS/polar UHMWPE was tested at a normal load of 0.3 N and a sliding velocity of 0.042 m/s. (d) EDS spectrum outside the wear track and (e) EDS spectrum inside the wear track for Si/GPTMS/polar UHMWPE as shown in (c). (f) AFM topography inside the wear track of Si/GPTMS/polar UHMWPE as shown by the rectangular box in (c). The scan area is $40\ \mu\text{m} \times 40\ \mu\text{m}$ and the vertical scale is 200 nm. The RMS roughness over a scan area of $40\ \mu\text{m} \times 40\ \mu\text{m}$ is ~ 20 nm. Note that the patches of material seen in (c) outside the wear track are not wear debris but a result of the coating procedure. Their presence in the film does not affect wear life in any way.

velocity of 0.084 m/s for more than 100,000 sliding cycles without any wear. Beyond these optimized normal load and the sliding velocity values, the wear of the UHMWPE film may begin. The maximum limiting PV value (multiplication product of the contact pressure and the velocity) that the presently chemisorbed UHMWPE film withstood was calculated to be ~ 6.56 MPa m/s (the contact pressure was calculated using the Hertzian con-

tact equation; the PV value was calculated using a normal load of 0.5 N and a sliding velocity of 0.084 m/s because the polymer film did not fail for these sliding conditions). In the calculation of the Hertzian contact pressure, we used the elastic modulus value of 3.78 GPa (obtained from the nanoindentation test) and a Poisson's ratio of 0.4 for the chemisorbed UHMWPE film (similar to bulk UHMWPE).²⁰ Further, we also calculated the

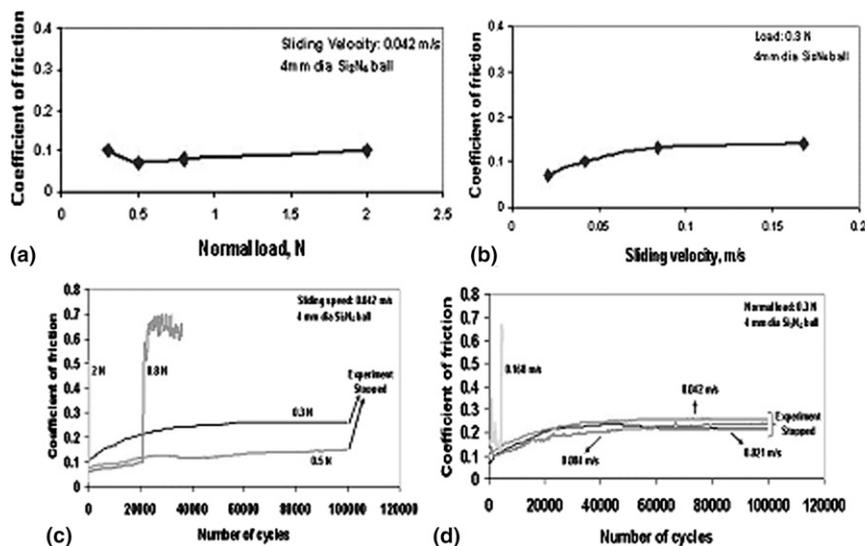


FIG. 4. (a) Effect of the normal load on the coefficient of friction and (b) effect of the sliding velocity on the coefficient of friction of Si/GPTMS/polar UHMWPE samples. (c) Effect of the normal load on the wear lives and (d) effect of the sliding velocity on the wear lives of Si/GPTMS/polar UHMWPE samples. All tests were conducted against a 4 mm diameter Si_3N_4 ball.

PV value from the measured nominal contact pressure. The nominal contact pressure was calculated using the expression: load divided by the contact area (πa^2),²¹ where a is the ball-film contact radius which was measured using FESEM as the half width of the wear track. The PV value calculated using this method was in the range of 4–9 MPa m/s, which is close to those values measured using the Hertzian contact model as shown above. The tribological results at different normal loads and sliding velocities gave some information of the safe working region for the presently demonstrated polymer film as a load bearing coating material.

IV. CONCLUSIONS

In the present study, we have formed chemisorbed ultra-thin UHMWPE film on Si using an intermediate self-assembled monolayer of GPTMS. Tribological properties such as the coefficient of friction and wear life of the resulting film are investigated. The effects of the presence of the intermediate SAM, the presence of the polar chemical groups (carboxyl and hydroxyl) on UHMWPE molecules, post-heating, normal load, and sliding velocity on the coefficient of friction and wear life data are also evaluated. The following conclusions are drawn from the present study:

(1) The chemisorbed polar UHMWPE film has a high water contact angle (109°). The AFM morphology shows the formation of irregular islands with deeper valleys (depths in the range of 300–700 nm) on the polymer film surface.

(2) The chemisorbed polar UHMWPE film of 1.4 μm thickness has a coefficient of friction of 0.1 and a wear life of more than 100,000 cycles in the sliding tests at a

normal load of 0.3 N and a sliding velocity of 0.042 m/s against 4 mm diameter Si_3N_4 balls.

(3) The presence of the intermediate polar SAM layer, the polar chemical groups on polymer molecules, and post-thermal treatment after dip-coating greatly helps in improving the tribological properties, especially the wear durability. This is because they all help in forming strong covalent bonds between the polymer molecules and the SAM layer. The GPTMS monolayer is itself bonded chemically to the Si surface through covalent Si-O-Si bonds.

(4) Neither normal load nor sliding velocity shows any major effect on the initial coefficient of friction of Si/GPTMS/polar UHMWPE but they have significant effects on the wear life. The wear life was more than 100,000 sliding cycles and invariant until a normal load of 0.5 N and then decreases with the increase in the normal load beyond 0.5 N. The wear life was more than 100,000 sliding cycles and invariant until a sliding velocity of 0.084 m/s and decreases with an increase in the sliding velocity beyond 0.084 m/s. The maximum allowable PV factor was found to be ~ 6.56 MPa m/s.

ACKNOWLEDGMENTS

The authors would like to acknowledge the help of Mr. Yan Yuxiang for conducting some initial experiments. The authors also wish to acknowledge the financial support given to this work by the National Research Foundation (NRF), Singapore (Award No. NRF-CRP 2-2007-04).

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