Tribological Behaviors of Polytetrafluoroethylene Composites Under Dry Sliding and Seawater Lubrication

Shibo Wang,[1,2] Li Pan,[1,2] Qian Li[1,2]

[1]School of Mechanic and Electronic Engineering, China University of Mining and Technology, Xuzhou, Jiangsu Province 221116, China
[2]Institute of Tribology and Reliability Engineering, China University of Mining and Technology, Xuzhou, Jiangsu Province 221116, China
Correspondence to: S. Wang (E-mail: wangshb@cumt.edu.cn)

ABSTRACT: The composites of polytetrafluoroethylene (PTFE) filled with expanded graphite (EG), poly(p-oxybenzoyl) (POB), and basalt fiber (BF) were prepared by heating compression and sintering molding. The tribological behavior of PTFE composites was investigated with a pin-on-disk tester under dry conditions and seawater lubrication. The worn surface of PTFE composites and the transfer film on the counterface were observed with a scanning electron microscope. The results indicated that the incorporation of EG and POB improved the hardness of PTFE composites, and addition of BF led to greater load-carrying capacity. Compared to pure PTFE, the coefficients of friction of PTFE composites slightly increased, but the wear rates were significantly reduced (the wear rate of composite with 3% EG being only 10.38% of pure PTFE). In addition, all the composites exhibited a lower coefficient of friction (decreases of about 0.03–0.07) but more serious wear under seawater lubrication than under dry sliding. The wear mechanism changed from serious abrasive wear of pure PTFE to slight adhesion wear of PTFE composites under both conditions. A transfer film was obviously found on the counterface in seawater, but it was not observed under dry conditions. Among all the materials tested, the PTFE-based composite containing 20% POB (mass fraction), 2% EG, and 3% BF exhibited the best comprehensive performance.
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INTRODUCTION

Marine tribology is a new developing subfield of tribology, of which a significant application is sea water hydraulic drive system. Sea water hydraulic drive system possesses outstanding superiority of saving energy, non-pollution, high efficiency, safe, and stable, however, due to the low lubricity and high corrosivity of sea water, the friction pair materials of hydraulic component are required to have great tribological property, and excellent corrosion resistant.[1] Compared with metals and ceramics, polymers, and polymer-based composites have many advantages, such as excellent corrosion resistance, good environmental adaptability, and outstanding tribological properties.[2,3] Investigations on tribological behaviors of polymers in aqueous environments have been widely reported by many researchers.[1,4–9] Tanaka[4] and Yamamoto[5] found that the introduction of water can play a lubricating role in a polymer–metal sliding friction pair and generally lowers the coefficient of friction, but it may raise the wear rate of the polymer. Tanaka suggested that the increase of wear in water may be caused by the modification

of the polymer surface structure by water instead of the effect of a polymer transfer film on its counterpart surface.[4] Meanwhile, the decreased hardness of polymers in aqueous environments may also lead to higher wear.[5] Evans[6] and Srinath[7] reported that polymer plasticization usually occurs after water absorption, which may result in the reduction of strength and hardness, and thereby increase the wear rate. Moreover, because of the strong corrosion of seawater, the friction surface will inevitably be affected by corrosion. Wang et al.[1] indicated that in a polymer–GCr15 friction pair in seawater, the surface roughness of the GCr15 significantly increased owing to the corrosion induced by the seawater. This would prevent the formation of a liquid film and sharply worsen the lubricating effect of the aqueous medium, consequently increasing the scuffing destruction of the polymer and dramatically increasing the wear rate. This wear mode in which the wear extent of the polymer depends on the corrosion extent of the surface of its counterpart can be called indirect corrosive wear.[1] Additionally, Jia[8] suggested that the cooling effect of water effectively hindered

frictional heating and inhibited the adherence and plastic deformation of the polymer.

However, many polymers have some disadvantages (e.g., not a low enough coefficient of friction under dry conditions and boundary lubrication, a degradation of properties as a result of swelling by water absorption, etc.). Polytetrafluoroethylene (PTFE), which is an excellent self-lubricating material, exhibits a very low coefficient of friction, an extremely low water absorption rate, and excellent chemical stability in various corrosive environments.[10,11] Nevertheless, PTFE has a compressive strength and hardness that are very low, undergoes high compression creep, and has poor thermal conductivity. Moreover, because of its low interface energy, the transfer film formed on the counterface may easily fall off and finally weaken wear resistance. These defects have limited the application of PTFE.[11,12] Therefore, it is imperative to reinforce PTFE with various fillers such as glass fiber (GF), carbon fiber (CF), and some high-performance polymers such as poly(p-oxybenzoyl) (POB).[13–16] Cheng[14] indicated that GF and CF could increase the coefficient of friction of PTFE composites while greatly improving the hardness and wear resistance and altering the wear mechanism of pure PTFE at the same time. POB exhibits excellent thermal stability, low compression creep, and high hardness, and the properties of POB and PTFE are complementary. Moreover, POB can blend with PTFE in any proportion and generally reinforces PTFE with several kinds of fillers acting together.[15,16] Actually, the addition of two or more kinds of fillers may achieve better reinforcement, because the synergism between different fillers can help to further reduce the wear rate and improve the overall performance of the composites.[17,18] Chen[19] found that PTFE blended with polyimide (PI) and CF exhibited much better tribological properties than PTFE reinforced with PI or CF alone, owing to the synergism between PI and CF. So far, the number of studies on polymers reinforced with expanded graphite (EG) and basalt fiber (BF) are still very few. Expanded graphite has strong corrosion resistance and strong compression resistance, as well as great plasticity and self-lubricity, properties that can be expected to improve the friction of the composite. Basalt fiber is a kind of high-performance fiber with high strength and modulus, excellent wear resistance, and good adaptability to various environments, which can enhance the load-carrying capacity of the PTFE matrix.[20]

In the past few years, investigations on the tribological behavior of PTFE have mainly been performed in the field of dry friction as well as under oil and distilled water lubrication. There are only a few studies on friction of PTFE composites in seawater. Chen[3] found that the tribological properties of CF/PEEK under sea water was better than those under dry friction and pure water, owing to the better lubricating effect of sea water. Wang[1] suggested that Mg^{2+} and Ca^{2+} were the key factors leading to the lower coefficient of friction in sea water by reducing the corrosion of counterface. In this article, the sliding tribological behavior of PTFE reinforced with POB, BF, and EG was studied, with the objective to investigate the tribological properties, wear, and transfer film mechanism under dry conditions and seawater lubrication. Moreover, we discuss the effect of reinforcement to the fillers, especially to provide some guidance for application of BF and EG in the tribological field.

EXPERIMENTAL

Materials
PTFE powder (with purity of 99% and molecular weight of 5,000,000) and POB powder (with purity of 99% and average grain diameter of 74 μm) were both obtained from Chenguang Technology Trading Co., Chengdu, China. The average diameter of single BF (Shanxi Basalt Fiber Technology Co., China) was 12 μm and the purity of EG powder (Shijiazhuang ADT Trading Co., China) was more than 98%.

PTFE and its composites filled with POB, BF, and EG (as listed in Table I) were prepared with a hot press method. First, the PTFE and filler powders as well as a silane coupling agent (KH550, A-1100 in America) were dissolved in absolute alcohol and stirred in a blender for 30 min. After alcohol evaporation in an oven, the dry mixture was compressed under a pressure of 25 MPa for 15 min at room temperature, followed by sintering at 370–380°C for 4 h, and then cooled to room temperature under a pressure of 50 MPa. The disk PTFE composites specimens were obtained by machining to dimensions of 40 mm in diameter and 8 mm thickness.

316L stainless steel (AISI, specific composition: ≤0.03 wt % C, ≤1.00 wt % Si, ≤2.00 wt % Mn, ≤0.045 wt % P, ≤0.03 wt % S, 12.0–15.0 wt % Ni, 16.0–18.0 wt % Cr, and 2.0–3.0 wt % Mo) was used as the counterpart material for the tribological test. The steel pin had a diameter of 10 mm and a length of 8 mm.

As a test medium, artificial seawater with a pH of 8.1 was prepared according to the ASTMD1141-98 standard.[21]

Water Absorption Testing
Water absorption of PTFE composites was measured according to the ISO62 1999 standard.[22] The amount of water absorbed by the PTFE specimen was determined by measuring its change in mass. The dry PTFE composite specimens were immersed in water at room temperature for 24 h, and then the difference of their initial masses from those after exposure to water was measured using an electronic balance with an accuracy of 0.1 mg. Water absorption of PTFE composites was expressed as the percentage of the initial mass.

Hardness Testing
In this article, the hardness of the PTFE composites was determined by a ball indentation method. The test was carried out on a UMT-2 tribometer in accordance with ISO2039-1 (2003).[23] The indentation load (preload of 9.8 N and normal

Table I. The Composition of Various PTFE Composites

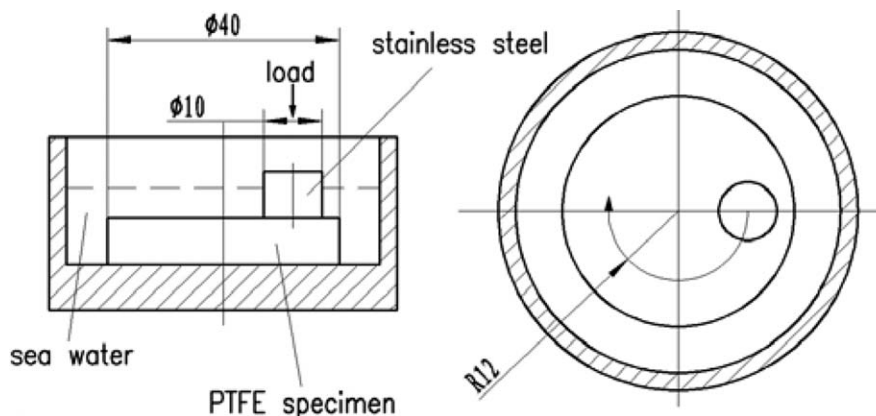| Material	PTFE (g)	POB (g)	EG (g)	BF (g)	Density (g/cm³)
PTFE	100	0	0	0	2.206 ± 0.095
PTFE-1	80	20	1	3	1.933 ± 0.074
PTFE-2	80	20	2	3	1.911 ± 0.062
PTFE-3	80	20	3	3	1.882 ± 0.083

Figure 1. Contact schematic of sliding friction pair.

load of 132 N) and depth could be controlled accurately. The indentation depth was recorded during the test, and the hardness was calculated by using the following equation:

$$HB = \frac{0.21 F_{max}}{0.25 \pi d(h_{max} - 0.04)}, \quad (1)$$

where HB is the ball indentation hardness of the PTFE composite (in MPa), F_{max} is the normal load (in N), d is the diameter of the indentation ball (in mm; chosen as $d = 5$ mm in this article), and h_{max} is the maximum indentation depth, which is determined by averaging the results of five tests.

Wear Testing

The sliding wear behavior of the PTFE composites at room temperature under dry conditions and seawater lubrication was evaluated on a MMW-1 pin-on-disk tribometer. The wear tests ran for 180 min under a normal load of 200 N with a linear velocity of 0.025 m/s.

The contact schematic of the sliding friction pair is shown in Figure 1. Before each test, the surfaces of the steel and the composite were polished to surface roughnesses of $R_a = 0.1$–$0.2\,\mu m$ and $R_a = 0.4$–$0.6\,\mu m$, respectively. Then, all the specimens were cleaned with absolute alcohol in an ultrasonic cleaner and dried in an oven for 4 h. The worn morphologies of the PTFE composites were measured with a three-dimensional profiler. Then the wear volume loss V (in mm^3) was calculated as

$$V = 2\pi Rbh, \quad (2)$$

where R ($= 12$ mm in this article) is the radius of sliding wear track as shown in Figure 1, b is the width of the wear trace (in mm), and h is the depth of the wear trace (in mm). The specific wear rate w [in mm^3/(N m)] of PTFE composites was calculated according to the formula

$$w = V/(F \cdot S), \quad (3)$$

where F and S are the normal load (in N) and sliding distance (in m) of the wear test. The wear test was executed three times for each kind of PTFE composite. During the test, fresh

artificial sea water was supplied to the solution pool every 30 min to adjust the concentration and avoid the accumulation of wear debris. The worn surface morphologies of PTFE composites and 316L stainless steel were observed with a HITACHIS-3000N scanning electronic microscope (SEM).

RESULTS AND DISCUSSION

Water Absorption, Hardness, and Microstructure of PTFE-based Composites

In aqueous environments, different polymers usually have different absorption rates of water. Water absorption will cause the plasticization of polymers and consequently reduce the strength and hardness, which accordingly highly influence their tribological behaviors.[6,7] Generally, the coefficient of friction of polymers (except some hydrophobic ones) may decrease in seawater, but the wear rate will significantly increase owing to plasticization. Srinath et al.[7] indicated that the water absorption of PA6 was 1.425%, and its hardness decreased by up to 25% after the material was immersed in water for 24 h as a result of plasticization. Table II shows the water absorption and the ball indentation hardness of PTFE composites before and after immersion for 24 h. From the table, one can see that the water absorption of PTFE composites are very low (only about 0.006–0.016%) and the decreases in ball indentation hardness are only 0.10–0.48 N/mm^2 (about 1.2–2.3%).

SEM photographs of the fractured surfaces of PTFE and its composites are shown in Figure 2. It is found that the fractured surface of pure PTFE is dense and characterized by crystalline

Table II. Hardness and Water Absorption Capacity of PTFE Composites

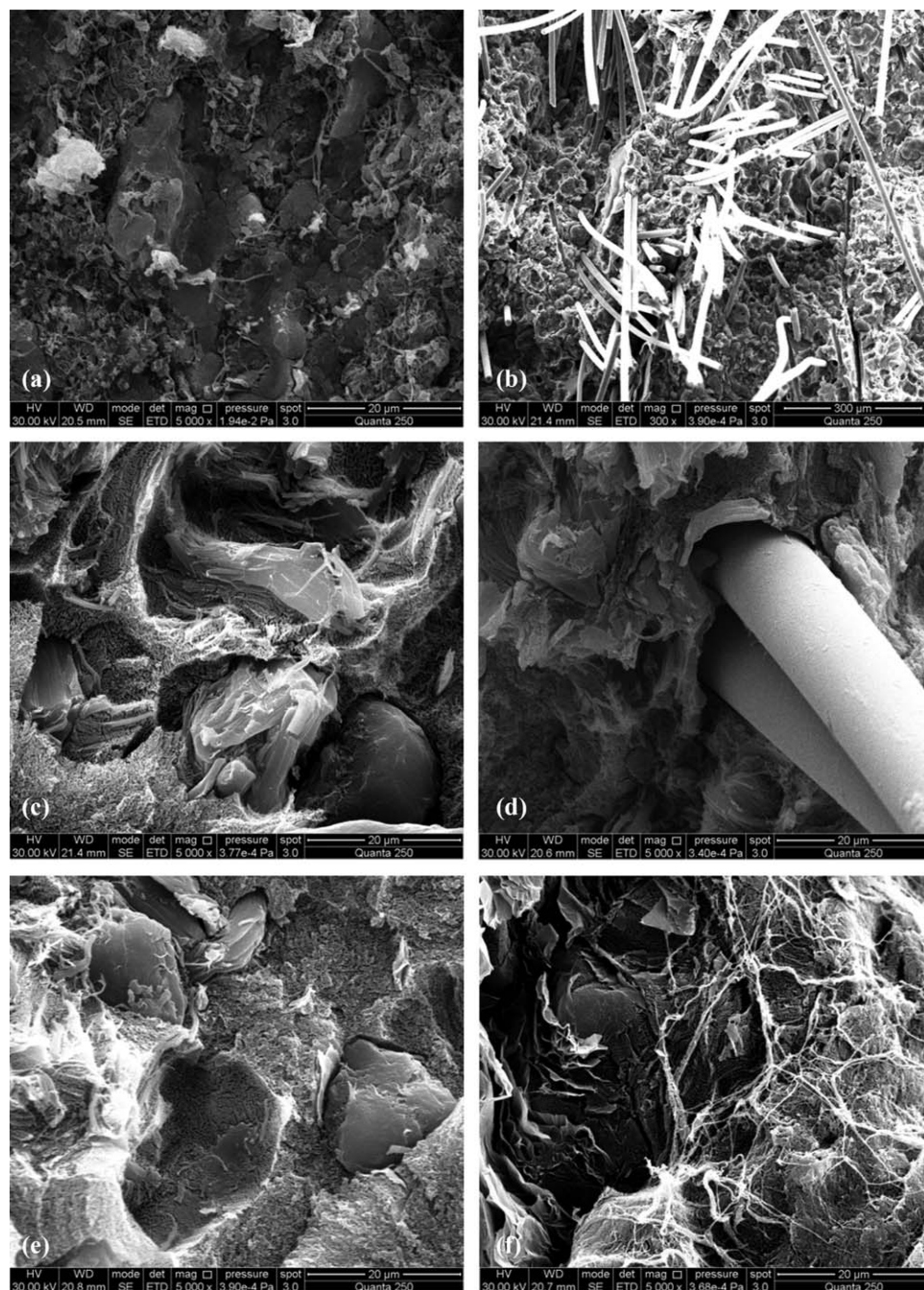| Material	Water absorption rate (%)	Ball indentation hardness (MPa)	
		Before immersion	After immersion
PTFE	0.0061	7.20 ± 0.05	7.11 ± 0.08
PTFE-1	0.0145	20.58 ± 0.14	20.10 ± 0.18
PTFE-2	0.0122	22.35 ± 0.27	22.05 ± 0.15
PTFE-3	0.0159	18.56 ± 0.19	18.17 ± 0.20

Figure 2. SEM photographs of fractured surfaces of PTFE and its composites: (a) PTFE, (b) and (c) PTFE-1, (d) and (e) PTFE-2, and (f) PTFE-3.

bands and dimple-like structure [Figure 2(a)]; however, the fracture surfaces of filled PTFE are uneven and contain a few big pits, which may be caused by the separation of a large amount of POB particles that have loosened from the PTFE matrix. Meanwhile, it can be observed that the PTFE-1 specimen exhibits a relatively loose microstructure [Figure 2(b,c)], which is probably because the incorporation of three kinds of fillers leads to internal defects. Especially, the microstructure of PTFE-2 (containing 2 wt % EG) shows great interfacial adhesion and the BF combines well with the PTFE matrix

[Figure 2(d,e)]. It can be demonstrated that the incorporation of EG can remove the defects between the matrix and fillers. Nevertheless, increasing the amount of EG does not further improve the microstructure; in contrast, the EG particles may disperse non-uniformly and even agglomerate into stacks [Figure 2(f)], which is not beneficial to effectively improving mechanical properties.[24]

The water absorption and hardness of PTFE composites were influenced by their microstructure. It can be seen that the
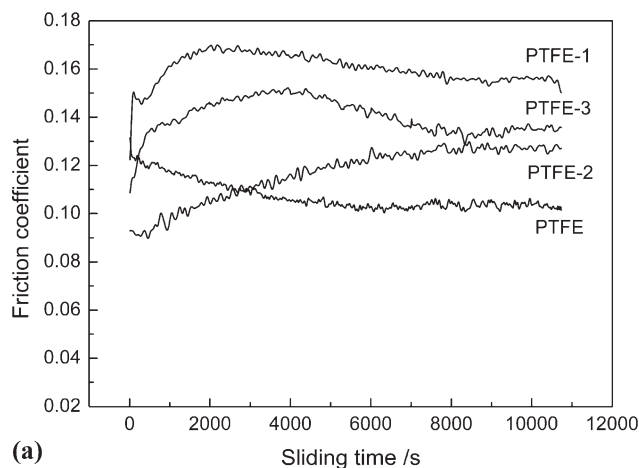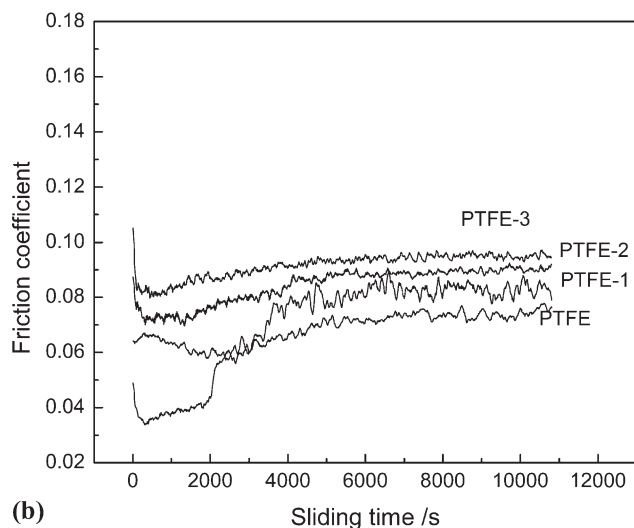
Figure 3. Variation of coefficients of friction of PTFE composites against sliding time at a normal load of 200 N and a sliding velocity of 0.025 m/s: (a) dry Friction; (b) seawater lubrication.

fracture surfaces of PTFE-1 contains some holes and is very loose, which agrees well with its water absorption being higher than that of pure PTFE. PTFE-2, with its dense microstructure, exhibited a lower absorption. Furthermore, when the mass fraction of EG reached 3 wt %, water absorption reaches its maximum, and this should be related to the loose and porous structure as well as the excellent absorption of EG. Table II shows that the values of ball indentation hardness of PTFE composites were much higher than that of pure PTFE, with the highest one being nearly 3.1 times that of pure PTFE. This is due to the greater hardness POB and EG's strong pressure resistance. Meanwhile, because of its high strength and modulus, BF can significantly improve the load-carrying capacity of the PTFE matrix.[20] After absorbing water for 24 h, PTFE-2, with its better microstructure, maintains the highest ball indentation hardness (22.05 MPa) among all specimens.

Friction and Wear Behaviors of PTFE-based Composites

The variation of the coefficients of friction of PTFE and its composites against sliding time under dry conditions and seawater lubrication are shown in Figure 3. With increasing sliding
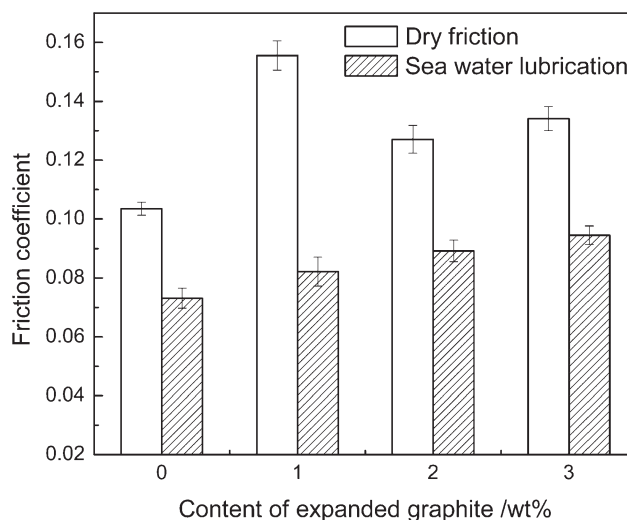

Figure 4. Variation of steady-state coefficients of friction of PTFE composites against content of expanded graphite at a normal load of 200 N and a sliding velocity of 0.025 m/s.

time, the coefficient of friction of PTFE decreases quickly to 0.1, and those of PTFE composites gradually decrease before reaching steady-state friction of between 0.12 and 0.16 under dry condition. In seawater, the coefficients of friction of all specimens become steady in the range of 0.07–0.10 at a sliding duration of 1 h.

Figures 4 and 5, respectively, show the variation of steady-state coefficients of friction and wear rate of PTFE composites against content of EG under dry conditions and seawater lubrication. Note that the coefficients of friction of PTFE composites are higher than that of pure PTFE under both conditions. This is because of the higher coefficient of friction of POB and BF normally. However, the increases in the coefficient of friction are very small, which shows that the incorporation of fillers still retains the advantage of the low coefficient of friction of the
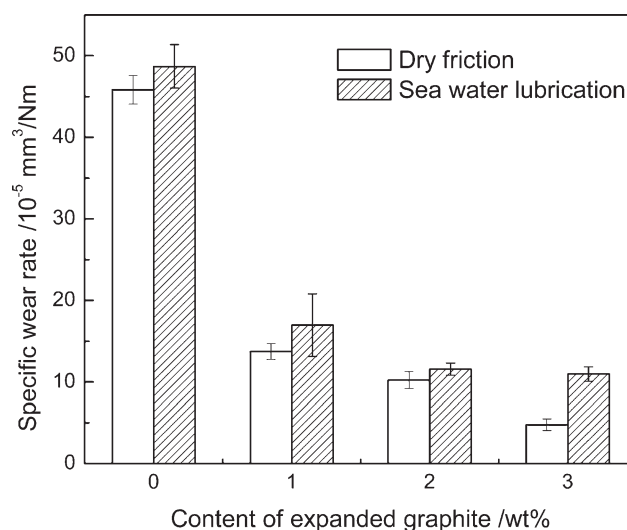

Figure 5. Variation of wear rate of PTFE composites against content of expanded graphite at a normal load of 200 N and a sliding velocity of 0.025 m/s.
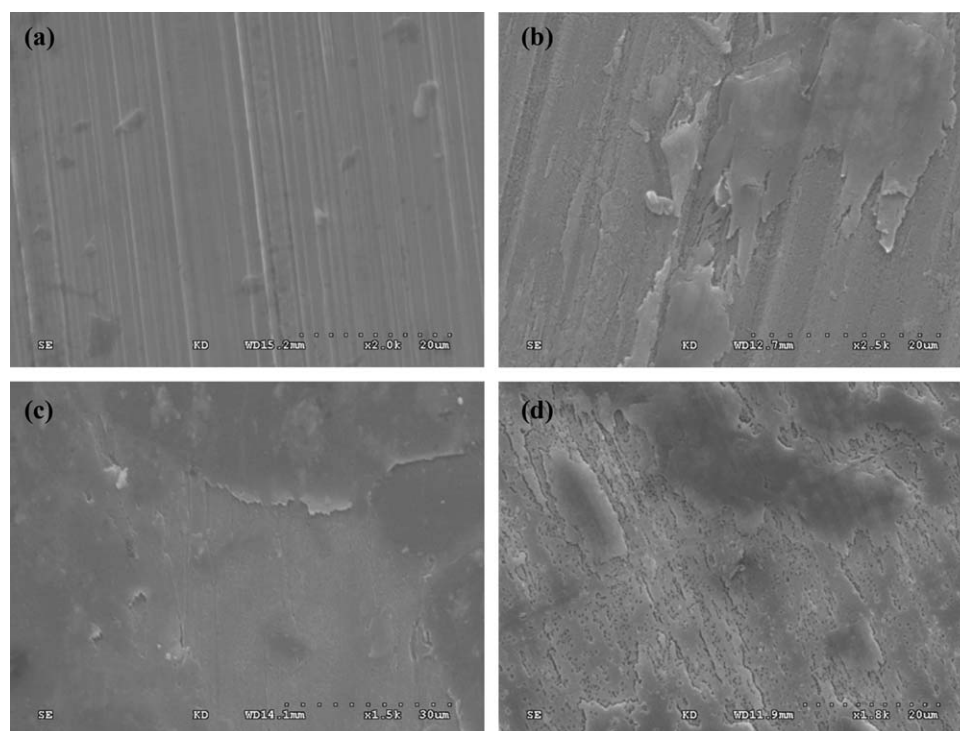
Figure 6. SEM photographs of worn surfaces of PTFE composites in dry friction: (a) PTFE, (b) PTFE-1, (c) PTFE-2, and (d) PTFE-3.

PTFE matrix, and this corresponds with the conclusion of some previous reports.[11,25] In dry sliding, with increasing content of EG, the coefficient of friction of PTFE composites first decreases until the content of EG reaches 2 wt % and then slightly increases. It is because that large amount of EG particles may agglomerate, which is not beneficial to further reduce coefficient of friction.[24] In this study, the PTFE composites simultaneously reinforced with POB, BF, and EG exhibit much better wear resistance than pure PTFE. The wear rate decreased gradually with rising mass fraction of EG. PTFE-3 has the lowest wear rate, with a value of 4.3×10^{-5} mm^3/(N m), which accounts for 10.38% of that of pure PTFE. On one hand, BF maintains its high strength and modulus as well as its great wear resistance, so its incorporation into the matrix can enhance the mechanical properties of PTFE. The exposed BF on the worn surface is able to bear the load for the PTFE matrix and prevents the crystalline band of PTFE from being drawn out during the friction process.[20] On the other hand, the synergism of EG with POB and BF significantly decreased the wear rate of PTFE and kept the coefficient of friction at a relatively low level.

Under seawater lubrication, the coefficients of friction of PTFE composites were about 52.8–70.6% (decrease by 0.03–0.07) of those under dry sliding conditions. They also increased with increasing EG content. Note that the coefficient of friction of PTFE-1 exhibits the largest decrease and nearly becomes half that observed in dry sliding. This is mainly because of the effect of fluid lubrication by the seawater on the contact surface; specifically, the fluid embeds into the bearing surface and is compressed, creating a high-pressure region to counteract the load, which consequently reduces the friction.[26]

The wear rates of PTFE composites under seawater lubrication are higher than those under dry conditions and decrease with increasing amounts of EG. On one hand, EG has great self-lubricity, and the interlayer is apt to slide under shear stress, leading to low abrasion.[27] On the other hand, the EG particles stripped off the surface during friction, reducing the direct contact between matrix and counterpart and may form lubricant film.[24] PTFE-3 presents the lowest wear rate under seawater lubrication, being 22.48% of that for pure PTFE but still about 2.3 times as high as that under dry sliding. The increasing wear of polymers in seawater has been reported in some investigations.[5,28,29] As indicated by the above-mentioned hardness data, the decrease of hardness resulting from plasticization is negligible and may not be the main factor that leads to the variation of wear rate of PTFE composites in seawater.[1]

Wear Mechanism

The morphology of the worn surface of PTFE and its composites under dry sliding was investigated by SEM, as shown in Figure 6. There are numerous wide and deep furrows and evidence of plastic deformation on the worn surface of pure PTFE, which implies that serious abrasive wear occurred. Under dry sliding, the PTFE contacts the steel directly and the asperities on the steel surface cause severe furrowing on the surface of PTFE as a result of its low hardness. The worn surface of PTFE-1 was characterized by slight furrowing and adhesive flaking [Figure 6(b)], which indicated that the wear mechanism changed from abrasive wear to adhesive wear. In the case of PTFE-2 including 2 wt % EG, the worn surface was much smoother, did not exhibit furrowing, and was characterized by slight adherence [Figure 6(c)]. The worn surface of PTFE-3
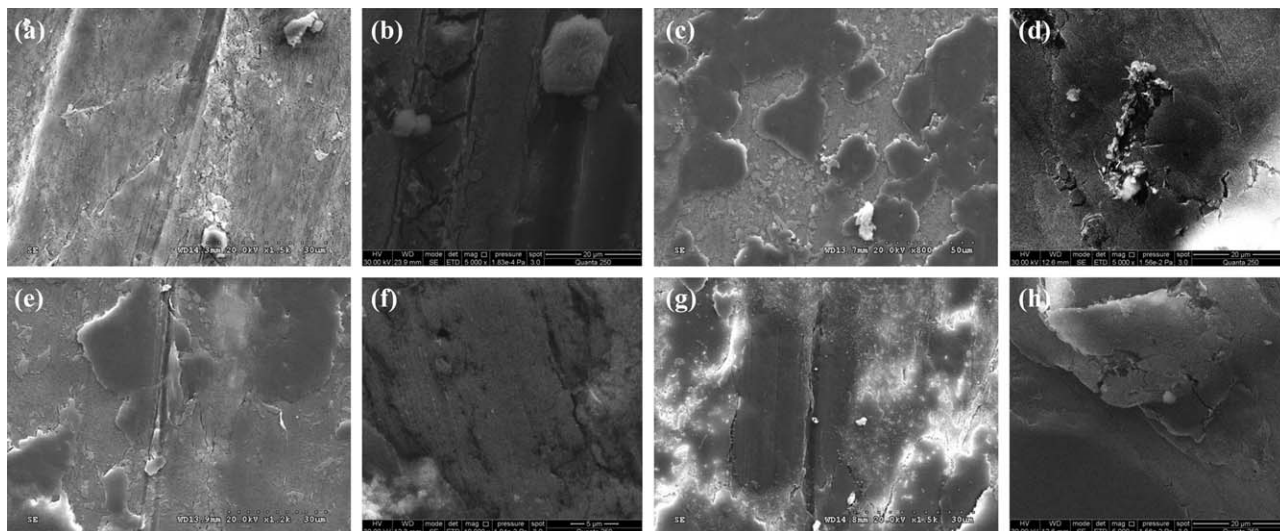
Figure 7. SEM photographs of worn surfaces of PTFE composites under seawater lubrication: (a) and (b) PTFE, (c) and (d) PTFE-1, (e) and (f) PTFE-2, and (g) and (h) PTFE-3.

incorporating 3 wt % EG exhibited slight adhesive flaking and contains large numbers of microholes [Figure 6(e)], which likely result from the larger mass fraction of EG with its loose and porous structure.

Figure 7 presents the SEM photographs of the worn surface of PTFE and its composites under seawater lubrication. In Figure 7(a) and (b), the worn surface of pure PTFE is characterized by severe furrows accompanied by plastic deformation, on which some large pieces of wear debris are observed. In contrast, the worn surfaces of composites show signs of slighter furrowing effects and smaller wear debris, indicating that the wear mechanism is mainly adhesive [Figure 7(d)]. However, the PTFE composites exhibit more serious adhesive wear and increased number of furrows compared with those in dry sliding. With higher EG content, the surface of composites becomes apparently smoother and the amount of wear debris that flakes off the matrix decreases, which is similar to the situation in dry sliding. As is clearly seen from Figure 7(c) and € the POB layers are exposed on the worn surfaces of the PTFE composites. At the initial stage of the wear, the PTFE matrix was first ground off because of its poor wear resistance, and then the POB, with its high hardness and great wear resistance, supported the main stress applied on the contact surface and prevented further flaking and plastic deformation of PTFE, thus reducing the wear rate. Overall, it can be demonstrated that the destruction of the worn surface in seawater is more serious than that under dry sliding, confirming the higher wear rate. Yamada and Tanaka suggested that the increased wear of composites in aqueous environment was caused by the permeation of water molecules into the interlayer between the fillers and the PTFE matrix, which led to a greater possibility of separation.[29,30]

The surface roughness of PTFE composites has an obvious relationship with the morphology of the worn surface. As seen from Table III, the worn surface roughness of pure PTFE is the highest whether under dry sliding or seawater lubrication; this

is attributed to the large amounts of wear debris and deep furrows on the worn surface. Under dry sliding, the worn surfaces of PTFE composites exhibit an abated furrowing effect and become characterized by slight adhesive wear, so the planeness is improved. In seawater, the surface roughness of composites is relatively higher (increased by about 19.8–59.3%), corresponding to serious adhesive flaking. With increasing EG mass fraction, the wear of PTFE composites is relieved gradually and the surface roughness thereby decreases.

Transfer Film

To study the transfer film of PTFE composites, the worn surfaces of 316L stainless steel under dry sliding and seawater lubrication were observed, as shown in Figure 8. Under dry sliding, no signs of transfer film are observed on the counterface of pure PTFE; also there are only a few pieces of wear debris on the surface of the steel against the PTFE composites. This is because the PTFE transfer films always fall off the counterface easily owing to its extremely low surface energy. In contrast, in seawater, some material particles adhere to the counterface of pure PTFE, and, moreover, some uniform and consecutive transfer films are obviously formed on the 316L stainless steel against the PTFE composites [Figure 8(B–D)]. Generally, water

Table III. Surface Roughness of PTFE Composites

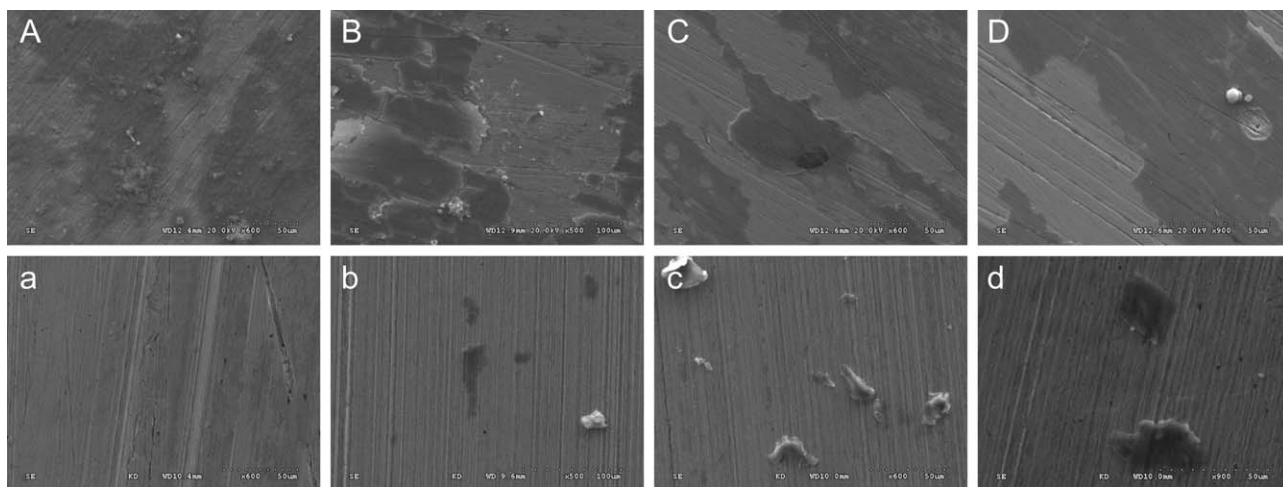| Material | Surface roughness (μm) | | | |
| | Dry friction | | Seawater lubrication | |
	Before friction	After friction	Before friction	After friction
PTFE	0.614	1.046	0.518	1.253
PTFE-1	0.593	0.816	0.572	1.101
PTFE-2	0.612	0.701	0.533	0.996
PTFE-3	0.656	0.649	0.541	1.034

Figure 8. SEM photographs of worn surfaces of 316L stainless steel: (a) and (A) against PTFE, (b) and (B) against PTFE-1, (c) and (C) against PTFE-2, and (d) and (D) against PTFE-3. (a)–(d) are for dry friction and (A)–(D) are for seawater lubrication.

can change the steel to form a metallic oxide, and since the PTFE particles more strongly adhere to the surface of the metallic oxide, this leads to a transfer film forming much more easily.[31] In particular, a large amount of PTFE stripped off the surface of the PTFE-1 specimen and became the transfer film during the wear process [Figure 8(B)]; this finally resulted in a significantly smaller coefficient of friction, one that was lower than either that of PTFE-2 or of PTFE-3.

CONCLUSIONS

In this study, the tribological behavior of PTFE-based composites filled with POB, BF, and EG against 316L stainless steel was studied. The wear mechanism and transfer film were analyzed. The results indicate that the content of EG influenced the microstructures and tribological performance of PTFE composites. The ball indentation hardness, coefficients of friction, wear rates, wear mechanism, and transfer film properties are different under dry friction and seawater lubrication conditions. The following conclusions can be drawn:

(1) The incorporation of POB, BF, and EG altered the microstructures of PTFE and greatly enhanced its hardness. The hardness reached its maximum when the content of EG was 2 wt %. The water absorption rate of composites slightly increased by incorporating these fillers. The hardness of composites was seldom affected by water absorption.
(2) The wear rates of PTFE composites were much lower than that of pure PTFE under both dry friction and seawater lubrication conditions, although the coefficients of friction of composites were a little higher. The composite including 3 wt % EG had the lowest coefficient of friction, being only 10.38% that of pure PTFE. Under seawater lubrication, all the composites exhibited lower coefficient of friction (decreasing about 0.03–0.07) but showed more serious wear than under dry friction. The PTFE composite incorporating 2 wt % EG exhibited the best friction and wear performance characteristics among those materials tested.

(3) Under both dry friction and seawater lubrication, the wear mechanism changed from serious abrasive wear of pure PTFE to slight adhesion wear of PTFE composites. The worn surface of the counterpart steel showed no signs of transfer film but some wear debris under dry sliding. In contrast, under seawater lubrication, uniform and consecutive transfer films were formed on the steel worn surface. This was one of the causes of the decreasing coefficient of friction.

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