Tribological Behavior of Short-Fiber-Reinforced Polyimide Composites Under Dry-Sliding and Water-Lubricated Conditions

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Received 4 December 2006; accepted 29 June 2007 DOI 10.1002/app.27127 Published online 25 September 2007 in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: Polyimide composites reinforced with shortcut fibers such as carbon, glass, and quartz fibers were fabricated by the polymerization of monomer reactants process. The mechanical properties of the composites with different fiber contents were evaluated. The friction and wear properties of the polyimide and its composites were investigated under dry-sliding and water-lubricated conditions. The results indicated that the short-carbon-fiber-reinforced polyimide composites had better tensile and flexural strengths and improved tribological properties in comparison with glass-fiber- and quartz-fiber-reinforced polyimide composites. The incorporation of short carbon fibers into the polyimide contributed to decreases in the friction coefficient and wear rate under both dry and water-lubricated

INTRODUCTION

Polymers are finding wide acceptance in tribological applications because of their low friction against steel counterparts and their self-lubricating ability; in particular, polymer matrix resins reinforced with fibers have been widely accepted as tribo-materials and used as components that are run without any external lubricants.^{1–13} The tribological performance of such components is sensitive to application conditions.14-16 Lancaster2 summarized the influence of environmental humidity and water on the tribological properties of polymers and composites and pointed out that the friction of polymers and their composites is sensitive to the presence of surface contamination. Recently, many studies on the tribological performance of fiber-reinforced polymer composites under different conditions have been reported. Xu et al.¹⁷ demonstrated that a polyphenylene sulfide (PPS) composite reinforced with carbon fiber (CF) showed much better wear resistance under

Journal of Applied Polymer Science, Vol. 107, 788–796 (2008) © 2007 Wiley Periodicals, Inc.

WVILEY InterScience® conditions and especially under water lubrication because of the boundary lubrication effect of water. The polyimide and its composites were characterized by plastic deformation, microcracking, and spalling under both dry and water-lubricated conditions, which were significantly abated under the water-lubricated condition. The glass and quartz fibers were easily abraded and broken; the broken fibers transferred to the mating metal surface and increased the surface roughness of mating stainless steel, which led to the wear rate increasing for the glass-fiber- and quartzfiber-reinforced polyimide composites. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 107: 788–796, 2008

Key words: composites; polyimides; mechanical properties

the water-lubricated condition against stainless steel than under dry sliding. Yamamoto and Hashimoto¹⁸ found that both poly(ether ether ketone) and PPS were considerably improved in friction and wear characteristics under hydrodynamic lubrication conditions by the addition of glass fiber (GF) or CF. Unal and Mimaroglu¹⁹ reported that the specific wear rate of poly(ether ether ketone) and its CF-reinforced composite was significantly influenced by the environmental conditions (from dry to water-lubricated conditions). Wang and Gu²⁰ reported that the tribological and mechanical properties of nylon were remarkably improved by the addition of CFs and that the wear rate and the friction coefficient of 10% CF/ nylon in water showed great decreases in comparison with those under dry-sliding conditions. Generally, fiber reinforcement or/and solid lubrication can improve the tribological performance of polymers significantly; however, this is not always true. The fillers successful in one typical wear mode may not be equally successful in other wear situations.²¹⁻²⁴

Polyimides (PIs) are well recognized as high-performance polymers because of their high thermal and chemical stability, good mechanical properties, high wear resistance, excellent dielectric properties, and radiation stability.^{25–28} To our knowledge, there

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have been few systematic studies on the wear behavior of PIs in diverse wearing situations and operating conditions. Therefore, it is necessary to understand the tribological behavior of PI composites under different working conditions. It has been confirmed that the tribological performance of PIs can be improved by the incorporation of CFs with high strength and modulus.^{1,2,26} High-performance PI composites are applicable under many conditions and especially in situations in which metals are liable to corrosion or fatigue.^{29–33} As a matter of fact, there are numerous sliding applications in which water either is deliberately introduced as a coolant (e.g., in rolling mill bearings) or is present as a working fluid (e.g., in pumps). In the latter case, a marine environment can pose special problems because of the abrasive contamination and corrosiveness of sea water toward many metals.²

In our previous work,¹⁶ the tribological behavior of PI composites filled with short CF and traditional solid lubricants, such as graphite, MoS₂, and polytetrafluoroethylene, was investigated. We found that the incorporation of CF and solid lubricants into PIs could improve the friction and wear behavior considerably. The PI composites showed a great reduction of the friction coefficient and wear rate in sliding against stainless steel under both dry and waterlubricated conditions in comparison with pristine PI.

As a continuation of work aiming at clarifying the influence of fiber reinforcements on the tribological behavior of polymers, PIs reinforced with short-cut fibers, such as CF, GF, and quartz fiber (QF), were investigated. The friction and wear behavior of the PI composites was evaluated under dry-sliding and water-lubricated conditions. The wear mechanisms of the composites under the two different sliding conditions were also comparatively discussed on the basis of a scanning electron microscopy (SEM) examination of the worn composites and steel counterpart surfaces.

EXPERIMENTAL

Materials

4,4'-Methylenedianiline (MDA; Beihua Fine Chemicals Co., Beijing, China) was recrystallized in anhydrous ethanol before use. *p*-Phenylenediamine (*p*-PDA, Beihua Fine Chemicals) was purified by vacuum sublimation before use. 3,3',4,4'-Oxydiphthalic anhydride (Shanghai Research Institute of Synthetic Resins, Shanghai, China) was recrystallized from acetic anhydride before use. *cis*-5-Norbornene-*endo*-2,3-dicarboxylic anhydride (Nanxiang Chemical Reagents Co., Shanghai, China) was vacuum-sublimated before use. Anhydrous ethyl alcohol (Beijing Fine Chemicals Co., Beijing, China) was dried with molecular sieves and freshly distilled before use. Short-cut CF (T-300) with a length of 6–8 mm and a diameter of 6–8 μ m was provided by Jilin Chemical Industrial Co., Ltd. (Jilin, China). Short-cut GF (E-glass) with a length of 4–6 mm was purchased from Taishan Fiberglass, Inc. (Taishan, China). Short-cut QF with a length of 4–6 mm was purchased from Jingzhou Feilihua Quartz Glass Co. (Jing Zhou, China). All the fibers were used as received.

Preparation of short-fiber-reinforced PI composites

PIs were prepared by *in situ* polymerization of monomer reactants (PMR), as shown in Figure 1. The PMR monomer solutions in ethyl alcohol were formulated from diethyl ester of 3,3',4,4'-oxydiphthalic acid (ODPE), aromatic diamines MDA and *p*-PDA, and monoethyl ester of *cis*-5-norborneneendo-2,3-dicarboxylic acid (NE) according to the literature.^{34,35} The ODPE/(MDA + *p*-PDA)/NE molar ratio was 2.467 : 3.467 : 2, in which MDA/p-PDA was 1 : 1, ensuring a PI matrix resin with a calculated molecular weight of 1500. CF, GF, and QF were mixed with PMR monomer solutions with different fiber contents. The mixtures were then treated at 80, 120, 200, and 220°C in vacuo to afford imidized Bstaged molding powders. The powers were placed in a stainless steel mold at room temperature, and this was followed by placement in a preheated hot press (270°C) with a contact pressure. After that, the hot press was heated to 320°C and held for 2 h under a pressure of 3-4 MPa. Then, the mold was cooled to 200°C, and this was followed by pressure release. The composite plate was removed from the mold at room temperature. The fully cured PI composite was obtained through postcuring at 320°C for 10 h in an oven with circulating nitrogen at a flow rate of 100 mL/min. Three kinds of short-fiber-reinforced PI composites with different fiber contents were prepared. The PI composites reinforced with short-cut CF, GF, and QF were labeled CF/PI, GF/ PI, and QF/PI, respectively.

Characterization

Tensile strength and flexural strength measurements were performed with 6-mm-wide specimens in accordance with GB1447-83 at a rate of 5.0 mm/min and in accordance with GB1449-87 at span-to-depth ratios of 15–16 at a rate of 1.0 mm/min, respectively.

The friction and wear behavior of neat PI and PI composites sliding against stainless steel was evaluated on an MM-200 model ring-on-block test rig (Jinan Shijin Group Co., Ltd., Jinan, China). The blocks ($30 \times 7 \times 6 \text{ mm}^3$) were made of the PI composites, and the ring of $\emptyset40 \times 10 \text{ mm}$ was made of stainless steel (1Cr18Ni9Ti, Brinell hardness = 1.48 GPa). The tests were carried out at a linear velocity of 0.43 m/s with a load of 200 N for 120 min.



Figure 1 Chemical and thermal processes for a PMR PI.

The stainless steel ring and the PI composite block were abraded with no. 900 water-abrasive paper (Beijing Dong Sheng Coated Abrasive Industrial Co., Beijing, China) and then ultrasonically cleaned with acetone before each test. The average surface roughness of the rings and blocks was about 0.1-0.2 µm. The water lubrication between the sliding surfaces was realized by continuous dropping of distilled water on the sliding surface at a rate of 65-70 drops per minute. The friction force between the tested block and the counterpart steel ring was measured with a torque shaft equipped with strain gauges. The width of the wear tracks was measured with a digital-reading microscope to an accuracy of 0.01 mm. Then, the wear rate of the specimen was calculated with the following relationship:

$$\omega = \frac{B}{L \times N} \left[\frac{\pi r^2}{180} \arcsin\left(\frac{b}{2r}\right) - \frac{b}{2}\sqrt{r^2 - \frac{b^2}{4}} \right]$$
(1)

where ω is the specific wear rate (mm³/N m), *B* is the width of the specimen (mm), *L* is the sliding distance (m), *N* is the load (N), *r* is the semidiameter of the stainless steel ring (mm), and *b* is the width of the wear trace (mm). The average of three replicate test results was reported. The details about the friction and wear tests have been reported previously.^{16,35}

Journal of Applied Polymer Science DOI 10.1002/app

The wear tracks of the composites and stainless steel specimens were examined on a JEM-5600LV scanning electron microscope (Japan Electron Optics Laboratory Co., Ltd., Tokyo, Japan). To increase the resolution of the SEM observation, the tested composite specimens were plated with a gold coating to render them electrically conductive.

RESULTS AND DISCUSSION

Mechanical properties of PI composites

The tensile strength and flexural strength of PI composites with different fiber contents are shown in Figures 2 and 3. All the composites exhibited good mechanical properties, with the tensile strength and flexural strength in the ranges of 75–101 and 105–154 MPa, respectively. The tensile strength and flexural strength of the PI composites increased with increasing fiber content and then decreased gradually, no matter what kind of fiber was applied. The results indicated that the PI composites generally had improved tensile and flexural strength properties in comparison with neat PI (except PI composites with 30 wt % QF). The CF/PI composites exhibited better tensile and flexural strength than the GF/PI and QF/PI composites. The CF/PI with 20 wt % CF showed the highest tensile strength of 101 MPa,



Figure 2 Variation of the tensile strength of PI composites with the fiber content.

which was 26 MPa higher than that of the neat PI. The CF/PI with 10 wt % CF showed the highest flexural strength of 154 MPa, which was 40 MPa higher than that of the neat PI. It was also found that the influence of the fiber content on the tensile strength and flexural strength of the PI composites made a difference; the tensile strength decreased as the fiber content reached 15–20 wt %, whereas the flexural strength decreased as the fiber content exceeded 10 wt %. We suspect that the decreasing tendency observed for the PI composites with relatively high fiber contents can be attributed to the existence of fiber clusters or aggregation to some extent.³⁶

Friction and wear properties of the PI composites

Figure 4 shows the friction coefficients of a PI resin and its composites with 10 wt % fibers against stain-



Figure 3 Variation of the flexural strength of PI composites with the fiber content.

less steel as a function of the sliding time under ambient dry-sliding and water-lubricated conditions at a linear velocity of 0.43 m/s with a load of 200 N. The initial friction coefficients of the PI resin and its composites were relatively high under dry sliding, then decreased with the sliding time, and finally reached a steady state in the range of 0.2-0.4 as the sliding time increased beyond 20 min [Fig. 4(a)]. The decrease in the friction coefficient with the sliding time could be explained by the formation of a transfer film on the steel surface because of the adhesion of the resin,^{16,17} which can also be proved by the observation of SEM micrographs. On the other hand, the initial friction coefficients of the PI resin and its composites were relatively low under water lubrication, increased abruptly and then decreased gradually with the sliding time, and finally achieved a steady state in the range of 0.2-0.3 [Fig. 4(b)]. The increase in the friction coefficient of the PI composites under water lubrication in the early stage of



Figure 4 Variation in the friction coefficient of a PI resin and PI composites with 10 wt % fibers under (a) drysliding and (b) water-lubricated conditions.

Journal of Applied Polymer Science DOI 10.1002/app



Figure 5 Variation in the steady-state friction coefficient of PI composites with different fiber contents under (a) dry-sliding and (b) water-lubricated conditions.

sliding might be due to the incomplete formation of the water-boundary lubricating layer. With the prolongation of the sliding time, a complete waterboundary lubricating layer was formed on the surface of the composites by saturated adsorption of water, which caused the decrease in the friction coefficient. The PI composites exhibited lower steadystate friction coefficients under water lubrication than the composites under dry sliding. This result could be interpreted by the boundary lubrication effect of water and the cooling effect of water.^{1,2,16,18} Moreover, it has been reported that the water connected to macromolecules by hydrogen bonds might cause changes in properties;³⁸ the lowered friction coefficient of a PI composite in the presence of water as a lubricant medium is related to the polar imide groups in the PI structure, which is liable to adsorb water and leads to a reduction in the strength of the surface layer. In addition, water also acts as a cooling agent to abate the friction-induced thermal and mechanical effects, which may increase the friction fluctuation by enhancing the softening and plastic deformation of the polymer matrix.^{1,18,19}

The steady-state friction coefficients of PI composites with different fiber contents under dry sliding and water lubrication are shown in Figure 5. The friction coefficients of the CF/PI composites sliding against stainless steel under dry sliding and water lubrication decreased gradually with increasing CF content. In contrast, the friction coefficients of the GF/PI and QF/PI composites under dry sliding increased with increasing fiber content. The friction coefficients of the GF/PI composites under water lubrication underwent no obvious changes with increasing fiber content. Although the friction coefficients of the QF/PI composites under water lubrication exhibited an irregular change with increasing fiber content in comparison with neat PI, there were no significant differences in the friction coefficients.



Figure 6 Variation in the wear rate of PI composites with the fiber contents under (a) dry-sliding and (b) water-lubricated conditions.

Journal of Applied Polymer Science DOI 10.1002/app



Figure 7 SEM micrographs of the worn surfaces of neat PI and counterpart steel: (a) PI and (b) steel under dry sliding and (c) PI and (d) steel under water lubrication.



Figure 8 SEM micrographs of the worn surfaces of CF/PI (15 wt % CF) and counterpart steel: (a) CF/PI and (b) steel under dry sliding and (c) CF/PI and (d) steel under water lubrication.



Figure 9 SEM micrographs of the worn surfaces of GF/PI (15 wt % GF) and counterpart steel: (a) GF/PI and (b) steel under dry sliding and (c) GF/PI and (d) steel under water lubrication.

The PI composites reinforced with different fibers exhibited lower friction coefficients under water lubrication than under dry sliding (except for the PI composite with 5 wt % QF). Moreover, the CF/PI composites showed lower friction coefficients than the GF/ PI and QF/PI composites, and this indicated that CF is superior to other fibers as the reinforcement phase.

Figure 6 shows the wear rate of PI composites with different fiber contents under dry sliding and water lubrication. For the composites under the ambient dry-sliding condition [Fig. 6(a)], the CF/PI composites exhibited a gradual decline in the wear rate with the fiber content increasing. However, the GF/PI and QF/PI composites showed a continuous rise in the wear rate with the fiber content increasing; in particular, the latter gave an abrupt increase in the wear rate as the fiber content increased beyond 15 wt %. For the composites under water lubrication [Fig. 6(b)], the CF/PI also showed a similar decrease in the wear rate, and the GF/PI and QF/PI gave an increase in the wear rate with the fiber content increasing at a slow pace. The CF/PI composites had the lowest wear rate in comparison with GF/PI and QF/PI because of the excellent mechanical properties of CF/PI. The CF/PI with 30 wt % CF gave a wear rate of $1.15 \times 10^{-6} \text{ mm}^3/\text{N} \text{ m}$ under the water-lubricated condition.

SEM micrographs of the worn surfaces of PI composites

The worn surfaces of neat PI and fiber-reinforced PI composites after dry sliding and water lubrication at a linear velocity of 0.43 m/s with a load of 200 N for 120 min as well as the corresponding stainless steel counterparts were investigated with SEM micrographs. Figure 7 shows the SEM micrographs of the worn surfaces of the neat PI and its stainless steel counterpart. The worn surface of PI under dry sliding is characterized by severe plastic deformation and microcracking [Fig. 7(a)]; a large amount of transferred PI debris can be observed on the stainless steel counterpart surface [Fig. 7(b)]. In contrast, the worn surface of PI is characterized by slight scuffing in sliding against the stainless steel under the water-lubricated condition and is smoother than that under dry sliding, and no signs of PI transfer can be observed on the stainless steel counterpart surface [Fig. 7(c,d)].¹⁶ It is suggested that water inhibited the transfer of PI onto the metal counterface.^{1,19,20} Lancaster¹ also found that fluids such as water, sea water, and other solutions inhibit the formation of transfer films of fiber/polymer debris on the counterface, and this results in a greater wear rate than that under dry conditions. Although PIs of



Figure 10 SEM micrographs of the worn surfaces of QF/PI (15 wt % QF) and counterpart steel: (a) QF/PI and (b) steel under dry sliding and (c) QF/PI and (d) steel under water lubrication.

an amorphous structure and limited ductility might experience melting at high pressure velocity values, the cooling effect of water hindered the melting of the PI surface layer and inhibited the transfer and generation of wear debris on the sliding counterpart steel surface. In addition, the water cleaning effect also prohibited the gathering of wear debris on the surfaces. Therefore, a smoother worn surface and lower wear rate of neat PI was observed under water lubrication in comparison with those under dry sliding. This observation is in accordance with what has been reported elsewhere.^{16,39}

Figures 8–10 show SEM micrographs of worn surfaces of PI composites with 15 wt % short fibers and the stainless steel counterparts under dry sliding and water lubrication, respectively. All the composites were characterized by plastic deformation, microcracking, scuffing, and spalling under both dry and water-lubricated conditions. Such plastic deformation, microcracking, scuffing, and spalling, however, were significantly abated under the waterlubricated condition. In comparison with the worn surfaces of composites under dry sliding, we found that the worn surfaces of composites under the water-lubricated condition were smoother [Figs. 8(c), 9(c), and 10(c)]; the polymer matrix combined the fibers well, and some of the protruded fibers were thinned and fractured. It is suggested that the PI composites possessed better wear resistance under the water-lubricated condition. The result agrees with that we deduced from the investigation of the friction and wear properties. In addition, comparing the worn surfaces of CF/PI composite with those of GF/PI and QF/PI composites under dry sliding [Figs. 8(a), 9(a), and 10(a)], we found that the former showed only slight microcracking and spalling; in contrast, the latter exhibited severe cracking and spalling. The indestructible worn surfaces of CF/PI composites under latter both dry and water-lubricated conditions could be attributed to the improved wear resistance.

The worn surfaces of the counterpart steel rings appeared to be smooth and showed some small grooves as well under dry sliding [Figs. 8(b), 9(b), and 10(b)], which indicated that a transfer film was formed on the counterpart steel ring surface. However, the worn surfaces of the counterpart steel rings showed many grooves and were dominated by severe scuffing under water lubrication, especially for GF/PI and QF/PI composites. This observation is in accordance with what has been reported previously: GF and QF are easily abraded and broken when sliding against metals, and the broken fibers transfer to the mating metal surface and increase the surface roughness of mating stainless steel in a water environment.⁴⁰ This is probably the governing or primary cause of the increased wear rate of GF/PI and QF/PI composites under the water-lubricated condition. In this case, the slightest scuffing on the worn surface of the steel ring of the CF/PI composite accounts for its excellent wear resistance.

CONCLUSIONS

First, short-CF-reinforced PI composites (CF/PI) had improved tensile and flexural strength and tribological properties in comparison with GF/PI and QF/PI composites. The incorporation of CF into PI contributed to decreases in the friction coefficients and wear rates in sliding against stainless steel under both dry and water-lubricated conditions.

Second, the initial friction coefficients of a PI resin and its composites were relatively high under dry sliding but decreased abruptly with the sliding time because of the formation of a transfer film on the steel surface and finally reached a steady state of 0.2–0.4. On the other hand, the initial friction coefficients of the PI resin and its composites were relatively low under water lubrication but increased and then decreased to a steady state in the range of 0.2– 0.3.

Third, the PI composites showed better friction reduction and wear resistance under water lubrication than under dry sliding because of the boundary lubrication effect of water. PI and its composites were characterized by plastic deformation, microcracking, and spalling under both dry and waterlubricated conditions, which were significantly abated under the water-lubricated condition.

Fourth, GF and QF were easily abraded and broken when sliding against metals; the broken fibers transferred to the mating metal surface and increased the surface roughness of the mating stainless steel, which probably increased the wear rate of the GF/PI and QF/PI composites.

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