Tribological Behavior of Polyetheretherketone Composites Containing Short Carbon Fibers and Potassium Titanate Whiskers in Dry Sliding Against Steel

N. Ma, G. M. Lin, G. Y. Xie, G. X. Sui, R. Yang

Institute of Metal Research, Chinese Academy of Sciences, Shenyang 110016, China

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ABSTRACT: Polyetheretherketone (PEEK) composites reinforced by short carbon fibers (SCF) and potassium titanate whiskers (PTW) were prepared using twin-screw extrusion compounding and injection molding. The tribological properties of hybrid composites were investigated in dry sliding condition against steel. The effects of filler contents on the wear behavior were studied. It was found that the hybrid composite showed an excellent tribological property in dry sliding condition. Applied load had great effect on the tribological behavior of the composites. In most cases, the friction coefficient of the composite decreased with the load rising. The composites with higher CF contents showed outstanding tribological performances at low load but could worsen the wear behavior at high load. Because of the positive effect of PTW, high PTW loading composites presented low wear rate at low load. At high loads, the composites with lower PTW contents had better wear resistance. The scanning electron microscopy (SEM) observation revealed that abrasion wear was attributed to the lower wear resistance of the high PTW content composite at high load. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 123: 740–748, 2012

Key words: PEEK hybrid composite; tribology; carbon fiber (CF); potassium titanate whiskers (PTW); applied load

INTRODUCTION

Polyetheretherketone (PEEK) is a high performance, semicrystalline thermoplastic with outstanding thermal and mechanical properties. It has a high glass transition temperature (143°C) and a high continuous service temperature (250°C) with the advantage of easy process ability by injection molding. Compared with other polymers usually used in engineering such as nylon, ABS and polytetrafluoroethylene (PTFE), PEEK has excellent properties. Therefore, PEEK has been considered as one of the most ideal material for tribological applications. In some severe instances such as in nonlubrication, moist and corrosive conditions, PEEK is applied as journal bearings to replace the traditional metallic materials.[1] When the increase in interfacial temperature can be neglected, the friction coefficient of PEEK is independent of sliding velocity and contact pressure. However, a temperature increase corresponds to high friction coefficient.[2] Under some operating parameters, the friction coefficient of PEEK is as high as 0.7, which limits its utility as an antifriction material. It has been reported that the reinforcement of some fibers and inorganic particles has a beneficial effect on the strength and tribological properties of PEEK.[3–8] To reduce the friction coefficient, lubricants such as PTFE[2,9–13] and graphite[2,13] were also frequently incorporated.

Carbon fibers as a candidate of PEEK to improve tribological performance are more effective than glass fibers.[3,14] The reinforcement of carbon fibers could greatly advance the mechanical and tribological properties of PEEK even under elevated temperature[15] and water condition.[4,16] Both the friction coefficient and the wear rate decrease because of graphite lubrication from the carbon fiber debris.[17] However, an increase in testing temperature (above 150°C) could result in high wear rates and low friction coefficients for PEEK-30CF.[15] Although over a wide range of temperature, short carbon fiber reinforcement improves the wear resistance of PEEK, more than 20 vol % carbon fibers could cause stick-slip behavior, especially at very high testing temperatures.[12] Xu et al.[18] used artificial neural networks to predict the tribological behavior of PEEK-CF30 according to *pv* factor and contact temperature. The result shows that friction coefficient is influenced by the *pv* factor, and the wear rate is influenced by the contact temperature.

Whiskers were considered to be promising fillers to upgrade the properties of polymers.[19–22] Whiskers are generally recognized as being free from internal defects owing to their small diameters. Hence they exhibit high yield strength and high stiffness. Potassium titanate whiskers (PTW) show promise as

Correspondence to: G. X. Sui (gxsui@imr.ac.cn).

TABLE I
The Mass Ratios of PEEK/CF/PTW Composites

Materials	PEEK	CF	PTW	PTFE	Graphite
1	85	5	10	5	5
2	80	10	10	5	5
3	75	15	10	5	5
4	70	20	10	5	5
5	75	10	15	5	5
6	70	10	20	5	5

reinforcement materials owing to their good physical, mechanical properties, and relatively low cost. PTW could improve the tensile strength and impact strength of PTFE significantly.[23] The friction coefficient of PTW/PTFE composites decreases with the increase of PTW content and with 20 wt % PTW content, the best wear resistance occurs, which is over 1000 times larger than that of pure PTFE.[19] Zhuang et al.[24] found that the incorporation of PTW into PEEK could result in an increase in tensile strength and modulus with increasing PTW content within the used loading range until 30 wt %. It was reported that PTW could effectively reduce the friction coefficient and wear rate of the PEEK.[25,26]

Both CF and PTW are excellent short fibers reinforcement for the Polymer composites. The carbon fibers with a diameter of 7 μm have high strength and high hardness. In the dry sliding tribological application, the composite of high carbon fiber content could cause high frictional force followed by high temperature rise. The carbon fibers could also scratch the counterpart surface due to high hardness. However, Potassium titanate whiskers (PTW) with a diameter of 0.1–1 μm are of promise reinforcement and lower hardness than CF. The PTW can help to reduce the frictional force and can enhance the mechanical properties of the matrix but no to scratch the counterpart surface. Therefore, a combination of CF and PTW in the polymer composite is expected to produce good tribological properties for dry sliding condition. Recently, the tribological performances of some inorganic particles and short fibers hybrid reinforced composites were reported.[27–30] But the hybrid reinforcements of short carbon fibers and whiskers are few investigated by now.

In this work, the tribological properties of PEEK composite reinforced by both PTW and carbon fibers in dry sliding against steel were investigated and the roles of carbon fibers and PTW contents on the wear performance at dry sliding were studied.

EXPERIMENTAL

Materials

Commercially available PEEK and PEEK/30 wt %CF were provided by Degussa Polytetrafluoroethylene (PTFE) powder was provided by Jinan 3F Fluoro Chemical Co. Ltd. Graphite powder was produced by Sinopharm Chemical Reagent Co. Ltd. (particle size ≤ 30μm). Potassium titanate ($K_2Ti_6O_{13}$) whiskers (PTW) were supplied by Jinjian Co. Ltd. (Shenyang, China). The diameter of PTW was in the range of 0.1–1.0 μm. The density of the PTW was about 3.3 g/cm^3. The average diameter of short carbon fiber was about 7 μm.

Specimen preparation

PEEK and PTW were dried in an oven at 150°C for 6 h before blending in a twin-screw extruder with a screw speed of 360 rpm. The temperature of barrel from the hopper to the die was 360–365–370–380–385–390°C. The extruded strands were quenched in water followed by chopping into pellets. PTW/PEEK, CF/PEEK, PTFE, and graphite were dried at 150°C for 6 h for injection molding. PEEK/30 wt %CF was used as master batch to dilute to the composites during injection molding. PTFE and graphite were added as lubricants in all the PEEK/CF/PTW composites. Standard test bars were obtained through injection molding at mold temperature of 180°C. The mass ratios of PEEK/CF/PTW/PTFE/graphite (referred as PEEK/CF/PTW) composites are list in Table I.

Characterization

Wear tests were carried out on a MM-W1A universal wear testing machine (Jinan Shijin Co. Ltd.) with three-pin-on-disc configuration. The polymer pin (4.8 mm diameter and 12.8 mm length) and the counterpart ring (AISI 1045, hardness of HRC 44-55) were ground against the water-proof silicon carbide (SiC) abrasive paper of 1500 grade and ultrasonically cleaned in acetone before testing. The average roughness of the counterfaces (R_a) was 0.042 μm. All the tests were performed under dry friction and ambient condition at the sliding velocity of 2.0 m/s. The friction duration was 2 h. The loads applied were 0.5, 1.0, 2.0, and 4.0 MPa. During the tests, the friction data were recorded by computer every 2 s. The temperature change during the sliding was recorded by measuring the temperature of the counterpart ring. The final friction coefficient reported for the composite was the average value. Before and after tests, all the pins were ultrasonically cleaned and dried at 150°C for 6 h for weighing. Then the mass loss of the composite pins was measured by electronic balance (Mettler AE240, accuracy 0.01 mg) for the specific wear rate calculation. The special wear rate was calculated using the equation:

$$K = \Delta m / \rho F_N L$$

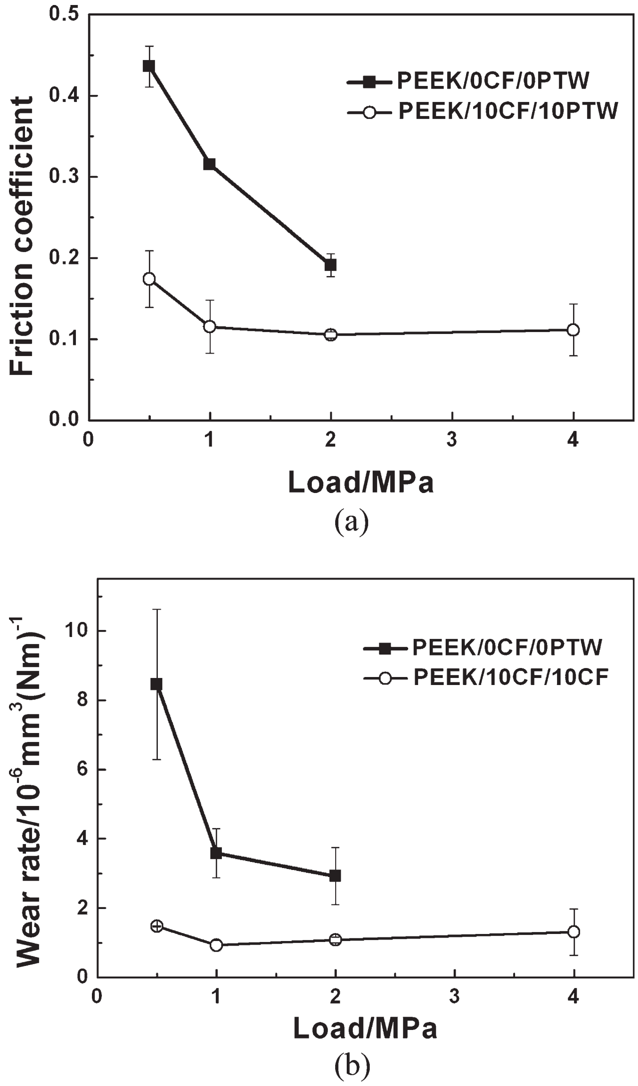


Figure 1 Effects of load on the friction coefficient (a) and wear rate (b) of PEEK/0CF/0PTW and PEEK/10CF/10PTW composites. Test conditions: sliding velocity, 2 m/s; sliding duration, 2 h.

where K is specific wear rate, Δm is the weight loss, ρ is the density of the sample, F_N is the applied normal load and L is the total sliding distance. The average of the three test results for each point was reported in this work.

The worn surfaces of the composite pins and the counterfaces were coated with a thin layer of gold and then investigated using a SSX-550 scanning electron microscopy (SEM).

RESULTS AND DISCUSSION

Effect of applying load

The results of the friction coefficient and wear rate of PEEK/0CF/0PTW and PEEK/10CF/10PTW composites at different loads are presented in Figure 1. Compared to PEEK/0CF/0PTW, PEEK/10CF/10PTW composite had excellent tribological behavior. It was evident that the friction coefficient of the composite decreased with the increasing applied load. For PEEK/10CF/10PTW composite, when the applied load increased from 0.5 MPa to 1.0 MPa, the friction coefficient changed dramatically from 0.174 to 0.115. Then, it decreased gently from 0.115 to 0.105 and slightly increased to 0.111 with the increasing load. The trend of the wear rate was similar to that of the friction coefficient. A dramatically reduction of wear rate could be observed when the applied load changed from 0.5 MPa to 1.0 MPa. Afterward, it gradually increased with the increasing load.

During the sliding process, the heat generation between two materials would lead to the rise of the temperature of the frictional surface. A higher pressure always resulted in a higher temperature rising. On the one hand, when the load increased, the shear stress of the materials decreased with the

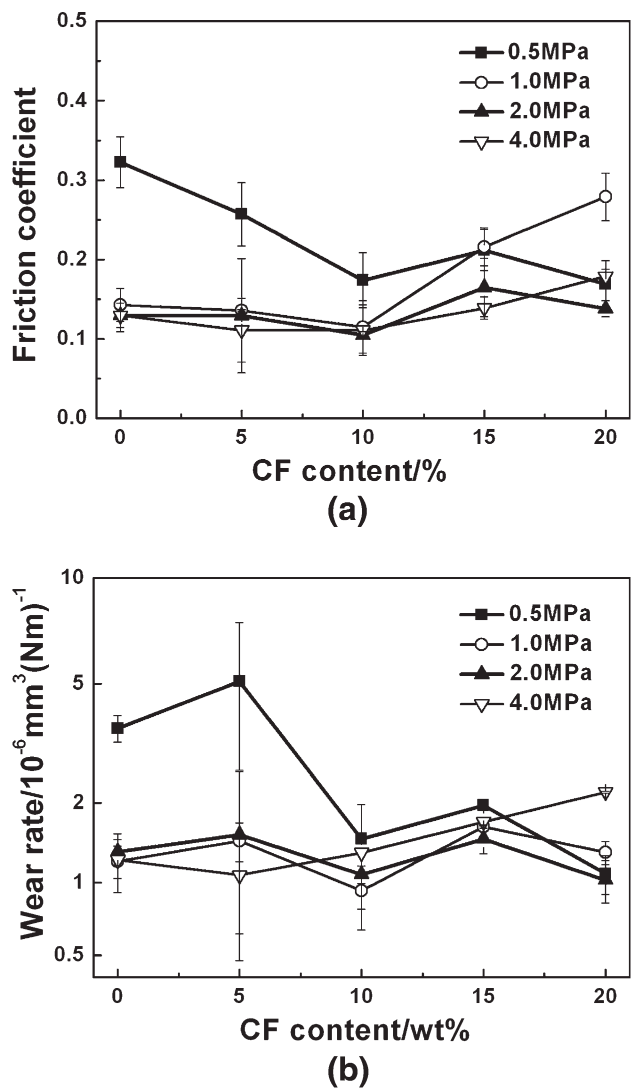


Figure 2 Effects of CF on the fiction coefficient (a) and wear rate (b) of PEEK/CF/10PTW composites. Test conditions: sliding velocity, 2 m/s; sliding duration, 2 h.

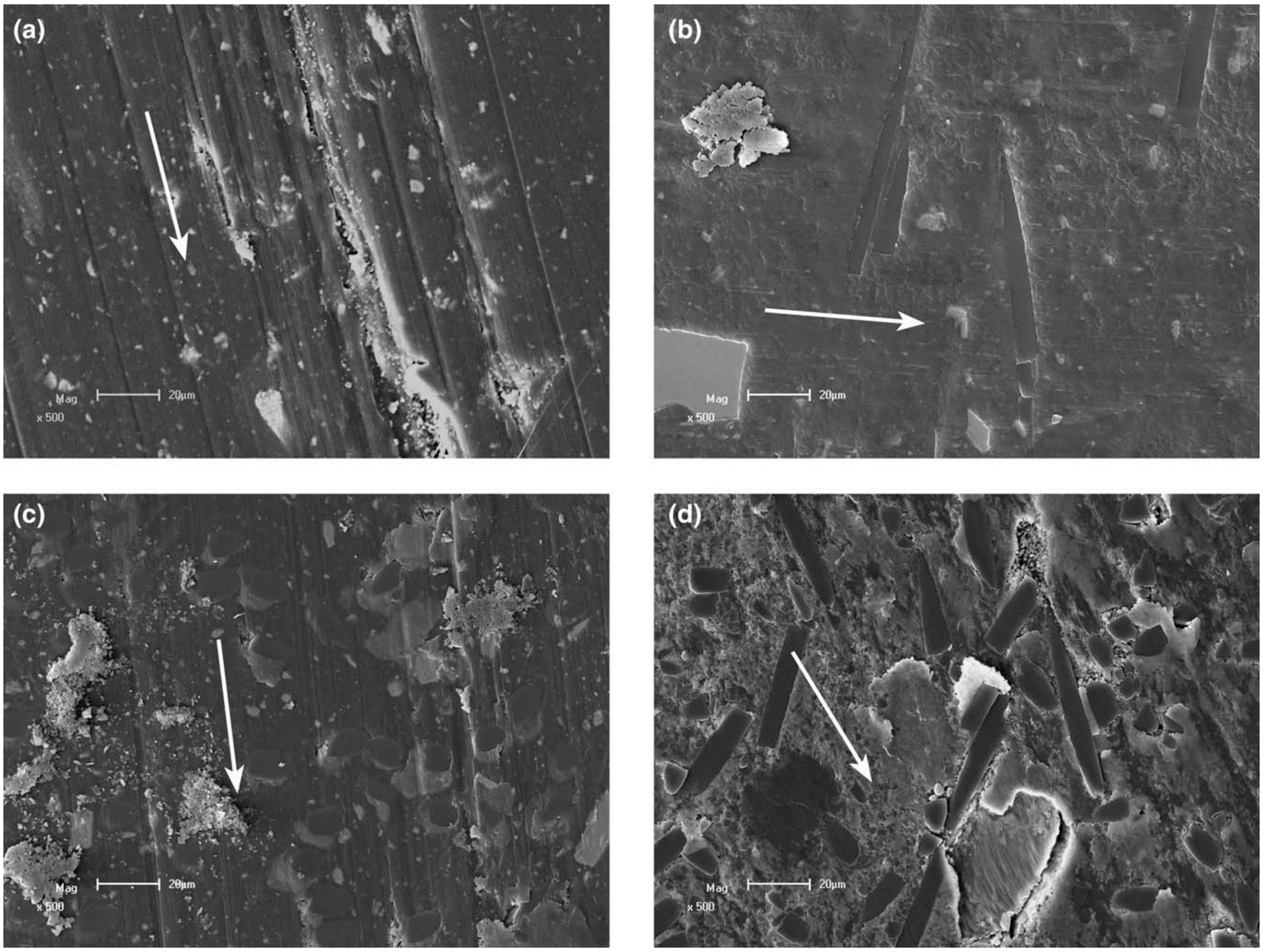


Figure 3 SEM morphologies of the worn surfaces of PEEK/5CF/10PTW composite (a) at 0.5 MPa and (b) at 4.0 MPa; PEEK/20CF/10PTW composite (c) at 0.5 MPa and (d) at 4.0 MPa. Test conditions: sliding velocity, 2 m/s; sliding duration, 2h. The arrow indicates the sliding direction.

temperature rising, which could decrease the friction coefficient. On the other hand, the enlarged real contact area resulted from the load rising would increase the friction coefficient. These two competitive factors would determine the final friction coefficient of the composite.[27] For PEEK/10CF/10PTW composite, at low load, the first factor was dominant, the friction coefficient decreased with the load increasing. At higher load, the effects of the two factors had a relative balance and then the increase of real contact area would be dominant eventually.

Moreover when the temperature was high, the polymer might be softened, which could cause the polymer to be detached easily and result in severe wear. During the sliding, some big debris on the wear surface would be crushed or sheared into small particles and acted as lubricants, the newly formed debris would come into being a more integrated layer on the worn surface and reduced the 'direct contact' between the composite and the counterpart, which could decrease the friction coefficient and wear rate.[31,32] However, the redundant debris likely became the third-body abrasives at the sliding, which increased the wear rate. It might be one reason that the wear rate of the PEEK/10CF/10PTW composite decreased with the load increasing at lower loads but increased slightly with the load increasing at higher load.

Effect of short carbon fibers

Figure 2(a) shows the friction coefficients of the PEEK/CF/10PTW composites with various mass ratios of CF at different loads. The CF content and applied load affected the friction behavior of the composites in different ways. At 0.5MPa, the friction coefficient of the composite with the CF mass ratio from 0 to 20 decreased first and then fluctuated slightly around 0.2. But at higher load, the friction coefficient increased when the mass ratio of CF increased. The friction coefficient of the PEEK/5CF/10PTW composite decreased with the increasing

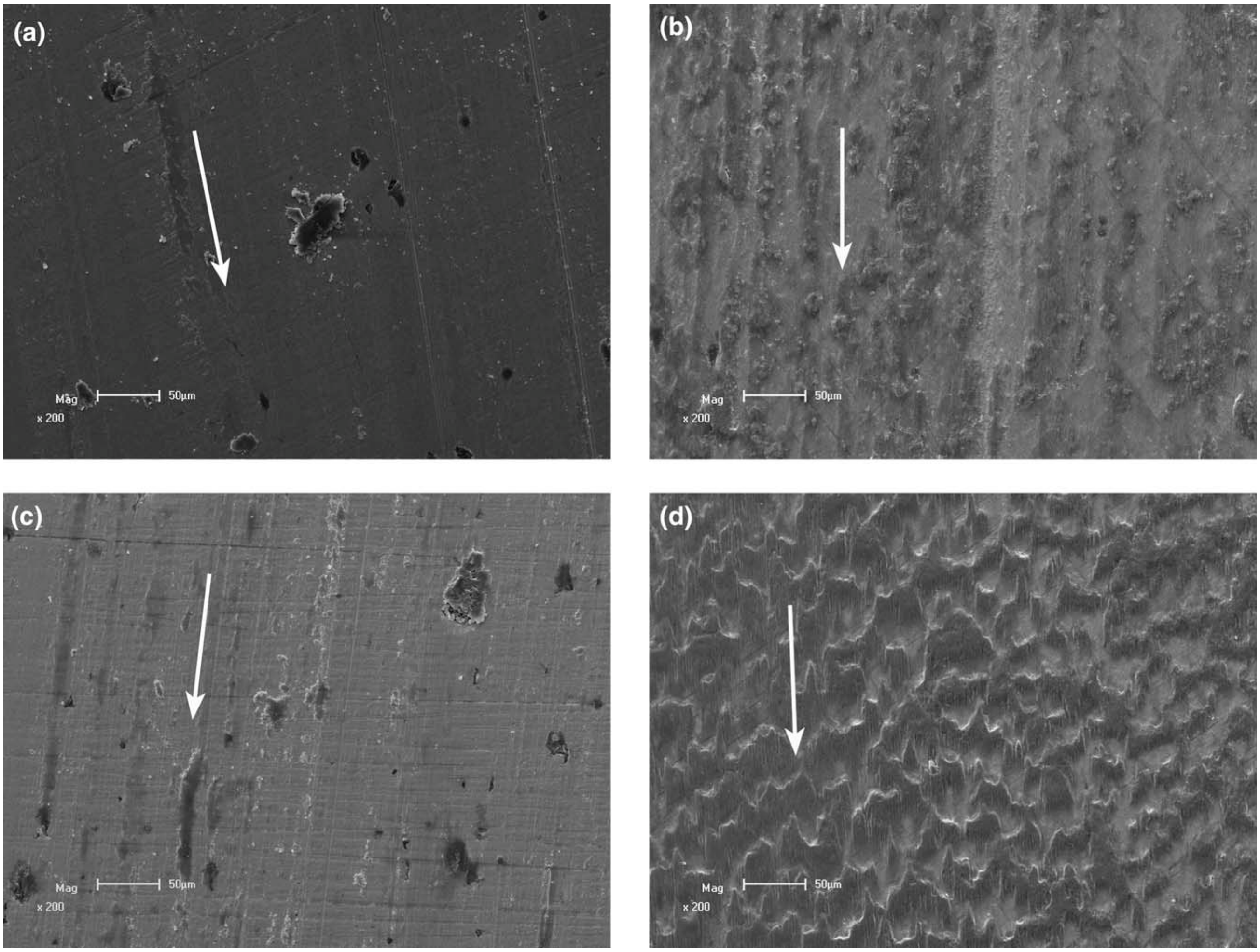


Figure 4 SEM morphologies of steel counterfaces of PEEK/5CF/10PTW composite (a) at 0.5 MPa and (b) at 4.0 MPa; PEEK/20CF/10PTW composite (c) at 0.5 MPa and (d) at 4.0 MPa. Test conditions: sliding velocity, 2 m/s; sliding duration, 2 h. The arrow indicates the sliding direction.

loads. However, at high CF contents, the composites revealed different fashions for the friction coefficient.

The wear rates of the PEEK/CF/10PTW composites with various mass ratios of CF in different loads are shown in Figure 2(b). The wear rates at 0.5 MPa were the highest in various contents of CF until the mass ratio of CF increased to 20. At 0.5 MPa, when the mass ratio of CF changed from 0 to 20, the wear rate of the composite increased slightly and then it decreased obviously with the mass ratios rising. However, at 1.0 MPa and 2.0 MPa, the changes of the wear rates of the composites were not so distinct. The wear rate decreased slightly first with the reinforcement of CF, then it increased linearly from 1.068×10^{-6} mm^3/Nm for PEEK/5CF/10PTW to 2.191×10^{-6} mm^3/Nm for PEEK/20CF/10PTW with an increase of 100% at 4.0 MPa. Those results indicated that the composites with high CF content exhibited more effective improvement on the wear resistance at low load condition. However, the increase of the CF content might worsen the wear behavior at high load condition.

The reinforcement of short carbon fiber affected the friction and wear behavior in many ways. The effective reinforcement of the fibers counteracted the effects of thermal softening at and above T_g.[15] However, when the fiber peels off from the matrix, the polymer would contact the steel counterpart directly without the protection from CF, so the polymer could be detached more easily, which increased the wear rate.[33] Figure 3 and Figure 4 show the SEM morphologies of worn surfaces and the counterfaces of the PEEK/CF/10PTW composites at dry sliding. The wear tracks were clearly visible in the sliding direction for PEEK/5CF/10PTW at 0.5 MPa, which indicated that there was an abrasive wear. Meanwhile, no apparent transfer film could be seen on the counterface. However, at 4.0 MPa, the worn surface of the composite was quite smooth. Fiber thinning could be seen clearly, which implied that carbon fibers borne most load during the sliding to protect the matrix from severe abrasion.[33] The transfer film was thin, continuous and uniform, which

TABLE II
The Temperature Rise During the Sliding for PEEK/CF/10PTW Composites (°C)

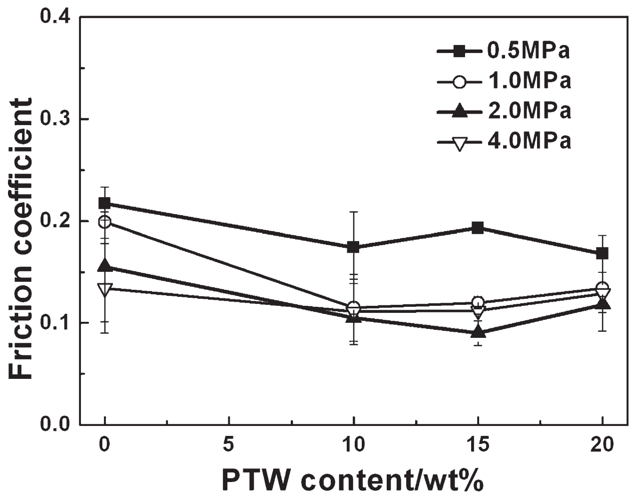
Load	PEEK/5CF/10PTW	PEEK/10CF/10PTW	PEEK/15CF/10PTW	PEEK/20CF/10PTW
0.5 MPa	21.4	13.0	15.6	15.3
1.0 MPa	18.0	41.0	75.2	77.3
2.0 MPa	85.0	82.3	91.3	99.4
4.0 MPa	96.8	123.8	130.2	190.8

reduced the 'direct contact' between the composite and the counterpart effectively. For PEEK/20CF/10PTW composite at 0.5 MPa, there were less scuffing tracks and exfoliations on the worn surface compared with that of PEEK/5CF/10PTW, which implied that the reinforcement of carbon fibers could improve the wear resistance property of the composite at low load. However, a higher load always means more heat generation and higher temperature, which could weaken the bonding between CF and PEEK matrix and cause fiber peeling-off easily. Furthermore, higher fiber content could promote the easy debonding of fiber-matrix and the generation of more debris. The wear debris would become the third-body abrasives during the sliding and resulted in a further temperature rising. As shown in Table II, the temperature rise of the composite was much higher at 4.0 MPa than that at 0.5 MPa and it rose dramatically with the increase of CF content. This was also evidenced by the worn surface of PEEK/20CF/10PTW composite at 4.0 MPa where the fiber peeling-off and the matrix broken could be seen [Fig. 3(d)]. Moreover, the transfer film of PEEK/20CF/10PTW at 4 MPa was thick and lumpy [Fig. 4(d)], which was also attributed to the high wear rate of the composite.

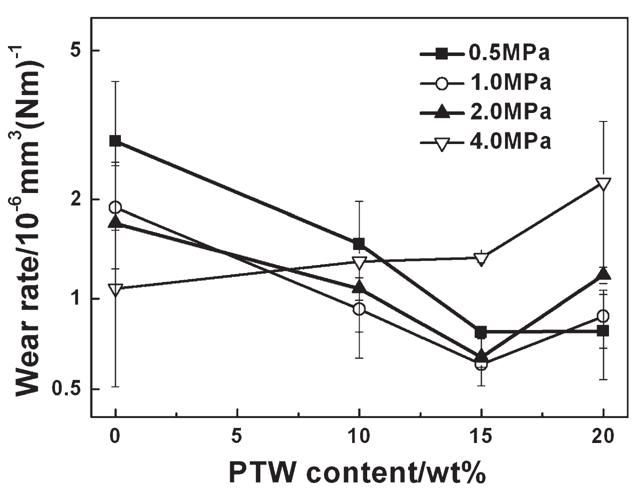
Effect of PTW

Figure 5(a) shows the friction coefficients of the PEEK/10CF/PTW composites with various mass ratios of PTW at different loads. The addition of the PTW decreased the friction coefficient of the composite. The friction coefficients of the composites with PTW were all lower than 0.2. The friction coefficients of the composites with different PTW content from 10 to 20 were almost in the same level at the same load condition. The wear rates of the PEEK/10CF/PTW composites with various mass ratios of PTW at different loads are shown in Figure 5(b). The addition of the PTW decreased the wear rate of the composite at 0.5, 1.0, and 2.0 MPa. At 0.5 MPa, the wear rate decreased with the mass ratio rising of PTW. For the condition of 1.0 MPa and 2.0 MPa, the lowest wear rates both achieved when the mass ratio of PTW was 15. However, the wear rate increased with the increasing contents of PTW at 4.0 MPa. Although the composite with high PTW content showed low wear rate at low load, it was apparent that the PEEK/10CF/20PTW was not suitable for high load application.

It was known that CF borne most of load during the sliding.[33] The breakage of the fiber would deteriorate the wear resistance of the composite. PTW could reduce the stress concentration on CF by increasing the stiffness of the composite to protect CF from failure.[28] Moreover, the mechanical properties of the matrix between the CFs would be improved by the reinforcement of PTW. Therefore the matrix would not be detached easily even in high temperature. To some extent, the small fillers might act as polishing agent during sliding.[34] However, high content of the whiskers could weaken the



(a)



(b)

Figure 5 Effects of PTW on the friction coefficient (a) and the wear rate (b) of PEEK/10CF/PTW composites. Test conditions: sliding velocity, 2 m/s; sliding duration, 2 h.

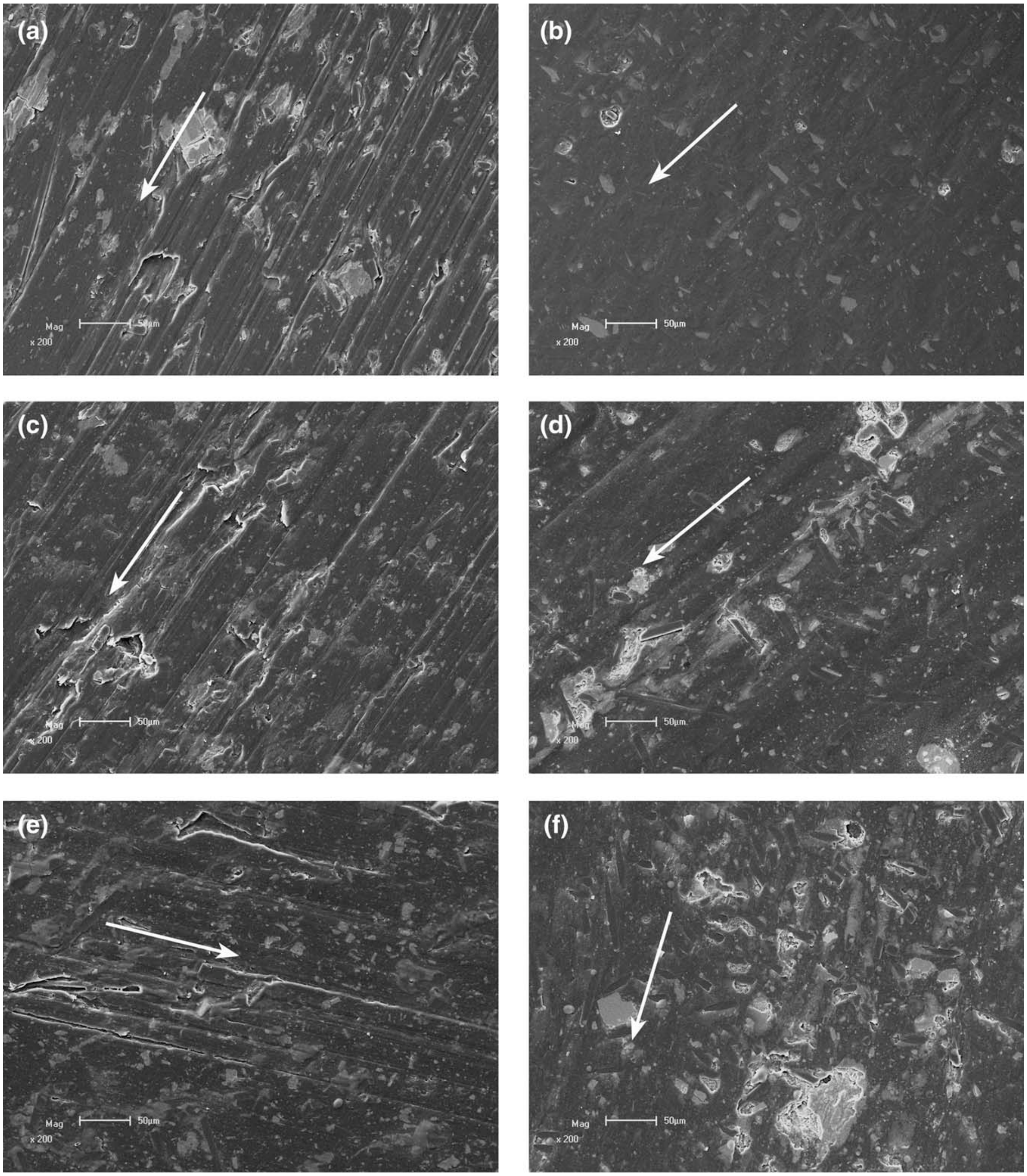


Figure 6 SEM morphologies of the worn surfaces of PEEK/10CF/10PTW composite (a) at 1.0 MPa and (b) at 4.0 MPa; PEEK/10CF/15PTW composite (c) at 1.0 MPa and (d) at 4.0 MPa; PEEK/10CF/20PTW composite (e) at 1.0 MPa and (f) at 4.0 MPa. Test conditions: sliding velocity, 2 m/s; sliding duration, 2 h. The arrow indicates the sliding direction.

bonding between the fillers and the matrix and result in the generation of more debris. And the friction and wear behavior would be deteriorated by the abrasive wear of whiskers. Figure 6 shows the SEM morphologies of the worn surfaces of the PEEK/10CF/PTW composites with the load of 1.0 MPa and 4.0 MPa. The wear tracks were clearly seen on the worn surface for the composites with different PTW contents at 1.0 MPa, which indicated that there could be abrasion wear during the sliding.

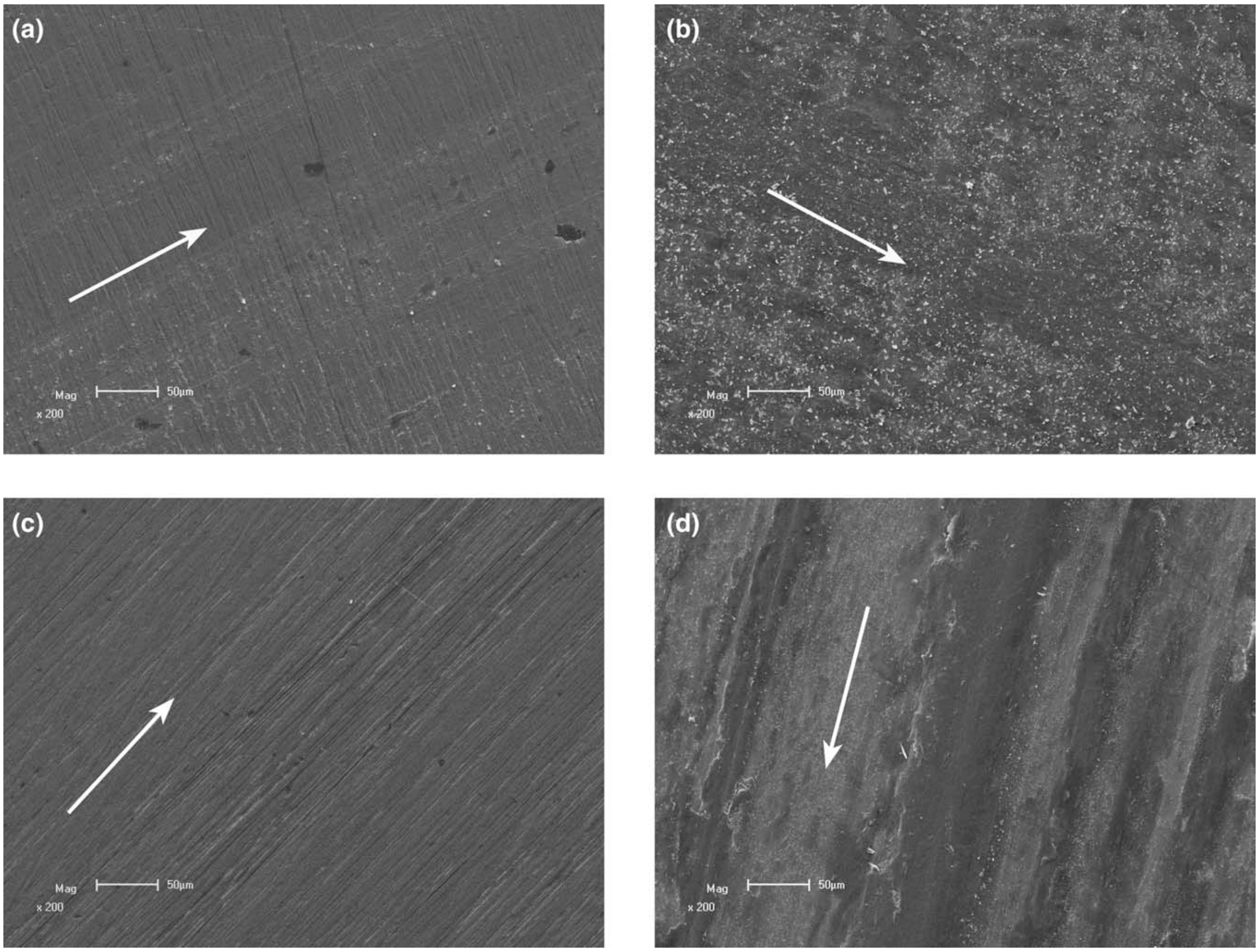


Figure 7 SEM morphologies of steel counterfaces of PEEK/10CF/15PTW composite (a) at 1.0 MPa and (b) at 4.0 MPa; PEEK/10CF/20PTW composite (c) at 1.0 MPa and (d) at 4.0 MPa. Test conditions: sliding velocity, 2 m/s; sliding duration, 2 h. The arrow indicates the sliding direction.

When the load increased to 4.0 MPa, the worn surface of PEEK/10CF/10PTW was much smoother than those with higher contents of PTW. The exfoliations on the wear surface increased with the PTW content, which indicated that more PTW could deteriorate the wear resistance of the composite at 4.0 MPa. Figure 7 shows the morphologies of the counterfaces of PEEK/10CF/15PTW and PEEK/10CF/20PTW composites at 1.0 MPa and 4.0 MPa. As shown in Figure 7(a,c), the transfer films of both composites at 1.0 MPa were thin and uniform. At 4.0 MPa, the transfer films were relatively thick and lumpy in present case. Especially, the quality of the transfer film was even worsen-off when the composite contained excessive PTW. Apparently, the formation of the ineffective transfer films at high applied load should be accounted for the high wear rate in some degree. The trend of the temperature rise with PTW content was similar to that of the wear rate (Table III). Apparently, a higher temperature rising on the worn surface would also lead to an easier detachment and result in higher wear rate. In present work, the composites with low PTW contents could have better wear resistance behavior at high load. PEEK/10CF/15PTW might be the optimum option for the application in present case.

TABLE III
The Temperature Rise During the Sliding For PEEK/10CF/PTW Composites (°C)

Load	PEEK/10CF/10PTW	PEEK/10CF/15PTW	PEEK/10CF/20PTW
0.5 MPa	13.0	12.6	18.4
1.0 MPa	41.0	10.3	20.2
2.0 MPa	82.3	45.9	73.5
4.0 MPa	123.8	139.3	191.9

CONCLUSIONS

The tribological properties of PEEK composites filled with CF and PTW were studied at different loads under dry sliding. The effects of CF and PTW content on the wear behavior were discussed. The following conclusions are drawn:

1. Compared with the PEEK/0CF/0PTW composite, the hybrid composite showed an excellent tribological property in dry sliding condition. For most composites in this work, the wear rate decreased first and then increased with the load rising.
2. Although the composites with low CF content showed higher wear rate at low applied load condition, they exhibited a high wear resistance at high load condition.
3. At low applied load, high PTW content in the composites could reduce the wear rate, but the excessive PTW deteriorated the wear resistance of the composite at high applied load condition. Contenting 15 wt % PTW in the hybrid composites might be the optimum option for the application in present case.

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