



Article

Tribological Properties of Porous PEEK Composites Containing Ionic Liquid under Dry Friction Condition

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Abstract: NaCl particles were added into Polyetheretherketone (PEEK) and its composites to produce porous PEEK-based materials by washing NaCl away after the high-temperature compression molding process. After that, an ionic liquid was added into the porous materials under vacuum condition. Carbon fibers (CF), as reinforcement, and PTFE, as an internal solid lubricant, were employed to prepare PEEK composites. Tribological properties under dry friction condition were studied on a ring-on-disc tribo-meter. The influence of CF and PTFE on tribological properties was carefully investigated. The results indicated that, in comparison with traditional PEEK composites (CF/PTFE/PEEK), the porous PEEK composites containing ionic liquid showed much better tribological properties. It is found that CF can help PEEK form effective pores to suck in the ionic liquid resulting in a better tribological performance. CF reinforced porous PEEK containing ionic liquid (p-CF/PEEK + IL) demonstrated the lowest friction coefficient (27% of CF/PTFE/PEEK) and the lowest wear loss (only 0.9% of CF/PTFE/PEEK). Long time tribological test revealed that the wear mass loss comes from the running-in period, while its wear is negligible after this period. It is also found that the addition of PTFE has a negative influence on the tribological behaviors, especially under high sliding velocity and applied load.

Keywords: polymer; tribology; wear; porous; ionic liquid; dry friction

1. Introduction

Self-lubricating engineering polymers, a kind of materials that can reduce friction by forming a third transfer film during sliding, are ideal sealing and wear-resistant materials [1–4]. Polymer-based self-lubricating composites coupled with steel under dry friction condition have many advantages over steel-steel pair under the oil-lubricated condition, such as light weight, strong resistance to high temperature and high vacuum, green with less contamination, etc. [5,6]. Solid lubricants, e.g., PTFE and graphite, and short fibers like carbon fibers (CF) and glass fibers (GF) are normally incorporated in self-lubricating polymers to improve their properties [7,8]. It is well-known that the fibers can increase the mechanical strength and loading capacity, while the solid lubricants contribute to lower friction coefficient and wear [9,10]. The friction coefficient and wear are generally higher than those under oil lubrication [11–13].

It has been proved that using a small amount of lubricant (oil-less condition) can greatly reduce the friction and improve the wear resistance of polymer composites [14]. Loy and Sinha et al. [15] applied an ultrathin layer of perfluoropolyether (PFPE) and Multiply-Alkylated Cyclopentane (MAC)

lubricants on the surface of PEEK. Samyn et al. [16] added silicon oil to the CF/Polyimide composites by mixing the oil with the matrix and the fillers before conventional processing. Guo et al. [17] incorporated liquid-filled capsules to the epoxy. Wang et al. [18] fabricated the porous PEEK composites by mold-leaching and vacuum melting process under high temperature. The NaCl has been used as micron pore-forming agent, and the mesoporous titanium oxide whiskers has also been applied to strengthen the suction and exudation of the liquid. During the friction process, the pre-stored internal lubricant enters the contact surface and forms an effective lubricating film, the friction and wear are greatly reduced.

Nowadays, more attention has been given in developing and using ionic liquids (ILs) as lubricants for various contacts and under harsh operating conditions. The combination of polymer composites and IL lubricants is promising [19,20]. In the previous work, we realized the high load capacity of pure PEEK-steel contacts under ILs lubrication. The contacts can give low friction and negligible wear under harsh conditions of 10 MPa, 0.7 m/s [21]. Due to the advantages of oil-less lubrication and convenience of polymer composites' fabrication, the IL lubricants can be used to polymer composites through the abovementioned ways [22–24]. Zhu et al. [25] filled porous PEEK composites with IL lubricants and achieved great improvements in antifriction and anti-wear properties. Under the effect of temperature and applied load, partial ionic liquid between the contacting surface areas can react with the steel counterpart surface by tribo-chemistry and form an effective lubricating film. However, the porous composites were designed based on the compositions for dry friction condition without internal oil. The influence of fibers and internal solid lubricant on the porous polymer composites containing liquid lubricant has not been studied.

The aim of this work was to find out the influence of fiber reinforcement and solid lubricant on the tribological properties of porous PEEK composites containing IL lubricants. We used PEEK as the matrix and NaCl as micron pore-forming agent. Pure porous PEEK (p-PEEK), porous PEEK composites strengthened with CF (p-CF/PEEK) and CF in company with PTFE (p-CF/PTFE/PEEK) were fabricated. One common ionic liquid, 1-butyl-3-methylimidazolium tetrafluoroborate [BMIm][BF₄], was added into the porous materials under vacuum condition. Tribological properties under dry friction condition were studied on a ring-on-disc tribo-meter, and the mechanisms were also discussed.

2. Experiment

2.1. Material Preparation

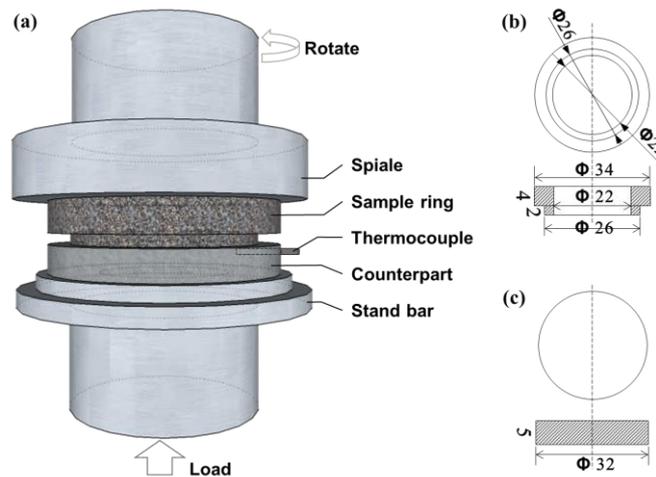
The PEEK powder in 10 µm size was purchased from Victrex plc (Victrex 450PF, UK). CF with an average diameter of 20 µm and the average length of 150 µm was supplied by Nanjing Fiberglass Research & Design Institute (Nanjing, China). PTFE powder in 25 µm size was supplied by DuPont (7A J, USA). [BMIm][BF₄] (99%; Linzhou Keneng Materials Technology Co., Ltd., Linzhou, China) were used as received. NaCl particle (purity ≥ 99.9 wt%, China National Medicines Corporation Ltd., Shanghai, China) was ground into smaller particles and the particles in size of 74–105 µm were filtered through sieves.

PEEK, CF and NaCl were dried at 120 °C for 4 h. Then, the fillers were blended with PEEK powder according to the desired percentages in Table 1. All the blends were processed by high-temperature compression molding at 355 ± 5 °C and 10 MPa for 90 min. After molding, the specimens were cut into a shape as in Figure 1, with an external diameter of 26 mm, an inner diameter of 22 mm, and a shoulder height of about 2 mm.

The composites with NaCl particles were immersed in water with a PTFE reactor, which was then heated at 90 °C for 24 h to remove NaCl. After that, the PEEK composite surfaces were polished with P400 and P800 grinding paper in sequence for uniform roughness. The porous composites were heated at 120 °C for 10 h to remove the water before weighing with the balance (accuracy to 0.1 mg). Then the samples were immediately immersed in [BMIm][BF₄] under vacuum condition for 2 h. Finally, the samples were cleaned and weighed to evaluate the liquid amount.

Table 1. Materials in the fabrication of different PEEK composites.

PEEK Composites	PEEK (%)	PTFE (%)	CF (%)	NaCl (%)
CF/PTFE/PEEK	70	15	15	0
p-PEEK	70	0	0	30
p-CF/PEEK	55	0	15	30
p-CF/PTFE/PEEK	40	15	15	30

**Figure 1.** Schematic diagram of (a) friction and wear test by ring-on-disc contact; (b) PEEK composites and (c) steel counterpart (in mm).

2.2. Friction and Wear Tests

Friction and wear tests were carried out with the configuration of a ring-on-block contact on MPX-2000 friction and wear tester (Xuanhua Testing Factory, Zhangjiakou, China), which was in accordance with the diagram shown in Figure 1. The round block counterpart is made of AISI 1045 steel, which has an external diameter of 32 mm and a thickness of 5 mm (Figure 1c). Prior to the test, the counterpart was polished with P400 and P800 grinding paper in sequence for uniform contact, then the weight was tested and recorded.

All the tests were performed at a room temperature of 25 ± 3 °C and relative humidity of $50 \pm 5\%$. Test conditions for the porous PEEK composites containing IL are listed in Table 2, where the corresponding PV values (pressure times velocity) are also given. The test duration was 4 h. The contact temperature was approximately measured with a thermocouple, which was 0.5 mm below the interface. After the test, the counterpart was weighed to obtain the weight gain (m_{con}) caused by the transferred IL from PEEK composites and/or the worn particles. If there are obvious worn particles, we would clean them before weighing. PEEK composites were cleaned with tissue to remove the absorbed IL and worn particles on the surfaces. Then the composites were dried at 120 °C for 12 h before weighing, and the mass loss of the composites was recorded as m_{com} . Finally, the wear mass loss of the PEEK composites was calculated using $m_{com} - m_{con}$. The friction coefficient and contact temperature for each test were calculated by averaging the steady-state values. Three replicate tests were carried out and the average values of friction coefficient, wear mass and contact temperature were reported.

Table 2. Test conditions for porous PEEK composites containing IL.

No.	Applied Load (N)	Linear Velocity (m/s)	PV Value (MPa·m/s)
1	200	1.4	1.87
2	200	2.1	2.80
3	300	1.4	2.80

2.3. Characterizations

The surfaces and internal structure of the porous PEEK composites were studied with an optical microscope and a scanning electron microscope (QUANTA-200 SEM, FEI, Hillsboro, OR, USA). To investigate the influence of porous structure on liquid wettability, the contact angle between the ionic liquid and the composites (the nonporous PEEK composites, porous PEEK composites with IL) were tested by an SL2008 automatic contact angle detector (HengPing Instrument and meter Factory, Shanghai, China). The test was conducted under ambient conditions. After friction and wear tests, the worn surface morphologies of porous PEEK composites and the steel counterparts were investigated by the optical microscope and HITACHI TM3000 SEM (HITACHI, Tokyo, Japan).

3. Results and Discussion

3.1. Fabrication of Porous PEEK Composites

The surface and the internal structure of p-CF/PTFE/PEEK are shown in Figure 2. There are well-distributed holes on the surface and inside the composites. The diameters of the holes well agree with the sizes of the NaCl particles. After the suction of IL, the mass gain was calculated and the IL content in porous PEEK composites is given in Table 3. The porous composites with CF show obviously higher IL content than p-PEEK, indicating that the addition of CF can enhance the IL absorption.

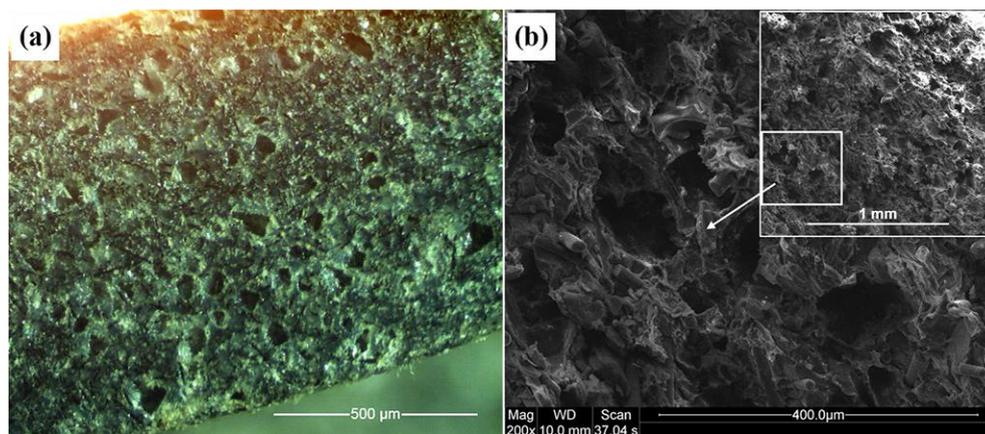


Figure 2. (a) Optical micrograph of p-CF/PTFE/PEEK surface ($\times 40$); (b) SEM image of the internal structure of p-CF/PTFE/PEEK ($\times 200$).

Table 3. The IL content in porous PEEK and its composites.

PEEK Composites	p-PEEK	p-CF/PEEK	p-CF/PTFE/PEEK
IL content (wt%)	1.1	2.7	3.4

Figure 3 compares the contact angles of [BMIm][BF₄] with different PEEK composites. The addition of PTFE leads to the decreased wettability, and the porous structure further decreases the wettability. After the suction of IL, the porous PEEK composites show better wettability. The p-CF/PEEK + IL still has better wettability than p-CF/PTFE/PEEK + IL.

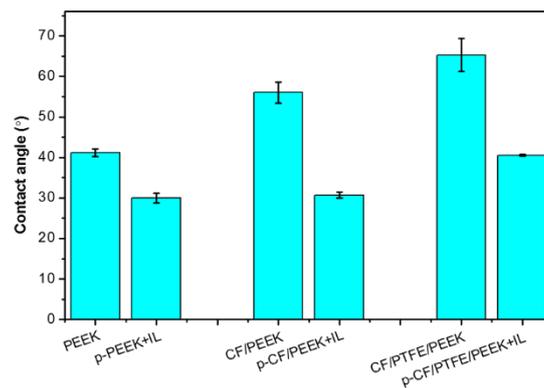


Figure 3. The contact angle of the IL on different PEEK composites.

3.2. Friction and Wear Properties

Under 200 N and 1.4 m/s, the mean friction coefficient, contact temperature and wear mass of different composites are shown in Figure 4. Under dry friction condition, the traditional CF/PTFE/PEEK gave a mean friction coefficient of 0.16 and wear mass loss of 46.13 mg. The contact temperature reached 315 °C, which has exceeded the long-term working temperature of PEEK. The high friction coefficient led to high contact temperature, which in turn increased the friction coefficient and wear [26,27]. In comparison with CF/PTFE/PEEK, porous PEEK composites containing ionic liquid show much better tribological properties. For p-PEEK + IL, the friction coefficient and contact temperature were obviously reduced, but the friction coefficient was unstable (Figure 5a). The composites reinforced with CF and CF + PTFE showed lower and stable friction during the testing process. CF reinforced porous PEEK containing ionic liquid (p-CF/PEEK + IL) showed the lowest friction coefficient and the lowest wear loss, which was 27% and 0.9% of CF/PTFE/PEEK. The filled CF can share the applied load and prevent the deformation of PEEK matrix [28]. Under the testing condition in this work, the reduced deformation is helpful to provide space for liquid release and storage. It is found that the addition of PTFE has no observed positive or a negative influence on the tribological behaviors of porous PEEK composites containing ionic liquid. Figure 5b shows the online friction coefficient and contact temperature. In the running-in period, the temperature remarkably increased and the friction coefficient was decreased. After one hour, the temperature and friction coefficient became steady and stable. After the test, IL was observed on the specimen and the contact faces.

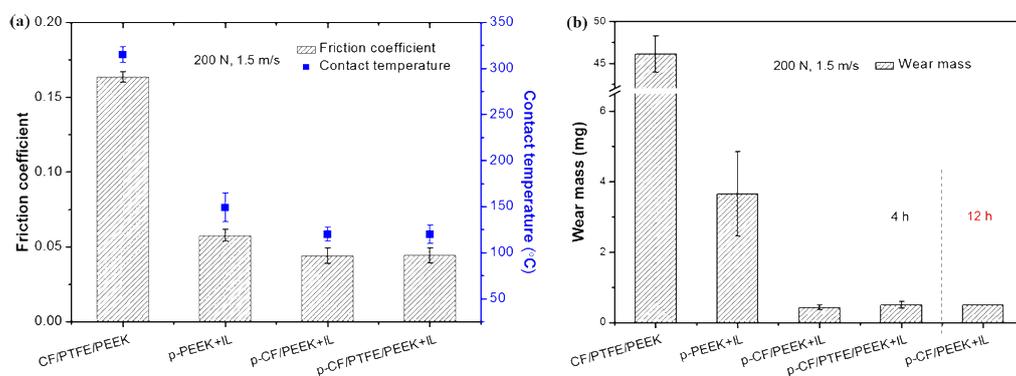


Figure 4. (a) Mean friction coefficient and contact temperature of different composites; (b) wear mass of the composites in 4 h test, and of p-CF/PEEK + IL in 12 h test. (test condition: 200 N, 1.4 m/s).

We extended the friction and wear test to 12 h with p-CF/PEEK + IL since it was the best performer in the 4 h test. The friction coefficient was stable and steady during the long-time test and the wear

mass loss was similar with that in the 4 h test. This indicated that the wear mass loss came from the running-in period and then became negligible. After the internal IL enters the frictional interface, the liquid prevents the direct contact between the friction counterparts. Due to the excellent lubricating ability and high stability of the IL, the oil-less lubrication maintains good performance for a long time.

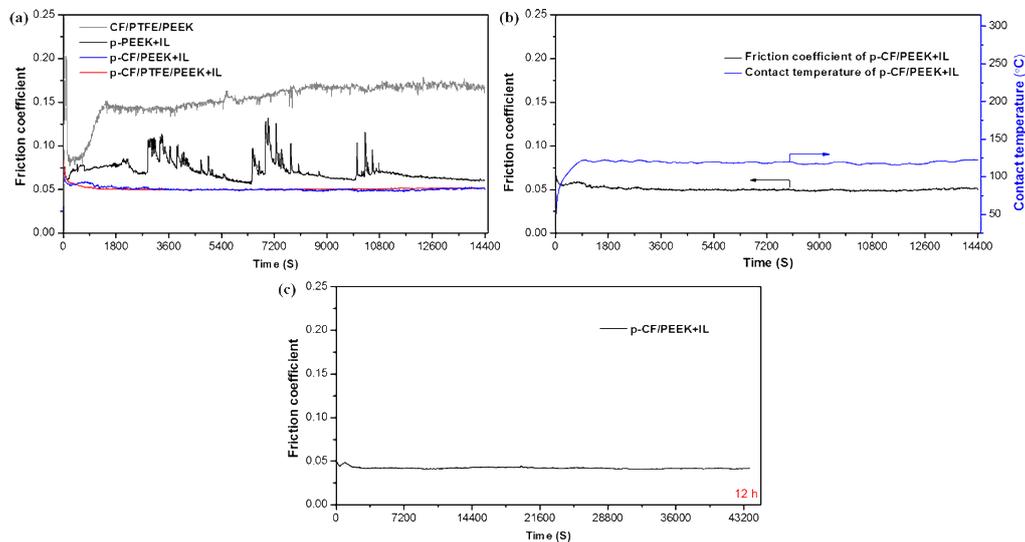


Figure 5. (a) Online friction coefficient of different composites; (b) the trend of contact temperature and friction coefficient of p-CF/PEEK + IL; (c) online friction coefficient of p-CF/PEEK + IL in 12 h test. (test condition: 200 N, 1.4 m/s).

It is well-known that PV value has a sound influence on the tribological properties. Tribological properties of the CF reinforced PEEK composites containing IL (p-CF/PEEK + IL and p-CF/PTFE/PEEK + IL) were further studied under higher sliding velocity and higher applied load. As the PV value increased, the advantage of p-CF/PEEK + IL became more obvious (shown in Figure 6). At higher velocity, the wear mass loss was 24% lower than p-CF/PTFE/PEEK + IL. When the higher load was applied, the wear mass loss was 12% lower than p-CF/PTFE/PEEK + IL. The wear mass of p-CF/PEEK + IL was respectively 3.6 and 1.4 times of that under 200 N, 1.4 m/s. Besides, friction coefficient is also affected by the increased velocity and speed. Figure 7 shows online friction coefficient and the corresponding contact temperature. p-CF/PTFE/PEEK + IL has higher friction coefficient, which leads to higher contact temperature. It needs to note that during running-in period, the friction coefficient of p-CF/PEEK + IL is much lower than p-CF/PTFE/PEEK + IL. This maybe because p-CF/PEEK + IL has more adhered IL on the surface before testing for its higher wettability by the IL.

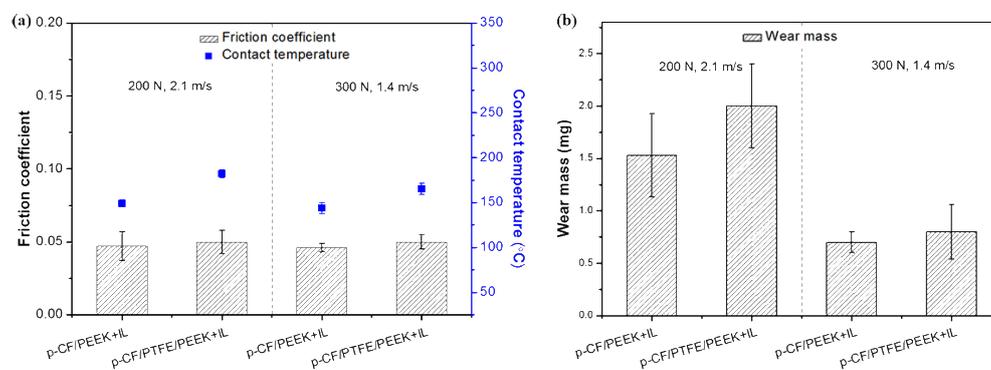


Figure 6. Mean friction coefficient, contact temperature (a) and wear mass (b) of p-CF/PEEK + IL and p-CF/PTFE/PEEK + IL. (test conditions: 200 N, 2.1 m/s, and 300 N, 1.4 m/s).

In this study, the contact temperature was very sensitive to PV value. It is known that the temperature increase is positively related to the friction coefficient, sliding velocity and applied load, and the resulting temperature will, in turn, affect the tribological properties [27]. To show the dependence of tribological properties on PV value, the friction coefficient and temperature increase are given in Figure 8. It is indicating that the contact temperature was more sensitive to the velocity, and the trends of temperature increase were consistent with the wear mass losses. At higher temperature, the liquid viscosity was decreased and easier for the counterpart to scratch the PEEK composites. The higher wear of p-CF/PTFE/PEEK + IL was thought to be caused by the lower liquid viscosity and poor scratch resistance of PTFE.

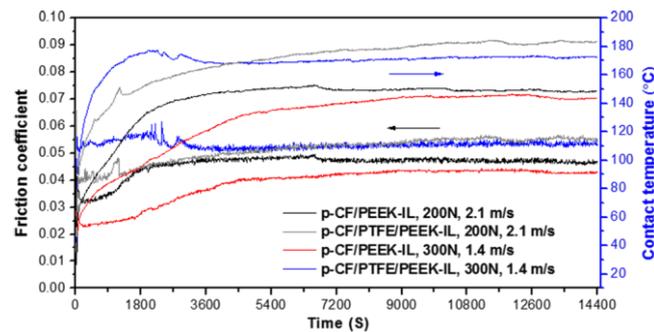


Figure 7. Online friction coefficient (lower lines) and the corresponding contact temperature (upper lines) of p-CF/PEEK + IL and p-CF/PTFE/PEEK + IL. (test conditions: 200 N, 2.1 m/s, and 300 N, 1.4 m/s).

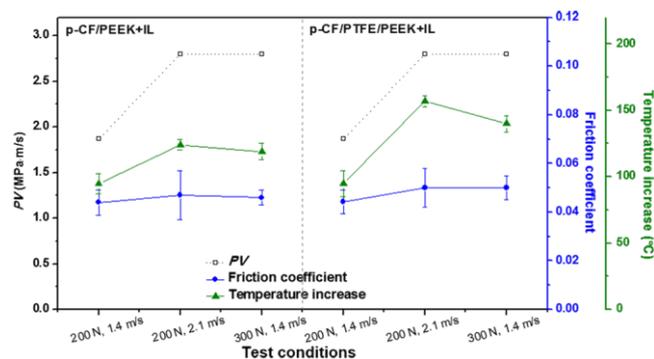


Figure 8. Influence of test conditions (PV value) on friction coefficient and contact temperature of CF reinforced PEEK composites containing IL.

3.3. Studies of Worn Surfaces

Figure 9 shows the optical micrographs and TM images of porous PEEK composites containing IL after test under 200 N and 1.4 m/s. After the friction was tested, the pure p-PEEK + IL showed a rough surface with micro furrows and worn particles. Both the p-CF/PEEK + IL and p-CF/PTFE/PEEK + IL showed slight furrows and little-worn particles. It can be found that the CF on the surface was not damaged, and the surfaces became smoother. The reinforcement of mechanical strength should be an important factor to improve the performance. For p-CF/PTFE/PEEK + IL, the PTFE is well distributed in the composites, and there is no indication of the deformed PTFE/PEEK mixture as CF/PTFE/PEEK under dry friction condition [29].

Figure 10 shows the worn surfaces of the counterparts against the composites in Figure 9. The counterpart against p-PEEK + IL shows many deep furrows, while p-CF/PEEK + IL and p-CF/PTFE/PEEK + IL have slight marks. There was no observed solid transfer film as the oil-free composites under dry friction condition. For CF/PTFE/PEEK under dry friction condition, as the

schematic illustration shows in Figure 11a, the internal PTFE forms an easy-shear transfer film under the interfacial heat and frictional force [30]. But for the ionic liquid contained composites (Figure 11b–d), the internal liquid enters the contact surface for the effects of load and temperature. Most part of the surface is covered with the IL, especially for the counterpart. The IL is thought to strongly adhere to the steel surface through electrostatic attraction, and generate the physicochemically adsorbed tribo-thin film [21,31]. In this condition, the addition of PTFE has very limited effect on the tribological behaviors.

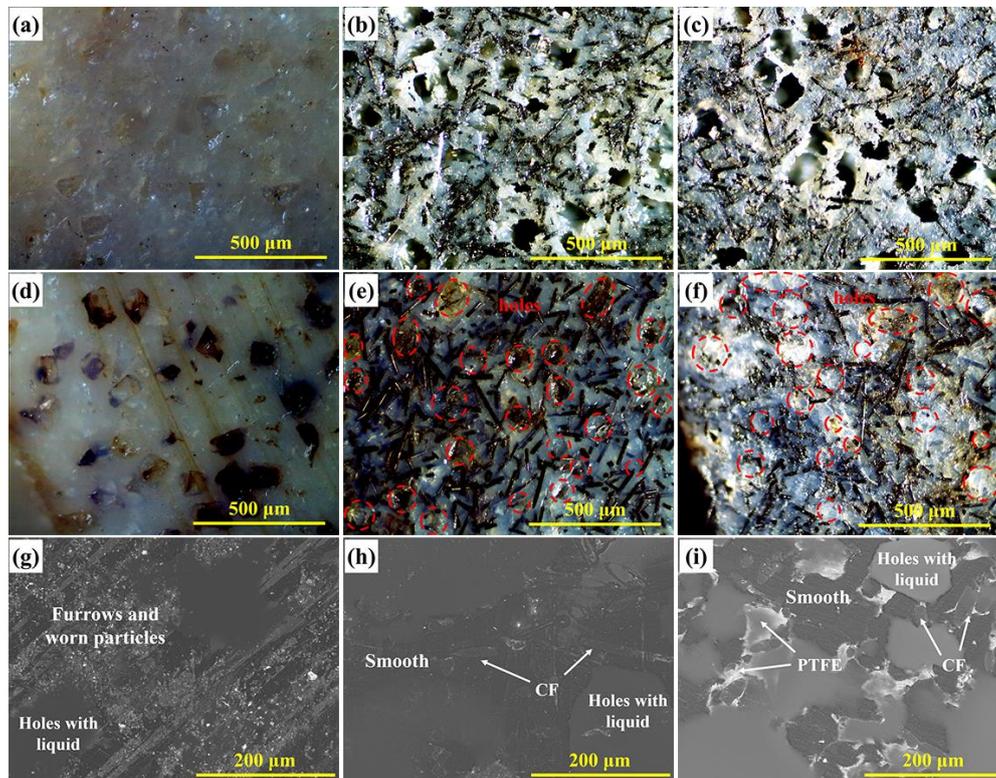


Figure 9. Optical micrographs ($\times 100$) of (a) p-PEEK, (b) p-CF/PEEK and (c) p-CF/PTFE/PEEK; (d) p-PEEK + IL, (e) p-CF/PEEK + IL and (f) p-CF/PTFE/PEEK + IL after friction testing. (g–i) are the TEM images ($\times 400$) of (d–f). (test condition: 200 N, 1.4 m/s, 4 h)



Figure 10. TEM images ($\times 1500$) of the counterpart against (a) p-PEEK + IL, (b) p-CF/PEEK + IL, (c) p-CF/PTFE/PEEK + IL, respectively. (Test condition: 200 N, 1.4 m/s, 4 h)

From the analysis above, it is obvious that the CF reinforcement could enable the porous composites containing IL with better tribological properties by forming effective pores to suck in the ionic liquid and reinforcing the mechanical strength, but there is no need to add PTFE from a frictional performance perspective. From the perspective of mechanical properties, the absence of PTFE is beneficial to avoid the decrease of the load-carrying capacity of porous PEEK composites [11].

The difference in wetting ability has little influence on the tribological behaviors. But in the long-term application, it is important to possess better wettability for the oil storage and slow release.

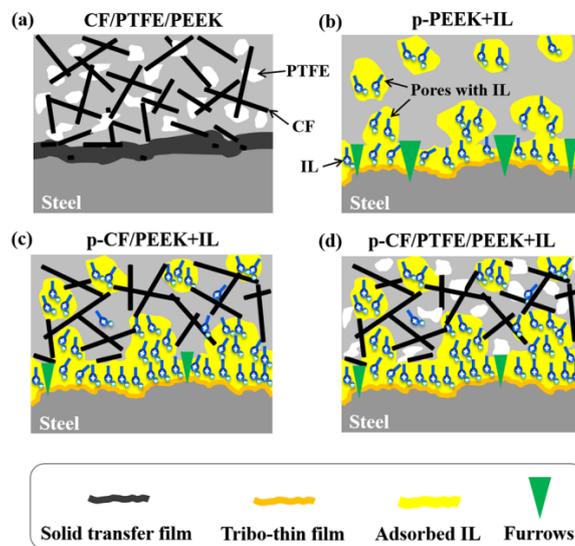


Figure 11. A schematic illustration of friction and wear of different PEEK composites: (a) CF/PTFE/PEEK, (b) p-PEEK + IL, (c) p-CF/PEEK + IL, (d) p-CF/PTFE/PEEK + IL.

4. Conclusions

In the present work, the tribological behavior of porous PEEK composites containing ionic liquid was investigated. The influence of fiber reinforcement and solid lubricant was carefully investigated. The following conclusions can be drawn:

- (1) In comparison with traditional PEEK composites (CF/PTFE/PEEK), porous PEEK composites containing ionic liquid show much better tribological properties.
- (2) CF reinforced porous PEEK containing ionic liquid (p-CF/PEEK + IL) show the lowest friction coefficient and the lowest wear mass loss, which is 27% and 0.9% of CF/PTFE/PEEK under the specific condition.
- (3) Long time tribological test indicates that the wear mass loss comes from the running-in period, while its wear mass loss is negligible after this period.
- (4) The addition of PTFE has no observed positive or a negative influence on the tribological behaviors, especially under high sliding velocity and high applied load.

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Author Contributions: Xin Feng and Yijun Shi conceived and designed the experiments; Xianzhu Huang performed the experiments; Xianzhu Huang and Jian Wu analyzed the data; Xiaohua Lu contributed reagents/materials/analysis tools; Jian Wu wrote the paper.

Conflicts of Interest: All authors declare no conflict of interest.

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