

The Tribological Properties of Poly(*p*-phenylene benzobisoxazole) Pulp Reinforced Friction Materials

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ABSTRACT: Automotive friction materials reinforced by home-made poly (*p*-phenylene benzobisoxazole) (PBO) pulp (fibrillated organic fibers) were prepared through compression molding. The friction and wear behaviors of the obtained composite materials were evaluated using a constant rotating speed type friction tester. The PBO pulp content and the testing loads showed clear influence on the tribological properties of the composites. Friction stability, wear rate, and morphology of sliding surfaces were carefully examined to investigate the effect of the pulp ingredient in the friction materials. Scanning electron microscopy was employed to study the morphology of the surface and wear particles. The significant wear reduction was achieved when the mass fraction of PBO pulp was 3%. Wear rates of the composites with 3% PBO pulp were measured over a load range from 0.3 to 1 MPa at different temperatures. The results pointed to two facts: (1) the wear rate of the friction material increased linearly with load at low temperature (below 200°C); (2) wear status varied with the testing loads at high temperature (above 250°C). © 2013 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 130: 4032–4039, 2013

KEYWORDS: PBO; composites; functionalization of polymers; mechanical properties

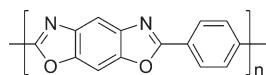
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INTRODUCTION

Tribology is the science and engineering of interacting surfaces in relative motion. It includes the study and application of the principles of friction, lubrication, and wear. The main parameters of kinetic friction are friction coefficient, wear rate, and physical stability during the sliding process. The brake performance of a vehicle is a typical sliding friction and is primarily determined by the tribological properties of a friction couple, which consists of a gray cast iron rotor and friction materials.^{1–3} Phenolic resin-based friction materials are nonasbestos and widely used in automobile and aviation industries, due to their high specific strength, low density, and good cost-effectiveness of raw materials. However, the tribological application of phenolic resin-based friction materials is usually limited because of the relatively poor stability and poor wear resistance.⁴ To overcome this disadvantage, among many ingredients currently used in the friction materials, reinforcing fibers are introduced to play important roles in determining the friction characteristics during braking. Fiber-reinforced polymer composites are one of the most common products and are found to possess unique self-lubrication capabilities and low noise as tribological materials.⁵ Recently, special fiber pulp is employed as a new fiber species to improve the performance of the friction materials⁶ due

to its thermal stability and good distribution. As an originally fibrillated product, special fiber pulp has a superior adsorption capacity. In addition, it is relatively easy to be dispersed uniformly in a polymer matrix because of the small size, and its high L/D (length to diameter) ratio also contributes to the reinforcement. These advantageous physical and mechanical properties make special fiber pulp an ideal substitution of asbestos and metal fibers in the areas of friction, seals, and other applications. It has been revealed that the fiber pulp can increase the strength (modulus) and consequently the physical/mechanical properties of the final component.⁷ After the middle of 1990s, special fiber pulp was developed and applied rapidly because of the prohibition of asbestos for environmental protection. Among many investigations, Aramid pulp attracted particularly attention and showed good filler retention as well as a processing aid, which provided sufficient green strength for a preform.^{8–12} Aramid pulp reinforced friction materials showed excellent friction and wear properties due to the unique heat resistant and high dispersibility of Aramid pulp,^{13–15} indicating aramid pulp can be served as an ideal substitution of asbestos. In the presence of aramid pulp, the friction materials can achieve lower friction coefficient, lower wear, and enhancing friction stability.^{7,16,17}



Scheme 1. The chemical structure of PBO.

As a kind of rigid-rod-like polymer, poly(*p*-phenylene benzobisoxazole) (PBO) has received great interest because of its excellent mechanical properties, and good thermal and chemical stability.^{18–25} The chemical structure of PBO is shown in Scheme 1. As compared to the widely used aramid fiber namely Kevlar, PBO in the fiber form has twice tensile strength and modulus reaching to 5.8 and 270 GPa, respectively. Besides, the thermal stability and dimensional stability of PBO are also much better than those of Kevlar. It can be expected that using PBO pulp as a reinforcing component can significantly enhance the properties of friction materials. Although the tribological properties of PBO film sliding against steel have been studied,²⁶ there are few results on the tribological characteristics of PBO pulp reinforced friction materials.

In this article, we studied the tribological behavior of PBO pulp reinforced automotive friction material under different temperatures and pressure. The effects of filler content were systematically investigated and the wear mechanism of the composites was discussed based on the SEM results of the worn surfaces under different loads.

EXPERIMENTAL

Sample Formulation and Preparation

PBO was synthesized following the procedures described in Ref. 27 and the intrinsic viscosity was 32 dL/g. PBO fibers were self-made by the dry-jet wet-spun method with average diameter 20 μm and tensile strength 4.52 GPa. The fibers were used for the preparation of PBO pulp via precipitation. Briefly, a concentrated solution of PBO fibers in methanesulfonic acid (MSA) was injected into a rapidly stirring coagulant and then the PBO pulp was produced. The specific surface area, length-diameter ratio, and average length are the main performance parameters of the precipitated pulp and the key factors, which influence these parameters, are including the concentration of the MSA/PBO solution, the stirring speed, the coagulant liquid and the temperature of the coagu-

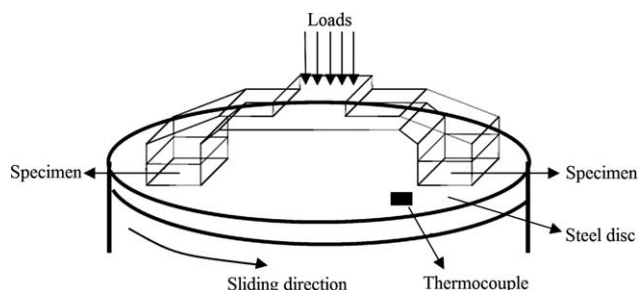


Figure 1. A schematic illustration of the constant rotating speed type friction tester.

lant. A PBO/MSA solution with 3 wt % PBO, distilled water as coagulant, and a stirring speed of 3000 rpm at room temperature were found to be the best for the precipitation. Under these conditions, the precipitated PBO pulp with high specific area and excellent surface morphology could be achieved. The raw materials of friction specimens used in this study are phenolic resin, PBO pulp, steel wool, barium sulfate, zirconium silicate, coke, and graphite. The mass fraction of the ingredients and the physical properties of the final specimens after mixing and molding are shown in Table I. Coke and graphite, BaSO₄, and zirconium silicate were used as fillers and friction modifiers in the composite. Because all the amounts of the ingredients are the same except PBO pulp and BaSO₄, to distinguish the specimens, we named the samples as “PBO Number” and the number is the mass fraction of each composite. In this way, a varied PBO pulp content of 1, 3, and 5 wt % were applied in the preparation of friction material and the corresponding composites were designated as PBO 1, PBO 3, and PBO 5, respectively. Similarly, the sample without PBO pulp was named as PBO 0.

The manufacture of friction materials consists of mixing, pre-forming, hot press molding, and postcuring. All ingredients were mixed in an electric mixer (Ele® EDF-400) for 5 min. The mixture was preformed at 100°C under 10 MPa for 2 min before it was molded at 150°C under 12 MPa for 5 min. Post-curing was carried out using a mechanical convection oven at 180°C for 5 h. The size of friction material specimens for friction tests was 25 × 25 mm² × (5 ± 2) mm. The counter disk used in the friction test was gray cast iron and the hardness of the counter disk was ~200 (Brinell hardness).

Table I. Composition and Physical Properties of Friction Materials

Raw material	PBO 0	PBO 1	PBO 3	PBO 5
PBO pulp	0	1	3	5
Phenolic resin	15	15	15	15
Steel wool	47	47	47	47
BaSO ₄	22	21	19	17
Zirconium silicate	7	7	7	7
Coke and graphite	9	9	9	9
Rockwell hardness (HB)	90.35 ± 4.12	94.63 ± 5.03	93.24 ± 3.43	95.18 ± 3.17
Density (g/cm ³)	3.87	3.67	3.51	3.33

Table II. Test Procedure Used in This Study

Test mode 1	Testing temperature:100, 150, 200, 250, 300, 350°C, testing specimens: PBO 0, PBO 1, PBO 3, PBO 5, applied normal load: 1 MPa, sliding velocity: 480 rpm, disk rotation: 5000
Test mode 2	Testing temperature:100, 150, 200, 250, 300, 350°C, testing specimens: PBO 0, PBO 3, applied normal load: 0.3, 0.4, 0.5, 0.6, 0.7, 0.8, 1 MPa, sliding velocity: 480 rpm, disk rotation: 5000

Measurement and Analysis

The hardness of the friction composites was measured using an optical hardness tester at a Brihell Rockwell Vickers scale. Each reported result is the averages of 8 different spots of the corresponding specimen.

The morphologies of PBO pulp, worn surfaces, and the particles left after testing were examined by using TESCAN scanning electron microscope (Model TS5136MM). SEM samples were plated with gold coating before examination.

Thermalgravimetric analysis (TGA) was carried out in air on a DuPont 1090B thermal gravimetric analyzer with a heating rate of 10°C min⁻¹.

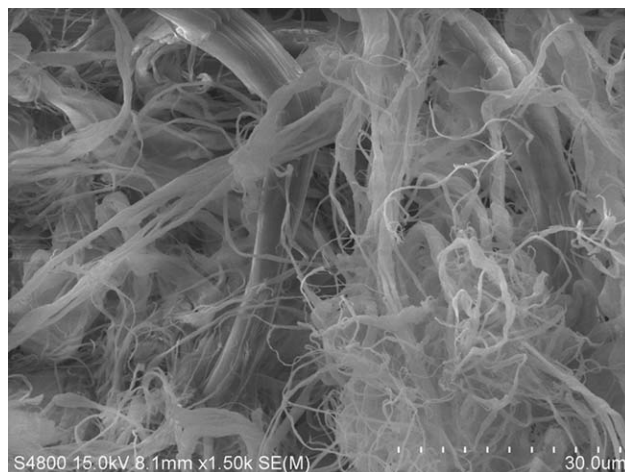
The specific surface area was measured using surface area and pore size analyzer (Model JW-BK132F) according to the BET (Brunauer-Emmett-Teller) theory.

The tribological measurements were performed in a pad-on-disk type friction tester with constant rotating speed. The schematic diagram of the tester is shown in Figure 1. The surface temperature of the rotating disk was measured by a contacting thermocouple placed at the radius of the disk specimen, and the monitored data including friction force and disk temperature were recorded by PC. The rotating speed was set at 480 rpm and the applied pressure was controlled by the weighs connecting to the pressure-add bearing. The friction and wear characteristics were analyzed according to the test mode 1 shown in Table II. Test mode 2 in Table II was employed to study the influence of load on the friction materials. All the friction tests were repeated twice under each test condition: the first test was to confirm that the friction materials were fully burnished whereas the second test was used for data analysis.

The wear rate of the friction material was obtained by measuring the change of the thickness and weight of the friction material after each wear test. The wear rate, V is calculated by the eq. (1):

$$V = 1.06 \frac{A(d_1 - d_2)}{N \cdot f_m} \quad (1)$$

where N is the number of disk rotation and in this test, $N = 5000$. A is the area of the testing specimen, f_m is the recorded friction force during the test, d_1 is the average thickness before the test, and d_2 is the average thickness after the test.

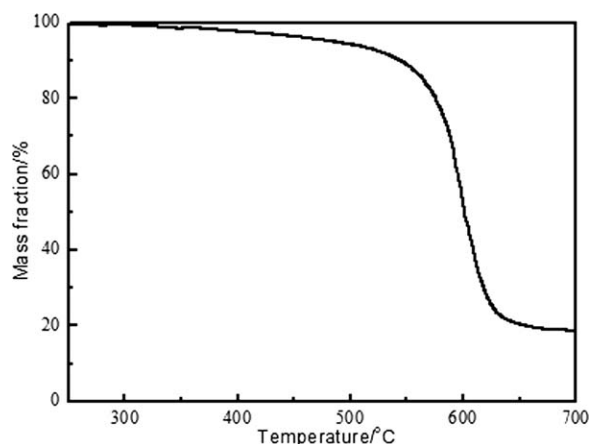
**Figure 2.** Scanning electron microscope (SEM) photographs of PBO pulp.

RESULT AND DISCUSSION

Morphology and Thermal Stability of PBO Pulp

The SEM image of the PBO pulps is displayed in Figure 2. It can be seen that the PBO pulps had a ribbon-like structure with many ramifications and different thickness and lengths of the fibrils, but the size of the fibrils was large. The PBO pulps were fibrillated only by high shear stirring and were not mechanically refined to reduced diameter. The typical PBO pulps had a diameter distribution of 7–20 μm, a length distribution of 0.5–2.5 mm, a specific surface area (SSA) distribution of 5–12 m²/g. A relatively large SSA was achieved for the PBO pulp, which made the PBO pulp adhere easily to the phenolic resin in the mixing process. Consequently, a homogeneous dispersion of all the content was attained in composites.

TGA was performed to study the thermal degradation behavior of the PBO pulp in air and the results are shown in Figure 3. It can be seen that the pulp had an outstanding thermal stability, because no significant mass change was observed before 550°C. Hence, the thermal stability of the friction materials with PBO pulp was expected to be improved to a great extent.

**Figure 3.** Thermalgravimetric analysis (TGA) curve of PBO pulp in the air.

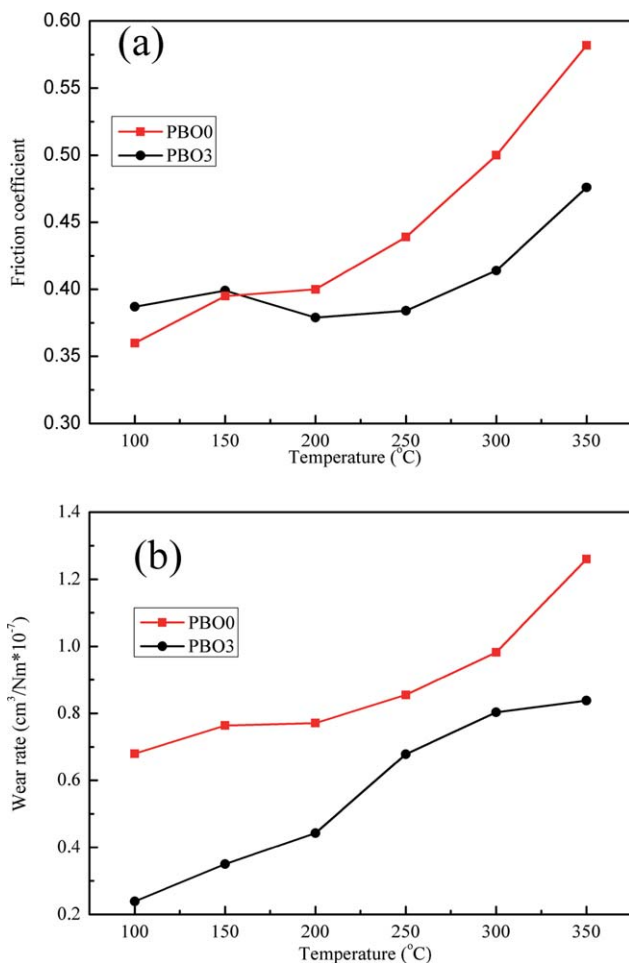


Figure 4. Friction coefficient (a) and wear rate (b) of friction materials as a function of temperature for PBO 0 and PBO 3. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Frictional and Wear Properties of Friction Materials with and Without PBO Pulp

The friction coefficient as a function of temperature was compared for the phenolic resin composites PBO 0 and PBO 3 in order to investigate the reinforcement effect of PBO pulp, and the results are shown in Figure 4(a). It can be observed that the friction coefficient for PBO 0 without pulp changed obviously with the increase of temperature, and had highest value of 0.58. The addition of PBO pulp increased the friction coefficient in low temperatures. The friction coefficient kept stable even at a relatively high temperature up to 350°C. The enhancement was due to the presence of PBO pulp. The PBO pulp, which had relatively high surface area, was thought to be easily dispersed because of the hairiness and comparatively high fibrillation. The other ingredients, especially the phenolic resin, can be adsorbed onto the PBO pulp leading to high dispersion in the matrix, which consequently formed a more stable material structure.

The wear property of a multiphase friction material depends on many factors such as temperature, applied load, sliding velocity, and the properties of friction film.^{28–31} The influence of one of the factors namely temperature was studied over PBO 0 and PBO 3 and the results are shown in Figure 4(b). The wear rate

of PBO 0 increased with temperature and reached a value of $1.31 \text{ cm}^3/\text{Nm} \times 10^{-7}$ at 350°C. As compared to PBO 0, PBO 3 showed lower and less changed (more stable) wear rate at lower temperatures, which reached a value of $0.82 \text{ cm}^3/\text{Nm} \times 10^{-7}$ at 350°C. The results suggested that the PBO pulp was beneficial for the wear characteristics of the friction material, which was due to the higher cohesive strength of the friction film resulting from the more dispersed ingredients in the presence of PBO pulp. The wear rate for PBO 3, although increased at high temperatures, was much lower than PBO 0 because of the better dimensional stability. The results also indicated the importance of PBO pulp for building durable friction films with high thermal stability. The size and shape of the wear debris was also likely to affect the wear rate, because the flake particles with no PBO pulp had shorter lifetime and transformed to fine powders on the friction surface during sliding due to the insufficient strength and low thermal stability.

The friction behavior and wear resistance of the friction materials on the rubbing surface were also studied (Figure 5). The well-developed friction material of PBO 3 displayed improved physical stability, resulting in a steady friction coefficient at high temperatures (above 250°C). Whereas the friction material

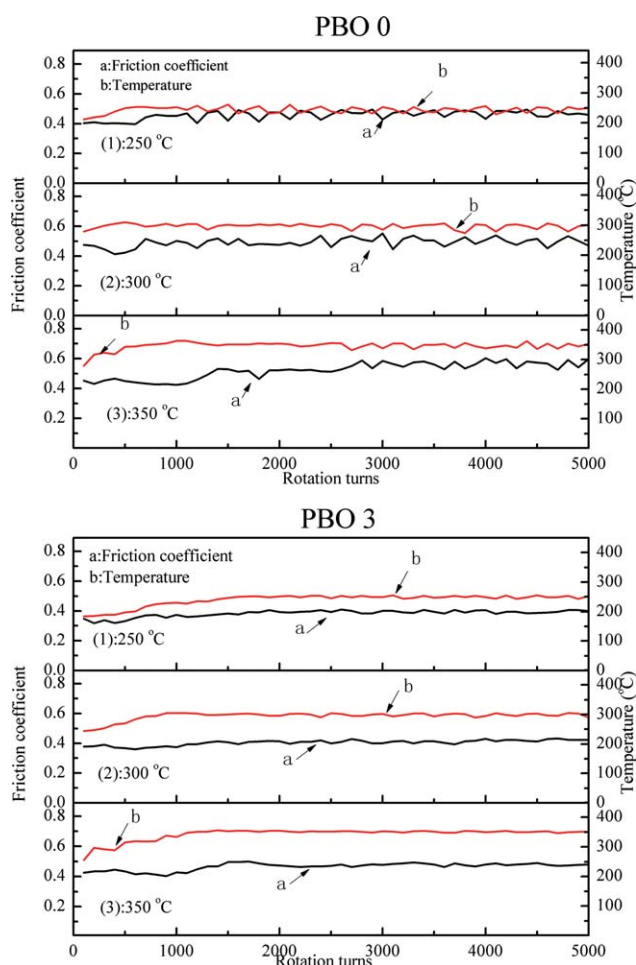


Figure 5. Instant friction coefficient of PBO 0 and PBO 3 samples at different temperatures. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

of PBO 0 showed unstable friction coefficient during the 5000 sliding turns (significant fluctuation phenomena). The significant fluctuation observed in the case of PBO 0 was attributed to the lack of reinforcing fibers, which can integrate other constituents and provide heat resistance.

It is known that friction film chemistry is strongly affected by the ingredients of the material, and reinforcing fibers and solid lubricants play a decisive role in the properties of friction films. Since, the simplified experimental formulation used in this study contained only seven ingredients; the constituent that may affect the formation of friction film was not complicated. The compaction process seemed similar to the molding of raw material ingredients in the friction material manufacturing process and, therefore, PBO pulp played important roles for the strength of the friction film as reinforcements.

Influence of the Content of PBO Pulp to the Frictional and Wear Property of Friction Materials

As the beneficial effect of PBO pulp is proved, the optimal content in the friction materials is still of great importance for further application. The frictional behavior of the material with varied PBO pulp content was tested and results are shown in Figure 6(a). It can be seen that the frictional coefficient increased continuously with the PBO content up to 3 wt % at all the temperature. The friction coefficient became irregular when the content reached 5 wt %, which increased with the increase of PBO pulp content at lower temperatures (below 200°C) and decreased significantly with the increase of PBO pulp content at higher temperatures (above 250°C). The possible reason is, on one hand, the density of PBO pulp was low, leading to rapid increase of volume percentage of PBO pulp and diminished the adhesion with the resin, so that it was easy to be peeled off, and resulted in the wear resistance degradation. On the other hand, transfer membrane formed on the surface during the friction if extra pulp was added, which decreased the roughness on the frictional surface so that the frictional force and the friction coefficient decreased.

The wear rates of the friction materials with different PBO pulp content were measured at different temperature, and the results are shown in Figure 6(b). It can be seen that the frictional property and the abrasive resistance increased along with the increase of the PBO content from 0 to 3 wt %. Irregular change of the wear rate was observed when the pulp content was as high as 5 wt %. A significant jump of PBO 5 was observed when temperature higher than 200°C and the friction coefficient reached a value of $1.6 \text{ cm}^3/\text{Nm} \times 10^{-7}$ at 300°C, then drop to the value of $1.13 \text{ cm}^3/\text{Nm} \times 10^{-7}$ at 350°C. It can be assumed that for samples with high pulp content tiny abrasive particles formed from the peeled fiber and consequently resulted in negative effect to the wear property of the materials.

The friction and wear of the brake friction material is strongly affected by the heat resistance of the ingredients. The friction film is basically a layer of compacted wear particles originated from the friction material and the gray cast iron. Therefore, the properties of the friction film are primarily determined by adhesive strength of the constituents from the friction couple at a sliding condition. Once the transfer film forms at the friction

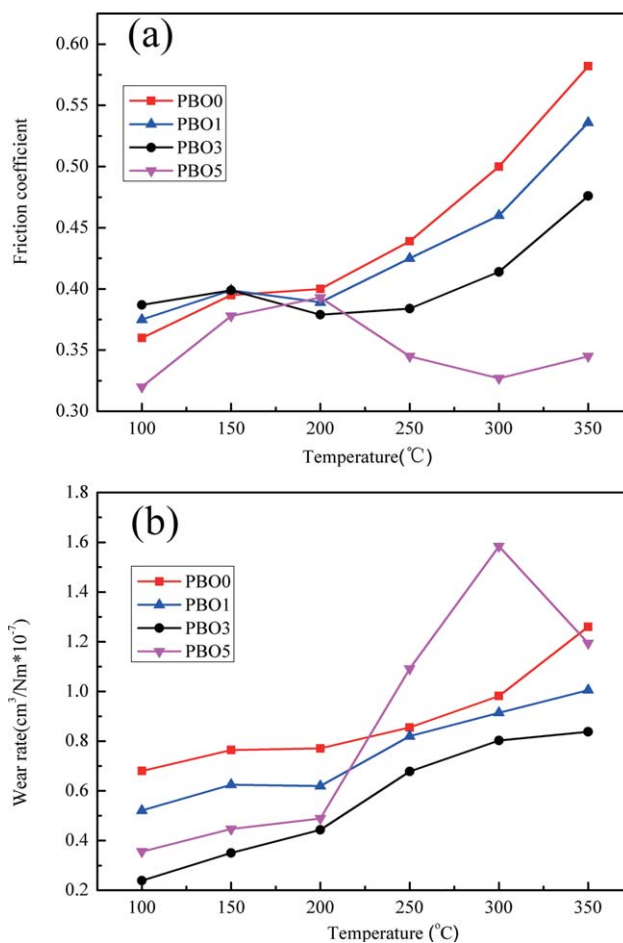


Figure 6. Effect of the PBO pulp content on friction coefficient (a) and wear rate (b) at different temperatures. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

interface during sliding, friction behavior will be determined by the properties of friction film. PBO pulps in proper content played a key role in forming the excellent friction film in this article due to its thermo stability, adsorption capacity, and dimensional stability.

Influence of the Applied Pressure to the Frictional and Wear Property of Friction Materials Reinforced by PBO Pulp

The wear rate of PBO 3 was also tested with varied loads ranged from 0.3 to 1 MPa and varied temperature ranged from 100 to 350°C. The results are shown in Figure 7 and revealed that the wear rates increased almost linearly with the applied pressure at low temperatures [below 200°C, Figure 7(a)]. More complicated behaviors were presented when the temperatures were higher than 250°C [Figure 7(b)]: (1) at low loads (under 0.6 MPa), wear rate of the PBO 3 sample showed an evolutionary behavior similar to that of the nonreinforced sample. This is probably because the stresses were lower than the particle fracture strength in this regime, the main factor to control the friction behavior was the ductility of steel fiber. The pulp as reinforcement was not significant, the wear behavior of both nonreinforced specimen and pulp-reinforced specimen showed the same trend. (2) In this regime (loads between 0.6 and 0.8

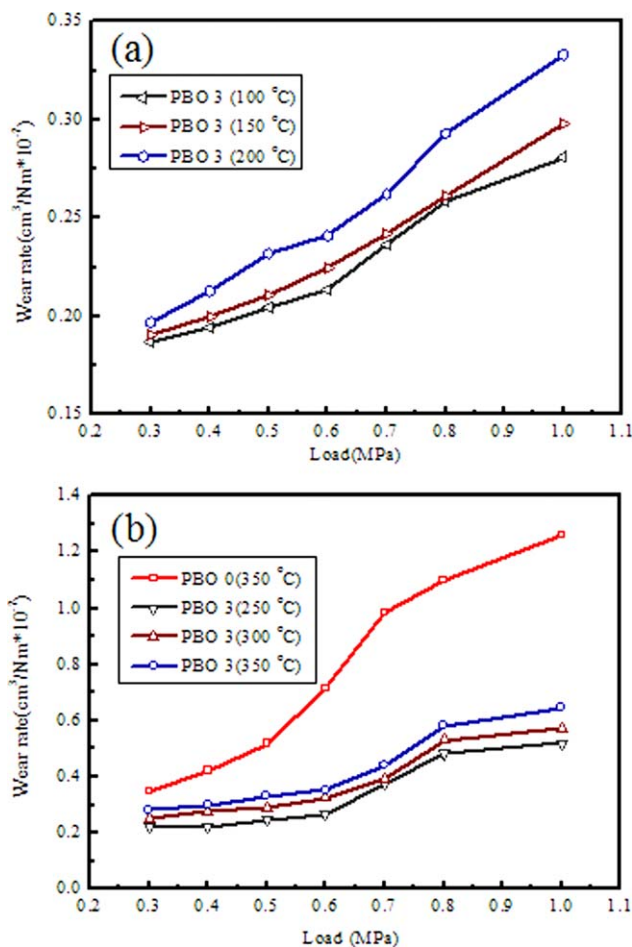


Figure 7. Variation of the wear rate with the applied load (a) at low temperatures (below 200°C) and (b) high temperatures (above 250°C). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

MPa), PBO pulp reinforced composites showed much lower wear rates than those of the unreinforced ones, because the PBO pulp with highly dispersed and strong adsorption capacity acted as load-bearing elements and their abrasive action on the metal counter face lowered the transition of metal-rich layers onto the contact surfaces. Moreover, a subsurface delaminating process by decohesion of pulp-matrix interfaces tended to control the wear, resulting in the trend of wear rates more stable than those in the nonreinforced matrix specimens. (3) Above a critical load (over 0.8 MPa), the wear behavior was determined by the size and volume fraction of steel fiber particles, and the spalling PBO pulp with high temperature stability acted as the self-lubrication particles on the contact surface, leading to a less increasing of the wear rates.

Specimens Surface Morphology

Figure 8 shows the scanning electron micrographs of the worn surface of the nonreinforced and the pulp-reinforced composites sliding under certain conditions. Nonreinforced specimen showed irregular worn surfaces exhibiting detached regions with trace of mass peeling and rough friction surface [Figure 8 (a)]. As compared to the nonreinforced specimen, PBO 1 and PBO 3

with PBO pulp had a more smooth friction surface, which can be related to the well dispersion and well adhesion of the PBO pulp and other ingredients by the blending process. The key role of these fibrous ingredients in the friction material produced durable and smooth friction surfaces [Figure 8(b,c)]. Moreover, PBO 1 showed a delaminated worn surface with traces of peeled off plates and with probe pulp content, PBO 3 with the smoothest surface suggested excellent capability of filler retention of PBO pulp [Figure 8(c)].

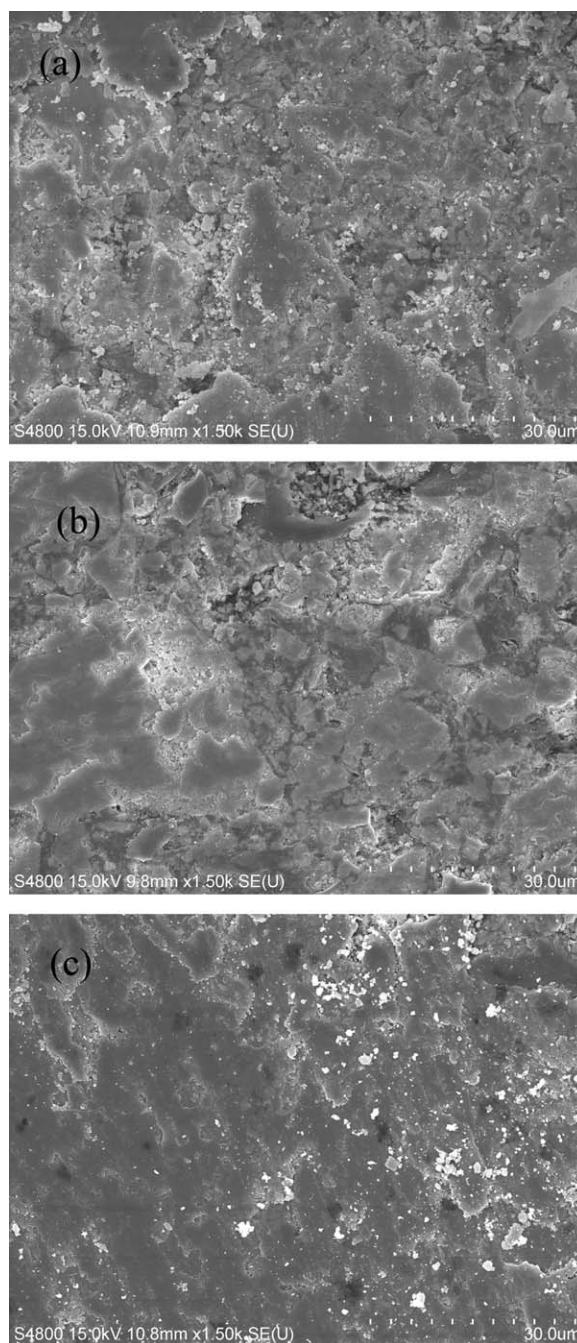


Figure 8. SEM photographs of the worn surface of the nonreinforced and the pulp-reinforced composites sliding at 1 MPa and 350°C. (a) PBO 0, (b) PBO 1, and (c) PBO 3.

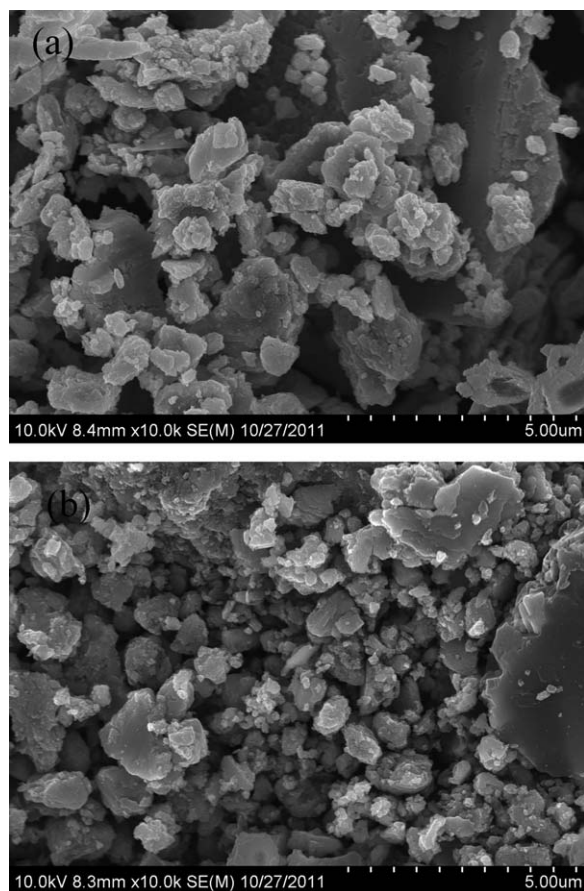


Figure 9. SEM micrographs of wear particles showing different morphologies: (a) PBO 3 and (b) PBO 0.

Figure 9 shows wear particles collected after friction tests at 350°C. As expected, the wear particles collected during the friction tests showed the same trend. The friction material with 3 wt % PBO pulp showing high wear resistance [Figure 9(a)] exhibited a plate shape wear debris, whereas the friction material without PBO pulp showing low wear resistance [Figure 9(b)] produced fine spherical wear particles. The wear rate of the friction material appeared closely related to the friction film and the wear debris formation during the test. The SEM figures also strongly suggest that the size and the shape of the wear debris were possibly linked with the wear resistance and other important friction characteristics.

CONCLUSIONS

In summary, PBO pulp used as a reinforcement component of friction material led to high and more stable friction coefficient and to lower wear rate, especially in high temperature conditions. The friction coefficient of the reinforced friction material kept stable along with the increase of the PBO pulp content up to 3 wt %. However, higher pulp content led to a reduced stability of the friction material. Wear rates of the composites related to the pulp content showed the same trend as the friction coefficient. The wear rate decreased along with the increase of PBO pulp content up to 3 wt %, but if the content exceeded a certain percentage, the wear performance became worse.

The friction film with proper PBO pulp maintained smooth friction surface throughout the test, and the durable transfer film resulted in improved wear resistance and steady friction force. The effect of PBO pulp on the sliding wear resistance of the friction material varied with the applied pressure. The reinforcement of PBO pulp was proved to be effective in suppressing the spalling behavior of the composites and under different loads and temperatures, the pulp-reinforced materials acted in different mechanism. PBO pulp reinforced the friction material leading to excellent wear resistance and stable friction coefficient under different sliding conditions, indicating that PBO pulp can be used as a potential reinforcement ingredient of the friction materials for practical applications.

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