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ORIGINAL PAPER

Carbon Nanotube Reinforced Polyimide Thin-film for High Wear Durability

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Abstract In this paper, the influence of single walled carbon nano tubes (SWCNTs) addition on the tribological properties of the polyimide (PI) films on silicon substrate was studied. PI films, with and without SWCNTs, were spin coated onto the Si surface. Coefficient of friction and wear durability were characterized using a ball-on-disk tribometer by employing a 4 mm diameter Si_3N_4 ball sliding against the film, at a contact pressure of ~370 MPa, and a sliding velocity of 0.042 ms^{-1}. Water contact angle, AFM topography, and nano-indentation tests were conducted to study the physical and mechanical properties of the films. SWCNTs marginally increased the water contact angle of PI film. The addition of SWCNTs to PI has increased the hardness and elastic modulus of pristine PI films by 60–70%. The coefficient of friction of PI films increased slightly (~20%) after the addition of SWCNTs, whereas, there was at least two-fold increase in the wear life of the film based on the film failure condition of coefficient of friction higher than 0.3. However, the film did not show any sign of wear even after 100,000 cycles of rotation indicating its robustness. This increase in the wear durability due to the addition of the SWCNTs is believed to be because of the improvement in the load-bearing capacity of the composite film and sliding induced microstructural changes of the composite film.

Keywords Polyimide film · SWCNTs · Tribology · Wear durability · Nano-indentation

Introduction

Polymer thin films are effective in improving tribological properties of various substrates such as Si [1–3], steel [4] etc. Apart from tribological applications, polymer thin films are also known for their better corrosion protection applications [5]. Polyimide (PI) thin films possess superior physiochemical properties such as high thermal stability, high chemical resistance, good mechanical strength, low dielectric constant, and reasonable tribological properties [2, 6–8]. In recent years there has been a great effort on improving the tribological properties of PI thin films; for example, the surface modification of PI films by ion implantation has shown good improvement in reducing the friction and wear of unmodified PI films on Si substrate [9]. Further developments in this respect are needed to enhance the tribological properties of the PI thin films.

Carbon nano tubes (CNTs) continue to be a subject of unabated scientific research and development, since their discovery in 1991 [10]. CNTs have been considered as a possible reinforcement material because of their exceptional mechanical properties, chemical stability, and tribological properties [11–13]. CNTs are effectively used to make the bulk polymer composites and have shown good improvements in tribological properties [14, 15] as well as mechanical [16] and electrical properties [17]. Zoo et al. [14] have observed that the addition of multi-walled CNTs (MWCNTs) (0.5 wt%) to ultra-high-molecular weight polyethylene (UHMWPE) have reduced the wear loss from 0.35 to 0.05 g. Cai et al. [15] reported that the addition of 30 wt% CNT to PI reduced the wear loss from 4.5 to

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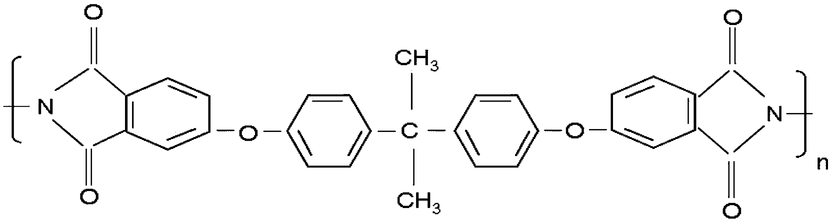
2.5 mm^3. But, only few studies involved the use of CNTs addition to the polymer/organic thin films to enhance their tribological properties [18] and this area needs further research. Pavoor et al. [18] have demonstrated that the addition of MWCNTs to polyelectrolyte multilayers (PEMs) on Si has greatly enhanced the wear resistance of pristine PEMs.

The primary objective of the present study is to investigate the influence of the addition of single walled CNTs (SWCNTs) to the PI film on Si on tribological properties especially wear life. There is no reported literature on the friction and wear behavior of PI film containing SWCNT. The present study is very important in view of many advanced technological applications (in the areas such as electrical and electronic, biological, nanotechnology etc) of carbon nanotube/polymer composites [19].

Experimental

Materials

Polished single crystal silicon (100) wafers were used as the substrate. The Si wafer was cut into pieces of approximately 2 cm × 2 cm and then used for the surface modification. Polyamic acid (PAA) (obtained from Hitachi Chemicals Asia Pvt Ltd, Singapore) was used for the preparation of the PI film on the Si surface. The chemical structure of the PI is shown below:



SWCNTs were used as the filler material for PI. The SWCNTs were obtained from Iljin Nanotech Co., Ltd, Korea, which were as processed grade and were produced using arc-discharge process. The SWCNTs were used without any further treatment for the preparation of the composite polymer solution.

N-methyl 1, 2- pyrrolidone (NMP) was used as the solvent for the preparation of the PI films, with and without the addition of SWCNTs.

Synthesis of polymer thin films

Prior to the deposition of polymer films on Si surface, Si was cleaned according to the reported procedure [20]. The samples were immersed in a piranha solution (a mixture of 7:3 (v/v) 98% H_2SO_4 and 30% H_2O_2) at 60–70°C for 1 h to hydroxylate and to remove any organic/inorganic contaminants. The root mean square roughness of the Si wafers, after piranha treatment, was 0.3–0.5 nm, which was measured using atomic force microscopy (AFM). The piranha treated Si samples were then subjected to polymer film coatings.

The solution of PAA in NMP at a ratio of 3:1 by weight was subjected to homogenization using a motorized homogenizer for ~2.5 h at a speed of 10,000 rpm, with an attached facility of continuous cooling. This solution was used to coat the polymer film onto the piranha treated Si surface using spin coating. The spin coating was carried out at a speed of 600 rpm for a period of 60 s. The samples were then heated in a vacuum oven successively for 12 h at 50°C, 1 h at 100°C, 1 h at 200°C, and finally for 1 h at 300°C, and slowly cooled to room temperature to complete the imidization of PAA [21].

For the preparation of the composite, mixture of SWCNTs and NMP was subjected to ultra-sonication for 15 min to ensure uniform dispersion without any agglomeration of SWCNTs. Subsequently, PAA was added to this NMP-SWCNTs mixture and subjected to the homogenization as explained previously. The concentration of SWCNTs used was 0.05% by weight. The coated and baked samples were then stored in a desiccator until further characterization or testing.

Depositional characterization and surface analysis

The static contact angles for distilled water on the unmodified and modified surfaces were measured with VCA Optima Contact Angle System (AST Products, Inc. USA). A water droplet of 0.5–1 μl was used for contact angle measurements. At least 5–6 replicate measurements, for three different samples, were carried out, and an average value was reported. The variation in water contact angle values at various locations of a sample was within ±2°. The measurement error was within ±1°.

Laser profilometer was used to measure the thickness of the polymer film as per reported practice [18, 22]. For this measurement, only 50% of the Si surface was coated with PI film and same sample was subjected to thickness measurement.

The surface topography of modified and unmodified surfaces was studied using Dimension 3000 AFM (Atomic force microscope) (Digital Instruments, USA). Images were collected in air using a silicon tip in tapping mode. A set point voltage of 1–2 V and a scan rate of 1 Hz were used for scanning.

The surface features of the polymer coated Si and the worn regions, after tribological tests, were observed using JEOL JSM 5600 HV scanning electron microscope (SEM) attached with Energy dispersive spectroscopy (EDS). Field Emission (FE)-SEM was used to observe the SWCNTs used in the present study. Figure 1 shows the FE-SEM image of the SWCNTs used in the present study.

Nano-mechanical property characterization using Nanoindentation

The elastic modulus and hardness of the thin films were measured by nanoindentation using MTS Nano Indenter® XP (MTS Corporation, Nano Instruments Innovation Center, TN, USA) 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 [23]. A triangular pyramid (Berkovich) diamond indenter was employed for all nanoindentation tests. The depth of indentation was set to 2,000 nm. The CSM technique has a load and displacement resolutions of 50 nN and <0.01 nm, respectively. In all CSM tests, a total of five indents on different random surface locations were averaged to determine the mean hardness and elastic modulus values.

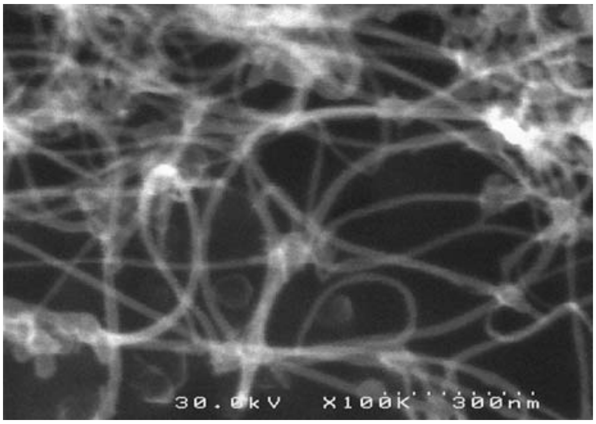


Fig. 1 FE-SEM image of the SWCNTs used in the present study, which were physically spread on a carbon tape to facilitate SEM imaging. The diameter of the SWCNTs is ~10 nm

Tribological characterization

Friction and wear tests were carried out on UMT-2 (Universal Micro Tribometer, CETR, USA), using ball-on-disk mode. A Si_3N_4 ball of 4 mm diameter was used as the counterface. The rotational speed of spindle was 200 rpm giving a linear sliding speed of 0.042 ms^{-1} at a track diameter of 4 mm. The roughness of the ball used was 20 nm, as provided by the supplier. Every ball was cleaned ultrasonically with acetone and ensured free from contaminants or manufacturing defects before the test. For each test, a new ball was employed. The normal load used was 7 g, which gave a contact pressure of approximately 370 MPa (calculated using the Hertzian Contact Model). All experiments were performed in air at room temperature (23°C) and at a relative humidity of approximately 70%. In this paper, the initial coefficient of friction was reported after 4 s of sliding (~13 cycles of disk rotation) i.e., after stabilization of the sliding process. 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 as per literature practice [20, 24, 25]. The wear data have been obtained on at least three different samples utilizing at least two different tracks on each sample and an average value is reported.

Results and Discussion

Contact angle results

The water contact angles of bare Si, PI, and PI + SWCNTs films coated on Si are shown in Table 1. The water contact angle for Si/PI is 72° while that for bare Si is 12°, which implies the differences in their surface energies/wetting characteristics. The variation in the water contact angle value after PI deposition confirms its successful formation and is in good agreement with the literature values [7, 26]. The addition of SWCNTs to PI increases the water contact angle from 72° to 82°. The surface property of the CNT, which is essentially a graphite material (contact angle of 84–86°) [27, 28] was responsible for the increase in water contact angle.

Laser profilometry

The average thickness of the PI film measured using laser profilometer was 6–7 µm. SWCNTs addition did not

Table 1 Mean water contact angle values, hardness, elastic modulus, coefficient of friction and wear life data of bare Si, Si/PI and Si/PI + SWCNTs

Material	Water contact angle, degrees	Hardness, (Gpa)	Elastic modulus, (Gpa)	Coefficient of friction	Wear life, number of cycles
Si	12	–	–	0.6	100
Si/PI	72	0.43	5.23	0.1	3,000[a]
Si/PI + SWCNTs	82	0.72	8.41	0.12	7,200[a]

[a] The lowest and highest wear life data among three tests for PI film are 1,200 and 4,600 cycles, respectively, and for PI + SWCNTs film are 6,500 and 8,200 cycles, respectively

influence the average thickness of the film, which is as per expectation/anticipation because of the nanometer dimensions of the SWCNTs.

AFM topography results

The surface topography, as examined using AFM, of Si/PI, and Si/PI + SWCNTs is shown in Fig. 2. PI film on Si (Fig. 2a) shows island structure where the islands and valleys are very uniformly distributed. The average surface roughness was 2.4 nm while the depth of valleys was ~8 nm. The topography observed for PI film in the present study is comparable to that reported for similar films in the literature formed by solution casting [29] and spin-coating [30]. After the addition of SWCNTs, the surface still showed the island structure (Fig. 2b) but the islands are non-uniform and very irregular with a surface roughness of 9 nm. The average size of the valleys increased to ~15 nm. The reason for the non-uniformity and high roughness after the addition of SWCNTs could be because of the agglomeration of nano tubes to some extent.

Nanoindentation results

The hardness and elastic modulus data obtained in the CSM nanoindentation test for Si/PI and Si/PI + SWCNTs are shown in Fig. 3. During the indentation, all the curves of hardness and elastic modulus versus the indentation depth are approximately horizontal lines except at the initial stage. The elastic modulus and hardness at the film surface are higher, which is attributed to the difficulty in determining the point of contact and the in-accuracy of the indenter tip function at the shallow depth of the indentation [31]. The average hardness and elastic modulus values of the PI and PI + SWCNTs films are reported in Table 1. The average values of hardness and elastic modulus are obtained from the data for indentation depths between 200 and 2,000 nm, because Oliver and Pharr [32] reported that the data below 200 nm are not very accurate due to the difficulty in accurate estimation of the area function at very small indentation depths. Table 1 suggests that the hardness and elastic modulus of the PI film increased by

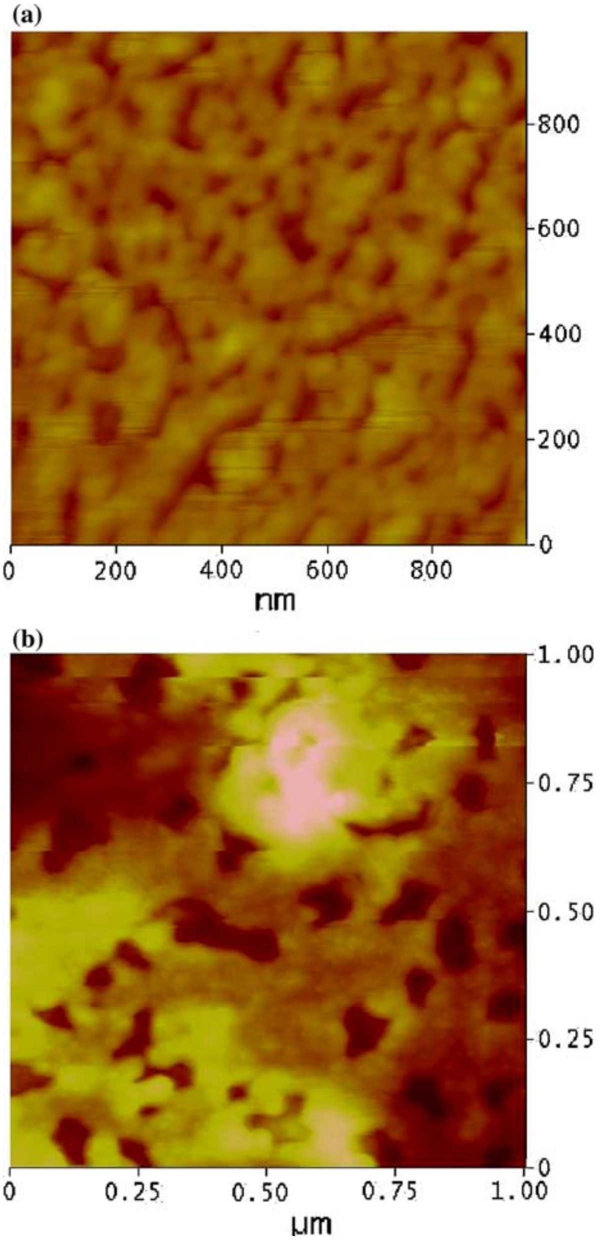


Fig. 2 AFM images of (**a**) Si/PI and (**b**) Si/PI + SWCNTs. The scan area is 1 µm × 1 µm and the vertical scale is 50 nm in both cases

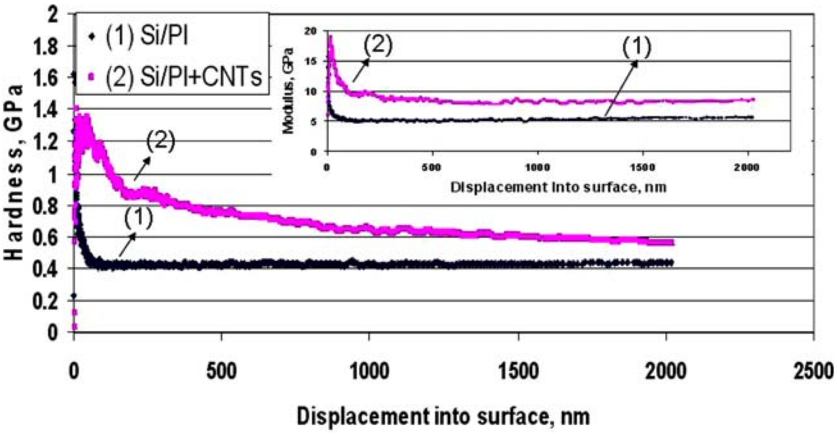


Fig. 3 Hardness with respect to the nanoindentation depth for Si/PI and Si/PI + SWCNTs during CSM nanoindentation test. Inset shows the Elastic modulus versus indentation depth curve

~1.6–1.7 times due to the addition of SWCNTs. The present observation is similar to that observed in bulk polymers where there is a good improvement in mechanical properties after the addition of CNTs [16]. Cadek et al. [16] observed that by adding various concentrations of MWCNTs, both Young's modulus and hardness increased by factors of 1.8 and 1.6 for 1 wt% MWCNT in polyvinyl alcohol (PVA) and 2.8 and 2 at 8 wt% MWCNT in poly (9-vinyl carbazole) (PVK), respectively. The reasons for the increase in the hardness and elastic modulus are due to the exceptional mechanical properties of CNTs. Moreover, it could be due to good interfacial bonding between the polymer and CNT which is possible because of high reactivity of the carbon tube surface as the graphene sheets of the carbon tube are strongly curved [33, 34]. These improved mechanical properties have led to an increase in the tribological properties as shown below.

Tribological results

Table 1 also shows the coefficient of friction of bare Si, Si/PI, and Si/PI + SWCNTs samples. PI film has shown a coefficient of friction of 0.1 whereas bare Si resulted in a value of 0.6. The addition of SWCNTs to the PI film has slightly increased the coefficient of friction of PI from 0.1 to 0.12. Similar increase in the coefficient of friction observed by Zoo et al. [14] for UHMWPE from 0.05 to 0.11 by the addition of SWCNTs (0.5 wt%) has been explained due to the increase in the shear strength and surface roughness by the addition of SWCNTs. The addition of SWCNTs to PI film was expected to reduce the coefficient of friction considering the fact of very low coefficient of friction of graphite, but there was an increase in the coefficient of friction. The graphite shows very low coefficient of friction because of its inherent lamellae structure which leads to easy shearing in the direction of the sliding [35], which is not observed in the case of CNTs. Neat PI film (without SWCNTs) is effective in reducing the friction because of its lower surface energy and flexible polymer chains [36]. Many thermoplastic polymer films provide low friction because of their lower shear strengths, which offer little resistance to sliding [1, 2].

The wear durability data that is the number of cycles after which the film fails for bare Si, Si/PI, and Si/PI + SWCNTs surfaces are shown in Table 1. The scatter in the wear durability data, in the present study, is comparable with the literature findings [25]. Bare Si has shown higher initial coefficient of friction and failed within few cycles of sliding. A clear visible wear track appeared very early during the sliding process. PI film has shown a wear life of ~3,000 cycles. This increase in wear durability, compared to bare Si, is comparable with literature findings on similar PI films, despite several differences in testing procedure and loading conditions [3, 8]. Good wear durability of PI film can be attributed to its good mechanical strength [37], flexible molecular chains [38], lower surface energy, and shear strength. After the addition of the SWCNTs to PI films, the wear life increased from 3,000 to ~7,200 cycles. It may be noted that these wear durability data are from the criteria of the coefficient of friction only, i.e., the film is deemed failed when the coefficient of friction exceeded 0.3. Actually, there was no visible wear after the above said number of cycles to both PI and SWCNTs added PI film and hence, if a coefficient of friction greater than 0.3 could be tolerated then the wear life would be much higher. In fact PI did not show wear until 20,000 cycles whereas PI + SWCNTs films did not show wear even after 100,000 cycles of sliding in a continuous wear test (see latter).

SEM/EDS analysis of the wear tracks, after appropriate number of sliding cycles, was carried out to investigate further details into the extent of wear of the film/Si surface. Eventhough the Si/PI film is considered failed after ~3,000 cycles based on the coefficient of friction data during sliding tests, there was no appreciable wear (SEM observation) until ~20,000 cycles. Figure 4 shows the SEM

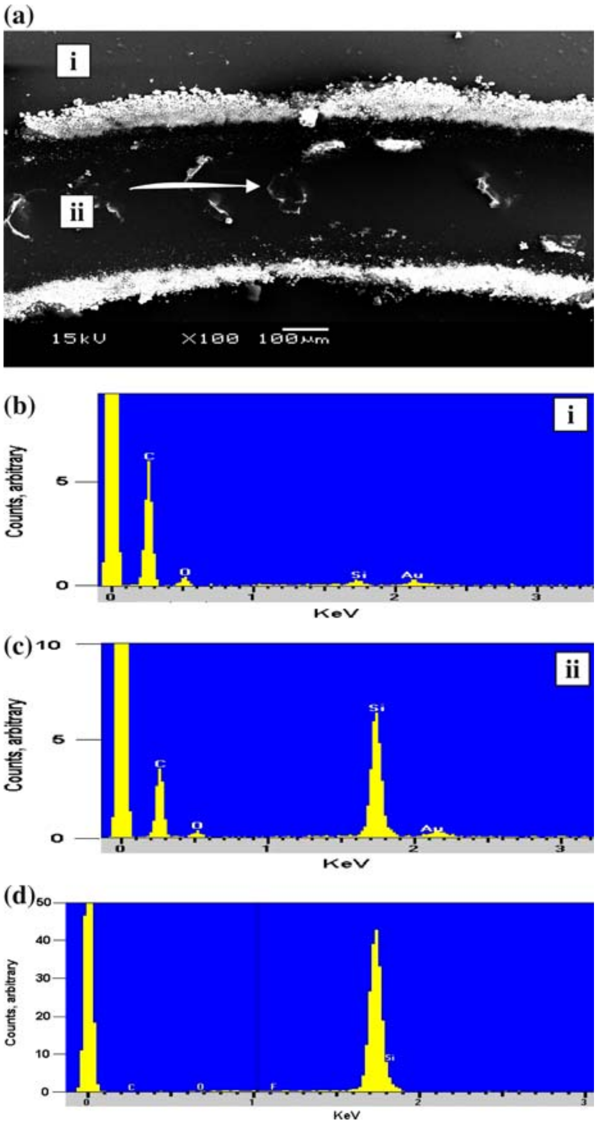


Fig. 4 (**a**) SEM image of the wear track of Si/PI, run upto 20,000 sliding cycles at a contact pressure of ~370 MPa (Arrow indicates the sliding direction). (**b**) and (**c**) show the EDS spectrum outside and inside the wear track respectively for the image shown in (**a**). (**d**) EDS spectrum on bare Si without any modification

micrograph of the wear track of Si/PI run until ~20,000 cycles of sliding. Greater accumulation of the wear particles along the sides of wear track is evident from the SEM micrograph. The worn surface of pure PI films shows indication of ploughing and delamination. Comparison of EDS peaks outside (Fig. 4b) and inside (Fig. 4c) the wear track of PI film and on original Si surface (Fig. 4d) confirm the following: (i) Lower intensity of C peak and higher intensity of Si peak inside the wear track indicate the wearing of PI film and appearance of original Si surface, (ii) Higher intensity of Si peak in Fig. 4d than in Fig. 4c confirms some PI film is still left on the wear track and (iii) Appearance of C peak in Fig. 4c and not in Fig. 4d also confirms some PI film is still present on the wear track. Hence, from SEM/EDS results, it is concluded that the PI film protects the Si surface upto ~20,000 cycles.

SEM image of the wear track of Si/PI + SWCNTs, run upto 100,000 cycles, is shown in Fig. 5a. No apparent wear track, even after sliding for 100,000 cycles could be seen clearly except slight compressed state of the surface protrusions. The FE-SEM images of Si/PI + CNTs, outside and inside wear track, are shown in Fig. 5b and c, respectively. The image shown in Fig. 5b supports that the polymer film after SWCNTs addition has shown island structure and valleys confirming the AFM image shown in Fig. 2b. EDS analysis inside the valleys showed a strong C peak with negligible Si peak confirming that the valleys were covered with PI. The image inside and outside wear tracks are similar except that the width of the valleys has been reduced and the islands have became flatter spreading over large area. Figure 5d and e show the AFM images of the film, outside and inside the wear track, respectively. The surface roughness of the worn region (10 nm), obtained from AFM images over a scan area of 10 μm × 10 μm, was very much less than that outside the worn region (66 nm) which in turn supports the absence of the wear to the film. The EDS analysis shows strong C peak both inside and outside the wear track (where the intensity was less inside the wear track) and absence of Si peak either outside or inside the wear track (data is not included). There is no appearance/accumulation of wear debris along the wear track (evident from SEM image shown in Fig. 5a). The low intensity (~50%) of the C peak inside the wear track than that outside the track implies the beginning of the wear of the film, but this is not evident from SEM image. The optical images of the ball surface (100× magnification) immediately after sliding and after cleaning with acetone are shown in Fig. 5f and g, respectively. The images of the ball imply that there is only a small amount of material transfer to the ball surface and there is no wear or scratching of the ball surface even after sliding for 100,000 cycles.

From the quantitative sliding tests data and qualitative SEM/EDS analysis, explained in the above sections, it can be deduced that the incorporation of SWCNTs into PI film contributes to restrain the wear of the PI film sliding against Si_3N_4 counterface. Two main reasons can be inferred for this improvement in wear durability. First, the addition of SWCNTs improves the hardness and elastic modulus, which must have increased the load-bearing capacity of the PI films. The similar increase in load-bearing capacity has been reported in bulk polymers such as polystyrene [39], UHMWPE [14] and PI [15], after the addition of CNTs. Therefore, the improved load-bearing capacity and smaller real area of contact because of the textured microstructure, after the addition of SWCNTs,

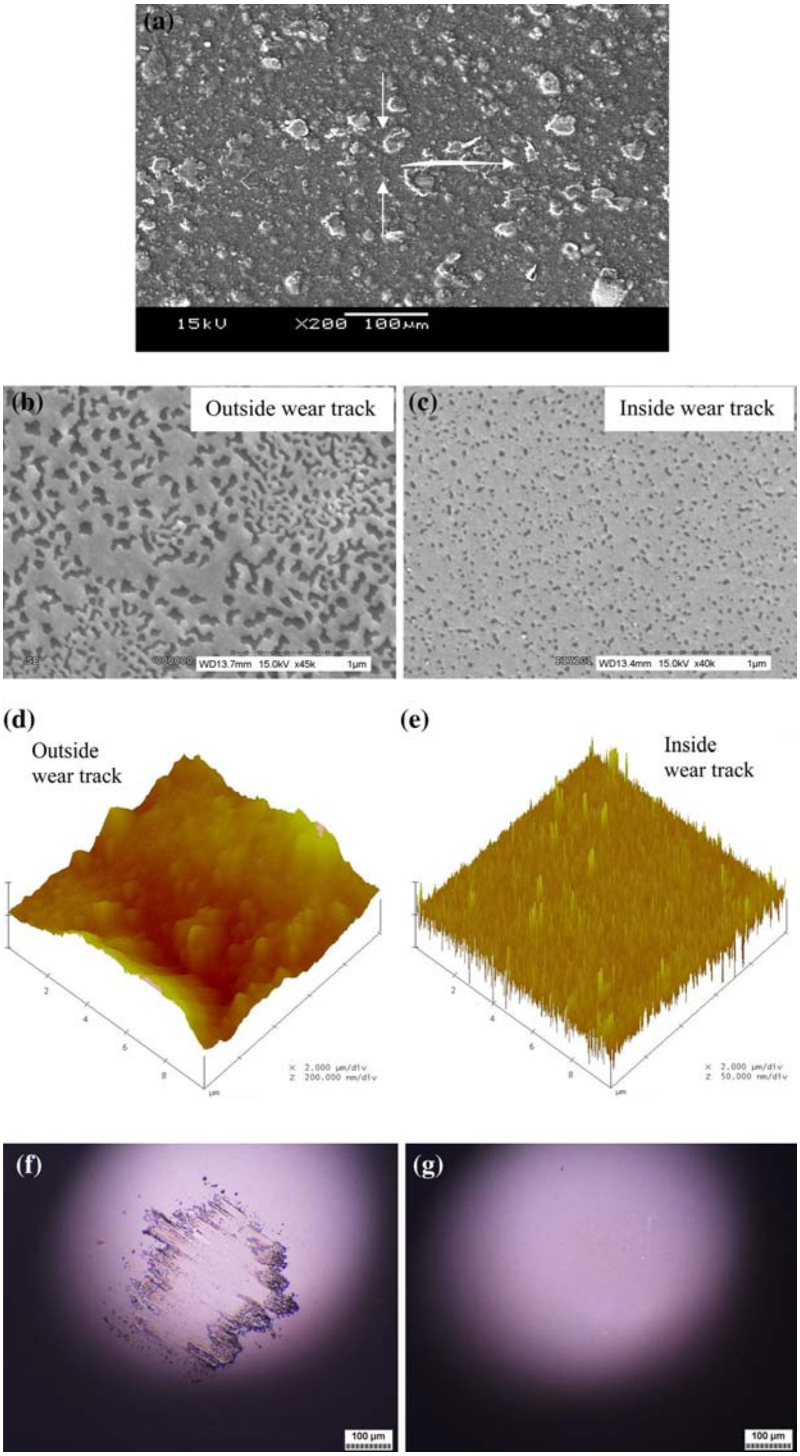


Fig. 5 (**a**) Wear track of Si/PI + SWCNTs, run upto 100,000 cycles (Arrows indicate the location of ball sliding and the sliding direction). FE-SEM images of Si/PI + SWCNTs, (**b**) outside the wear track and (**c**) inside the wear track. AFM images (10 μm × 10 μm scan area) of Si/Pi + SWCNTs, (**d**) outside the wear track and (**e**) inside the wear track. The vertical scale for the image in (**d**) is 200 nm whereas for (**e**) is 50 nm. Optical images of the Si_3N_4 ball slid against the sample in (**a**): (**f**) immediately after the sliding test and (**g**) after cleaning the transferred material on the ball surface with acetone

must have restricted the wear particle generation/material removal during sliding and resulted in very high wear durability. Second, the microstructural changes during sliding involving the vertical alignment/exposure of SWCNTs (see Fig. 5e), AFM image of the wear track) could contribute for wear durability enhancement because of the difficulty in breaking these nanotubes (and pulling out of polymer matrix), because of their exceptional elongational tensile strength properties. For example, Wong et al. [40] have reported an average bending strength of 14.2 GPa for MWCNT of 4.4 nm diameter. Moreover, the vertical alignment of SWCNTs induced during sliding coupled with the continuous increase in the contact area, explains the gradual increase in the coefficient of friction despite no wear to the film, because of the higher stiffness of CNTs, which offer greater resistance to the sliding.

Such CNT strengthened polymer, whether in bulk or film form, can be used for machines requiring wear resistance coating. Micromachine gears and parts are some possible application areas.

Conclusions

Polyimide was spin-coated onto bare Si, with and without the addition of SWCNTs, to investigate the effect of the addition of SWCNTs on the tribological properties of PI film.

Based on tribological studies, the following conclusions were drawn:

(1) Addition of SWCNTs to PI films resulted in an increase in the water contact angle from 72° to 82°.

(2) The addition of SWCNTs to PI film has shown an increase in the hardness (from 0.43 to 0.72 GPa) and elastic modulus (from 5.23 to 8.41 GPa) values, as evident from nanoindentation results.

(3) Both PI and (PI + SWCNTs) films are effective in reducing the coefficient of friction of bare Si. The addition of SWCNTs to PI film has resulted in slight increase in the coefficient of friction over that of neat PI films.

(4) Both PI and (PI + SWCNTs) films are very effective in reducing the wear of bare Si, which failed within 100 cycles. The PI composite film containing SWCNTs has shown wear life up to ~7,200 cycles with coefficient of friction less than 0.30. The composite film did not wear (evident from SEM/EDS analysis) even after 100,000 cycles with coefficient of friction reaching up to 0.5. Improved mechanical properties, after the addition of SWCNTs, and the microstructural changes induced during sliding, have helped to increase the wear durability.

The present study shows that CNTs can be considered as a promising additive for polymer-based thin films on Si surface for good wear resistance applications.

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