

Comparative study of tribological properties of insulated and conductive polyimide composites

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Abstract: Insulated polyimide (PI) composites filled with short glass fibers (SGF), polytetrafluoroethylene (PTFE), SiO₂, and polyphenylene (PPL) are specially designed, prepared, and the tribological properties are systematically investigated with references to the special requirements for frictional materials used in ultrasonic motors. The hardness and thermal decomposition temperature of the insulated PI composites are comparable to that of conductive PI composites. However, these insulated materials present excellent friction and wear performance, especially under high loads and speeds. Scanning electron microscopy (SEM) analysis of the worn surface indicates that adhesive and fatigue wear dominate the wear mechanisms.

Keywords: polyimide composites; insulation; friction; wear

1 Introduction

Ultrasonic motors (USMs) driven by the friction force between stator and rotor are becoming increasingly popular for use in medical applications, precision positioning, and aerospace engineering owing to their desirable properties, such as fast response, high torque at low speed, self-locking without requiring power, and flexible structure [1]. With the increasing popularity of USMs, tribo-materials are expected to have several desirable properties, such as high temperature resistance, exceptionally long service life, high strength, and radiation resistance. Frictional materials are core components of USMs, and can directly determine their mechanical output characteristics and service life. They also affect the stability and reliability of the entire driving system indirectly. Thus, frictional materials are one of the key factors that limit the development of USMs.

In the past decades, majority of the researchers devoted their efforts to the development of new

frictional materials or modification of tribo-interfaces [2–5]. Traditionally, certain polymer composites were employed as friction materials in traveling wave USMs in order to prolong their service life. Common methods for improving the wear resistance of such polymers included filling using fibers, solid lubricants, and nanoparticles. In composites, the fibers carry the applied load, while the solid lubricants improve the wear resistance of the polymer. In the early stage of USM development, Rehhein and Wallaschek [6] studied the friction and wear behavior of polyimide (PI) composites as frictional materials and found that filling carbon fibers could improve the wear resistance of PI sliding against steel. Concurrently, Ishii et al. [7] presented a method to predict the service life of USMs that use carbon fiber reinforced polymer as frictional materials, which was verified by the experiment. Subsequently, Lv et al. [8–14] researched a series of polymer-based frictional materials, such as polytetrafluoroethylene (PTFE), phenolic resin, and Ekonol composites, and compared their tribological properties

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under different conditions to provide guidance for selecting frictional materials according to the operating condition. Wang et al. [15, 16] systematically studied the effect of fillers, topography, temperature, and vacuum on the wear behavior of PTFE composites and found that filling some functional additives could improve the wear resistance of the PTFE matrix. High temperature and pressure strongly affect the wear mechanisms of PTFE-based frictional materials. In our previous studies [17–19], we designed some PI-based frictional materials with different tribological properties. However, it was difficult for these traditional frictional materials to meet the insulation requirements of the satellite system if it was modified using conductive fillers. Because the frictional materials in the USM stator that slides with the metal (a conductive material as a ground electrode) is running under a strong electric field, the current passing through frictional materials will affect the entire satellite system in the absence of suitable insulation. So, design of insulated frictional materials has an important role in the insulation of ultrasonic motors.

In the case of frictional materials, there are a few reports about polymer-based frictional materials. Until now, Fan et al. [20] prepared polyvinylidene fluoride (PVDF) and PTW/PTFE composites, which could improve the anti-wear ability of USM. Lin prepared PBT hybrid composites without filling solid lubricants, and compared their friction and wear behavior. They found that PBT-based tribo-compounds filled with short carbon fibers (SCF) and rigid particles without graphite still had excellent wear resistance. However, no literature reported insulated polyimide hybrid material. In our current work, we chose the PI matrix due to its excellent mechanical strength, thermal stability, high wear resistance, etc. Subsequently, insulated PI tribo-materials without the SCF and graphite filling were specially designed and their mechanical, thermal, and tribological properties were comprehensively compared with the corresponding conductive materials. The main aim of this study is to reveal the wear mechanisms to understand how isolated fillers produce the same lubrication effect, which can provide guidance for the design of high performance insulated frictional materials specially for USM applications.

2 Experimental

2.1 Materials

In this study, PI composites were prepared for use in USMs. PI YS-20 grade powder having average particle size $< 75 \mu\text{m}$ was supplied by Shanghai Synthetic Resin Institute (China). The chemical structure and properties of PI is shown in our previous study [17]. SCF were obtained from Nantong Senyou Carbon Fiber Co., Ltd. (China). The length of the fibers ranged from $20 \mu\text{m}$ to $50 \mu\text{m}$ and the diameter was approximately $7 \mu\text{m}$. SGF were supplied by Nanjing Institute of Glass Fiber Research and Design (China). The diameter of the fiber was $10 \mu\text{m}$ and the aspect ratio was 10:1. Flaky graphite powders ($< 1.5 \mu\text{m}$) was provided by Shanghai Colloid Chemical Plant (China). The PTFE powders (M18F, average diameter of $75 \mu\text{m}$) were obtained from Daikin Fluorochemicals Co., Ltd. (China). SiO_2 powders (500 nm) were purchased from Sigma-Aldrich. Polyphenylene (PPL) powders ($< 75 \mu\text{m}$) were purchased from Jiangsu Guorong Fluorine Plastic Products Co., Ltd. (China). Figure 1 shows the scanning electron microscope (SEM) morphologies of CF, GF, and SiO_2 . The volume and function of the individual components are listed in Table 1. The conductive PI composite was named as PI-1, and the insulated PI composite was designated as PI-2. Some key parameters of the two types of polyimide composites are listed in Table 2.

The PI composites were fabricated using the conventional hot press sintering technique. Firstly, the PI powders and all fillers were dispersed in ethanol by mechanical stirring followed by placing in an ultrasonic bath for 2 h, and dried in a vacuum oven for use. Then, the mixtures were compressed in a mold and heated up to $375 \text{ }^\circ\text{C}$ with intermittent deflation. The pressure was held at 20 MPa for 60 min to allow completely compressed sintering. Finally, the specimens were cut into different sizes for experiments after natural cooling. As can be seen from the fracture surface in Fig. 2, the PI composites were successfully prepared. Some fibers can be clearly seen on the fracture surface of the polymer composites. The other fillers without obvious morphologies may be coated by the polymer matrix. Generally, there was no cavitation or agglomeration on the surface, which indicated the even

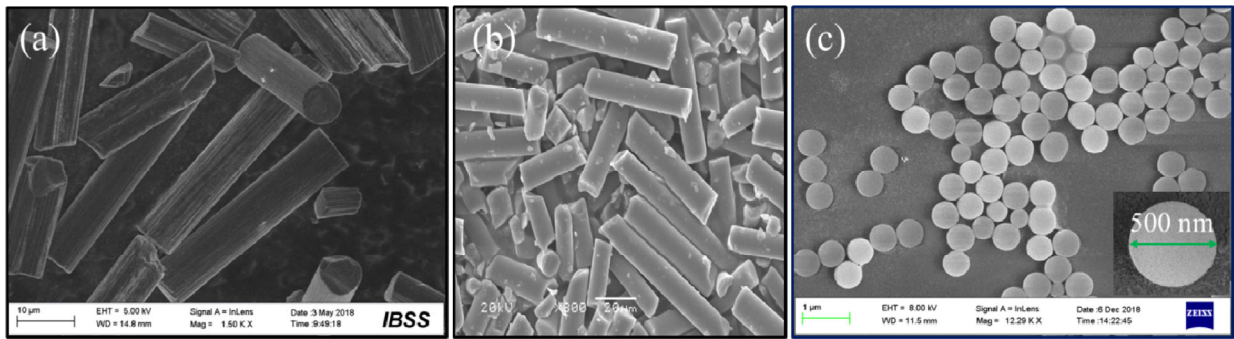


Fig. 1 SEM morphologies of (a) CF, (b) GF, and (c) SiO₂.

Table 1 Compositions of the tribo-materials.

Sample	PI	SCF	SGF	Graphite	PTFE	SiO ₂	PPL	(vol.%)
PI-1	69	15	—	10	—	1	5	
PI-2	69	—	15	—	10	1	5	
Function	Matrix	Reinforce	Reinforce	Lubricants	Lubricants + low surface energy	Fortifier	Thermal stability	

Table 2 Some main parameters of PI composites.

Sample	Tensile strength (MPa)	Elongation percentage (%)	Elastic modulus (GPa)	Electrical resistance
PI-1	273.4	5.3	13.47	300 Ω–400 Ω
PI-2	225.2	6.2	9.23	∞

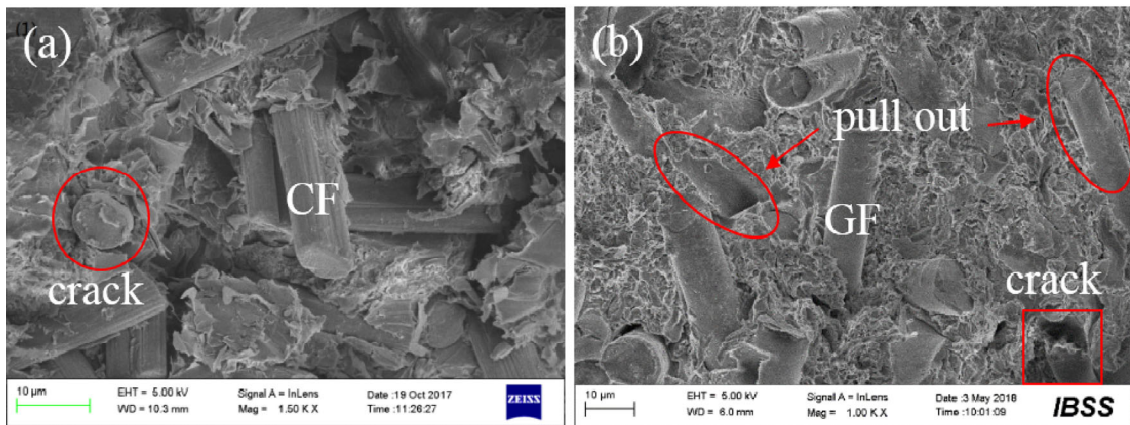


Fig. 2 Fracture morphologies of the polyimide composites: (a) PI-1 and (b) PI-2.

distribution of fillers into the PI matrix. Before tribo-tests, the PI composites were polished by metallographic sandpaper to a roughness below 0.1 μm.

2.2 Friction and wear tests

The friction and wear tests involving sliding against a Si₃N₄ ball were evaluated on a ball-on-disk tribometer (HSR-2M, China) referred to the ASTM G99-04 Pin-on-Disk pattern at room temperature. The schematic diagram of the friction couple is shown in Fig. 3. The

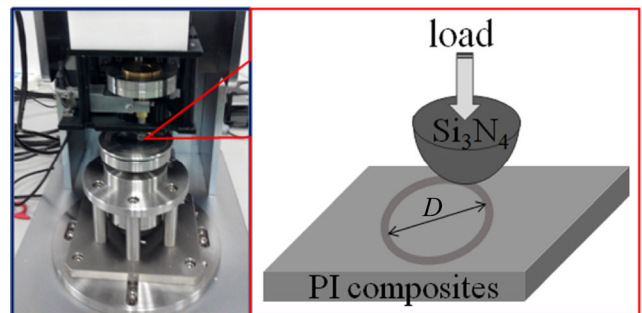


Fig. 3 Photograph of the tribo-meter and schematic of wear test.

Si_3N_4 ball is 4 mm in diameter. Before each test, the Si_3N_4 ball and the block samples were cleaned with cotton dipped in acetone. The wear volume was computed from the cross-sectional worn area multiplied by the circumference of the wear track and calculated using Eq. (1). The specific wear rate K ($\text{mm}^3/\text{N}\cdot\text{m}$) was calculated from the volume loss using Eq. (2).

$$\Delta V = \left[\frac{\pi(R/2)^2}{180} \arcsin \frac{b}{R} - \frac{b\sqrt{(R/2)^2 - (b/2)^2}}{2} \right] \pi D \quad (1)$$

$$K = \frac{\Delta V}{PL} \quad (2)$$

where ΔV is the wear volume loss (mm^3), R is the diameter of the ball (4 mm), b is the width of the wear track (mm), D is the diameter of the disk (6 mm). P is the load (N), and L is the total sliding distance (m). The coefficient of friction, which taken on the average value in the steady stage, was continuously recorded by an on-line data acquisition system attached to the tester.

2.3 Characterization

The hardness was measured using Shore D (SD) tester. Each sample was tested ten times and the average value was used. The tensile strength of the samples were measured using a universal tensile testing machine (SANS-CMT5, Shenzhen Sans Testing Machine Co., Ltd., China), according to Fiber-reinforced plastics composites–Determination of tensile properties (GB/T 1447–2005) and repeated at least three times. The average value was used for subsequent computations. The electrical resistance was measured by a multimeter at different points of the PI composite specimens. Thermo gravimetric analysis (TGA) and differential scanning calorimetry tests were carried out under a N_2 atmosphere with a Netzsch TG 209 on 19 TASC 414 41 analyzer (Germany) from 40 °C to 800 °C with a heating rate of 10 °C/min. The width of the worn track was measured under an optical microscope (VHX-1000 3D ultra-depth of field microscope, Japan) to calculate the wear rate. The worn surface morphologies of the samples were observed using a Zeiss-sigma (Germany) SEM. In order to increase the resolution of SEM observation, the surfaces were plated with a gold coating to render them electrically conductive.

3 Results and discussion

3.1 Hardness

Figure 4 shows the hardness (SD) of polyimide composites. Compared with PI-1, the hardness of PI-2 decreased slightly due to the addition of PTFE as it has a relatively lower modulus and hardness than PI [21]. That will affect the thermal and tribological properties of the composites. The detailed experimental results are presented in the section.

3.2 Thermo gravimetric analysis

In order to analyze the thermal stability of the PI composites, Fig. 5 shows the similarity of the thermo gravimetric analysis curves of two kinds of materials from 40 °C to 800 °C. The decomposition temperature of the composites mainly depends on the polyimide matrix (564 °C) [19]. The incorporation of other fillers will affect the actual decomposition temperature and residual mass. For PI-1, the rate of decomposition reached the peak value when the temperature rose to 548.6 °C and the residual mass was 74.35% after 800 °C.

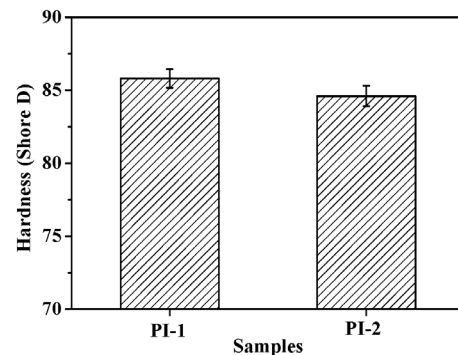


Fig. 4 Hardness of PI composites.

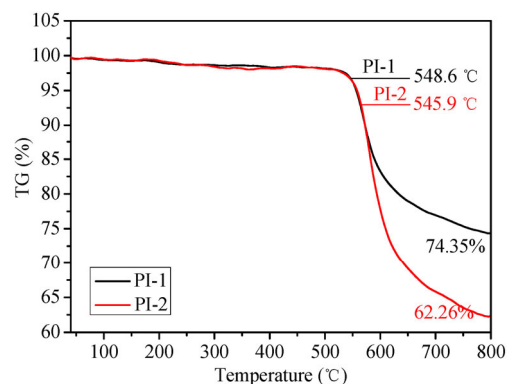


Fig. 5 TGA curves of the polyimide composites.

But PI-2 had the maximum rate of decomposition at 545.9 °C and 62.26% residual mass. The main difference between PI-1 and PI-2 was the difference in the composition of the fibers and solid lubricants. As far as fibers in the composite are concerned, both glass fibers and carbon fibers have excellent thermal resistance. So, the dominant reason for the variation can be identified as the addition of PTFE that had a relatively low decomposition temperature of approximately 460 °C according to Sun et al. [22]. So, introducing PTFE into the polyimide composite will reduce the decomposition temperature slightly and the residual mass by approximately 12.09%.

3.3 Friction and wear behavior

The tribological properties of the two composites were systematically compared to evaluate the wear resistance under different operating conditions. Firstly, the friction and wear tests were performed at the speed of 0.063 m/s and the load of 3 N under rotating wear. The total test duration is taken as 6 h to acquire the reliable data in the stable stage with the rotating radius of 3 mm. Figure 6 shows the variation of the coefficient of friction with time. It can be clearly seen that PI-2 has a smaller friction coefficient than PI-1. This can be ascribed to the excellent lubrication effect of PTFE. Judging from the variation of the friction coefficient of PI composites, PI-1 shows more stable friction behavior than PI-2, because the added graphite in the PI composites was easily transferred onto the surface of the counterpart. So, PI-1 will become stable in short running time. However, the friction coefficient of PI-2 filled with PTFE continuously increased with

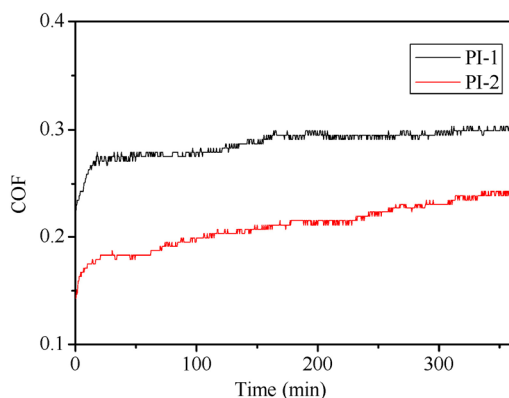


Fig. 6 Friction coefficient of PI composites under 3 N and 0.063 m/s.

time, and required longer time to attain stability. The fundamental reason can be ascribed to the difference in the materials. For polymer composites reinforced with fibers, some fibers will be exposed on the surface of the polymer after the superficial polymer matrix wears off. Then, the shear stress concentrates on the fibers that carry most of the applied load, while shielding the polymer. PI-2, which is reinforced with glass fibers, had a higher surface hardness and better wear resistance than PI-1. It indicates that PI-2 needs a longer time to reach the same wear volume under the same wear testing time than PI-1 [23]. The area of contact between the Si_3N_4 ball and PI-2 will enlarge with time. It has been reported that increase in the area of contact can lead to the increase in material transfer and increment in the friction coefficient [11]. So, the friction coefficient of PI-2 increases with time.

The same phenomenon can be observed from Fig. 7 under high loads and speeds. The PI-2 showed a smaller friction coefficient than PI-1 under the same conditions. In addition, high load and speed contributed to the reduction of friction for both composites. The effect of speed and load on the friction of the PI composites was also tested and compared.

As seen from Fig. 8, the friction coefficient of the PI composites decreased with an increase of speed from 0.063 m/s to 0.157 m/s under the load of 3 N with a duration of 6 h. It is obvious that an increase of speed will result in the rise of temperature on the surface of the PI composites. The higher the testing speed, the faster the accumulation of frictional heat at the friction surface. Rising temperature leads to the softening of some polymer molecular chains, which can result in

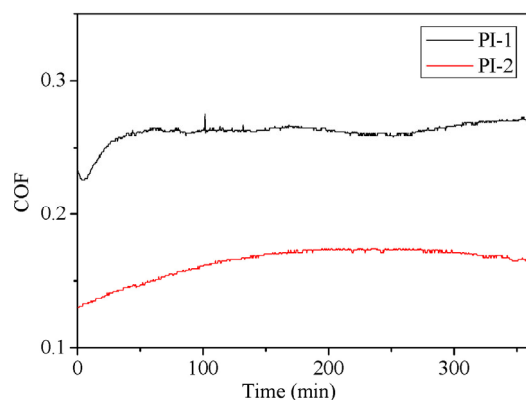


Fig. 7 Friction coefficient of PI composites under 9 N and 0.157 m/s.

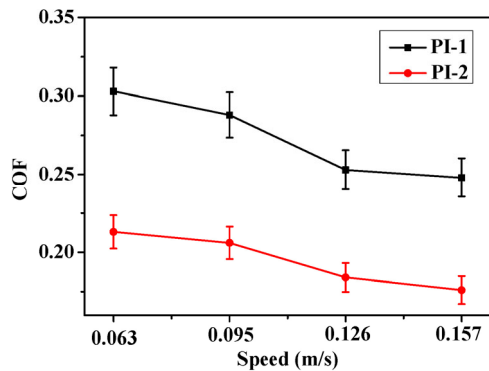


Fig. 8 Average friction coefficient of PI composites under different speeds.

a reduction of the friction coefficient [24, 25]. Besides, it can be clearly seen that PI-2 had a lower friction coefficient than PI-1 under the same testing conditions, which was determined by their essential properties. The fundamental reason can be attributed to the addition of glass fibers and PTFE into PI composites.

Except for the speed of rotation, the applied load was one of the most important factors affecting the tribological properties of polymer composites. In this study, the friction coefficient of PI composites decreased sharply when the applied load increased from 3 N to 5 N at a constant speed of 0.063 m/s with a duration of 6 h as shown in Fig. 9. Then, they had relatively small variations from 5 N to 9 N. Firstly, the high applied load will contribute to the increase of the actual area of contact between the Si_3N_4 ball and polymer composite because of elastic and plastic deformation. Reduction of the frictional force was detrimental for the tribo-pairs. However, the increasing amplitude of frictional force was generally smaller than the applied load. So, the friction coefficient of system will decrease with an

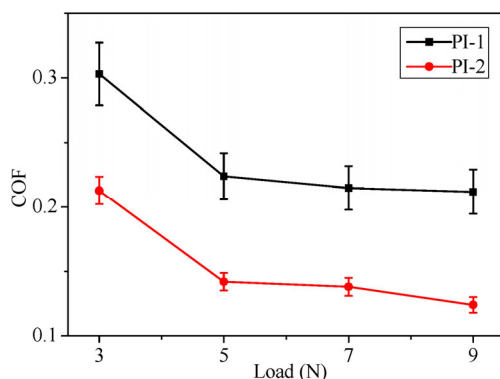


Fig. 9 Average friction coefficient of PI composites under different loads.

increase in the load. Besides, the frictional heat cannot be ignored, especially under high load as the high flash temperature on the tips of the rough surface of the polyimide composites was enough to make partial polyimide molecular chain soft, or even molten under the combined effect of speed and ambient temperature. Soft polymer chains will reduce the shear stress and improve the lubrication of the tribo-system. So, increasing the load will reduce the friction coefficient of the polyimide composite for both insulating and conductive tribo-materials.

Except for the friction that will vary with the speed of rotation and load, the wear behavior should be sensitive to the operating conditions. In this study, the specific wear rate was calculated along the wear track and compared by selecting typical low (3 N, 0.063 m/s) and high (9 N, 0.157 m/s) load and speed values. The aim is to obtain the wear resistance of two types of PI composites under the same conditions. Firstly, the wear width was measured using optical microscopy as shown in Fig. 10. Then, the wear volume loss was calculated using Eq. (1). Finally, the average specific wear rate with the standard error can be obtained from Eq. (2) under a certain load and sliding distance. As seen from Fig. 11, the PI-1 had a high wear rate ($3.84 \times 10^{-6} \text{ mm}^3/\text{N}\cdot\text{m}$) under 0.063 m/s and 3 N. By contrast, the insulated PI-2 had a low wear rate of approximately $2.03 \times 10^{-6} \text{ mm}^3/\text{N}\cdot\text{m}$, decreased by 47.14%. It indicated that substituting the CF and graphite with GF and PTFE could improve the wear resistance of PI composites under the synergistic effect of SiO_2 and PPL. Comparable results can be obtained under 9 N and 0.157 m/s. The specific wear rate of PI-2 decreased from $1.92 \times 10^{-6} \text{ mm}^3/\text{N}\cdot\text{m}$ to $0.81 \times 10^{-6} \text{ mm}^3/\text{N}\cdot\text{m}$. This

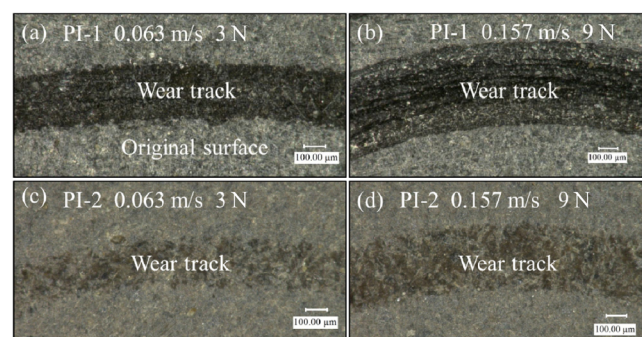


Fig. 10 Optical microscopies of wear track of PI composites. (a) PI-1 and (c) PI-2 under 0.063 m/s and 3 N, (b) PI-1 and (d) PI-2 under 0.157 m/s and 9 N.

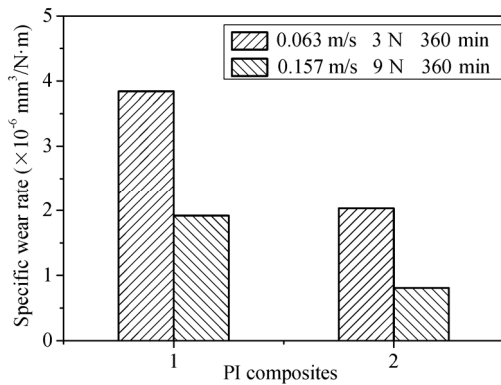


Fig. 11 Specific wear rate of PI composites under different conditions.

can be ascribed to the excellent self-lubrication effect of PTFE and good compatibility with the PI matrix. In summary, this insulation design for a PI composite has the potential to reduce the friction and improve the wear resistance, as evidenced by the above discussion about friction coefficient and change in wear rate.

To discover wear mechanisms, the worn morphologies of PI composites under different conditions were observed by SEM as shown in Fig. 12. It can be clearly seen that the worn surfaces of the PI composites are completely different from the original surface. For PI-1, the worn surface morphologies shown in Figs. 12(b) and 12(c) seem more smooth and homogenous after 6 h of rotating wear compared with the original surface (Fig. 12(a)). Under the load of 3 N and 0.063 m/s, the worn surface was comparatively smooth

because the asperities on the original rough surface were worn off. During this process, adhesive wear was dominant in the beginning, after which a steady state was attained when the tribo-films were formulated between the PI samples and counterpart. However, under the high load (9 N) and speed (0.157 m/s), some cracks were formed on the worn surface (Fig. 12(c)) due to fatigue wear. This explains the higher wear rate shown by PI-1 with respect to PI-2. For PI-2, the surface structure was different from PI-1 because of the addition of different fillers, especially the incorporation of glass fibers and PTFE. There were plenty of wrinkles on the worn surface as can be seen from Figs. 12(e) and 12(f). This phenomenon suggested that plastic deformation happened on the worn surface under the repeated shear stress, which is related to their mechanical and thermal properties. As discussed in Sections 3.1 and 3.2, PI-2 had lower hardness and decomposition temperature than PI-1. Compared with the worn surface under the relatively low load (3 N) and speed (0.063 m/s), more severe cracks were found on the worn surface under the high load (9 N) and speed (0.157 m/s). For the PTFE filled PI composite, adhesive and fatigue wear together dominated the wear process. However, according to Eqs. (1) and (2), the specific wear rate did not increase exponentially with the load and speed. Hence, the specific wear rate will decrease under a high load and speed. This is consistent with the results of wear rate shown in Fig. 11.

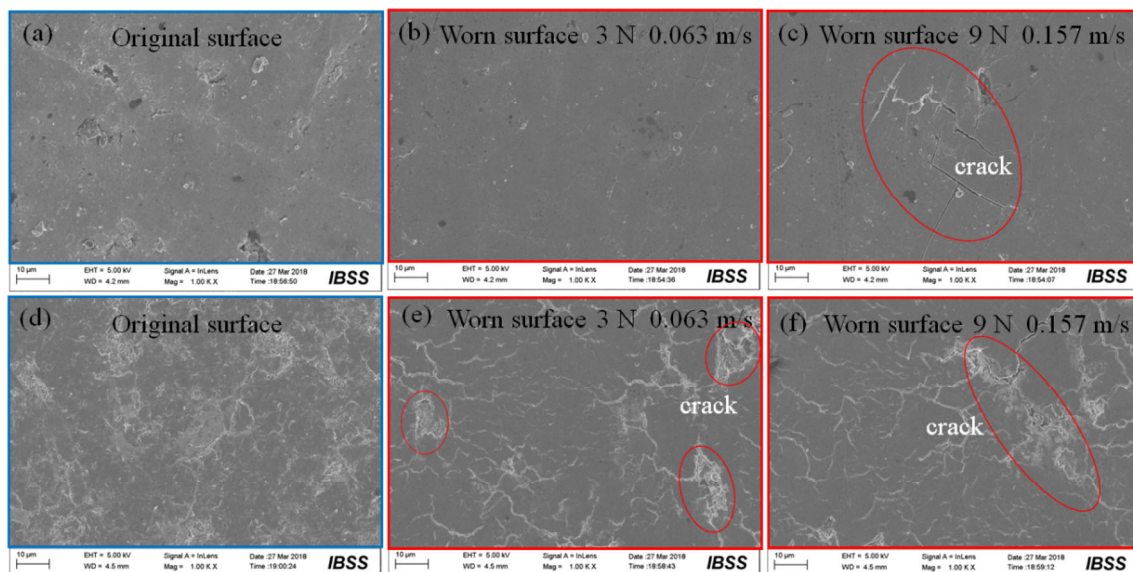


Fig. 12 Original surface (a) PI-1 and (d) PI-2, and worn surface morphologies of the polyimide composites (b, c) PI-1 and (e, f) PI-2.

4 Conclusions

The mechanical, thermal, and tribological properties of insulated and conductive PI composites were systematically compared and discussed. Although the conductive polyimide composites can be used as tribo-materials for USMs under normal conditions, the new polyimide composites after insulation design can also satisfy the requirements of low friction and wear rate. The substitution of carbon fibers and graphite with glass fibers and PTFE did not greatly affect the hardness and thermal decomposition temperature for this PI composite, which, contrarily, reduce the friction and wear rate analyzed from tribo-tests and worn surface characterization. Different wear mechanisms dominated the wear process of these two PI composites. The synergistic effect of glass fibers, PTFE, SiO₂, and PPL will contribute to the excellent tribological performance of the PI matrix, which can provide significant guidance for designing other types of insulated polymer composites.

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