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Potassium Titanate Whisker/Graphene Multi-Dimensional Fillers to Improve the Wear Resistance of Poly(Ether Ether Ketone) Composite

Zhengjia Ji *, Mingliang Ji, Qiuchen Lv, Minggui Liang and Chao Xu

School of Mechanical Engineering, Qinghai University, Xining 810016, China
* Correspondence: jzj@qhu.edu.cn

Abstract: To improve the tribological performance of poly(ether ether ketone) (PEEK), a potassium titanate whisker/graphene multi-dimensional hybrid filler was proposed. Hybrid fillers with one-dimensional (1D) potassium titanate whiskers and two-dimensional (2D) graphene nanosheets in different ratios were fabricated using direct mixing and grafting methods. The potassium titanate whiskers and graphene nanosheets are an excellent combination, as confirmed by SEM and FTIR. Furthermore, PEEK/hybrid filler composites with different mass percentages of fillers were prepared and investigated systematically. It was found that introducing multi-dimensional hybrid PTWs–GNPs (volume ratio 1:3) fillers led to the wear rate being as low as 3.214×10^{-6} $mm^3N^{-1}m^{-1}$, reduced by 60% compared with pure PEEK. In addition, the wear mechanism of PEEK composites was also investigated. The results demonstrate the superior tribological properties of the PEEK composites with multi-dimensional hybrid PTWs–GNPs fillers.

Keywords: potassium titanate whisker; graphene; composites materials; tribological performance; wear mechanism

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1. Introduction

With rapid progress in the field of polymer composites, poly(ether ether ketone) (PEEK) has attracted more and more attention due to its excellent high-temperature resistance, corrosion resistance, and self-lubricating properties [1]. At present, PEEK polymers are widely used in the mechanical industry, medical facilities, and marine equipment [2,3]. However, pure PEEK exhibits relatively low wear resistance in several cases, and thus it can no longer satisfy wear resistance requirements. With extreme operating conditions encountered in many applications, PEEK is required to have improved and stable tribological performance. In order to improve the tribological properties of PEEK, many methods have been developed; the following four methods are the most common: polymer blending modification [4–6], fiber filling modification [7], inorganic filler filling modification [8–10], and lubrication filler modification [11–13]. In addition, the effect of exposure to extreme environments on the tribological properties of PEEK will inevitably lead to premature wear and early failure [14]. Introducing different types of fillers into polymers is the most common method of improving or modifying the tribological properties of PEEK polymers.

Many two-dimensional (2D) layered materials are widely employed as additives, such as graphene [15], MoS_2 [16,17], h-BN [18], transition metal carbide/nitride [19], and phosphorene [20,21], because of their excellent tribological performance. Among these various types of fillers, graphene nanosheets (GNPs) are particularly preferable. Previous papers are devoted to the addition of graphene into polymers and the examination of the tribological behaviors of these composites [22]. Furthermore, potassium titanate whiskers (PTW) are a class of promising reinforcement fillers that significantly improve the mechanical, thermal, and tribological properties of various polymer composites, due to their high NIR reflectivity, low thermal conductivity, abrasion resistance, and excellent mechanical properties [23]. The unique crystal structure of PTW, making it have outstanding performance, has been revealed in a previous study. However, it is difficult to directly obtain composites with whiskers due to its smooth surface. To further improve the tribological properties of composites, an effective strategy to avoid aggregation is to form hybrid fillers and then add them to the polymer matrix. In recent years, a series of hybrid fillers were fabricated, and the hybrid fillers have previously been shown to reinforce polymers [24–26]. Hua et al. found that grafting graphene oxide on glass fiber as an additive can effectively improve the frictional stability of composites [27]. Feng et al. investigated the tribological properties of PEEK composite reinforced by MoS_2-modified carbon fiber. The results showed that the wear resistance was greatly improved by MoS_2-modified carbon fiber [28]. Compared with modifying the surface of the fillers and adding a single filler in polymer matrices, introducing hybrid fillers presents more potential applications and better performance [29–32]. Both PTWs and GNPs are ideal candidates for improving the tribological performances of the polymer composites. Furthermore, multidimensional hybrid fillers, which are composed of one-dimensional (1D) fillers and two-dimensional (2D) fillers, display great potential as high-performance polymer composites [33]. Therefore, exploring a PTWs(1D)–GNPs(2D) multidimensional hybrid filler is highly significant for obtaining anti-friction and anti-wear PEEK composites.

In this study, a facile and novel strategy was proposed for improving the tribological properties of PEEK composites through multidimensional hybrid PTWs–GNPs fillers and the strengthening of their interfacial interaction. Scanning electron microscopy, Fourier transform infrared spectroscopy, and friction tester were used to evaluate microstructures and tribological properties.

2. Experimental Details

2.1. Materials

PEEK powders (300-mesh size) were purchased from Jilin University High-Technology Co., Ltd. (Changchun, China). Graphene was purchased from XF Nano, Inc. (Nanjing, China), and PTW was obtained from Nantong Aoxin Electronic Technology Co., Ltd. (Haian, China) Potassium silicate and γ-aminopropyl triethoxysilane (KH550) were supplied by Tianjin Kaitong Chemical Reagent Co., Ltd. (Tianjin, China) and Sahn Chemical Technology (Shanghai) Co., Ltd. (Shanghai, China), respectively.

2.2. Synthesis of Multi-Dimensional Fillers

2.2.1. PTW-Graphene Blend Fillers (P-G)

First, the potassium titanate whisker and graphene were mixed at a certain volume ratio with ethanol solvent, and then ultrasonically dispersed for 30 min and magnetic stirring at 600 r/min for 15 min to acquire a homogenous suspension. The P-G composite fillers were obtained by drying the suspension at 100 °C in a vacuum oven. The different volume ratios (potassium titanate whisker/graphene) of 1:1 and 1:3 were named P-G1 and P-G2, respectively.

2.2.2. PTW–Graphene Multi-Dimensional Compound Fillers (PGMFs)

The PTW–graphene multi-dimensional compound fillers were synthesized via graft route as follows. Firstly, 0.9 g sodium silicate and 6 g potassium titanate whiskers were added to deionized water and ultrasonically dispersed for 30 min, and then magnetically stirred with an additional 2 mL ethyl acetate, before being dropped into at 700 r/min for 4 h. The samples were washed with deionized water and dried to obtain silica coated whiskers. Subsequently, the silica coated whiskers were modified by using silane coupling agent KH550. In detail, 0.18 g KH550 was dissolved in a mixture of ethanol/deionized water (the mass ratio of ethanol to water is 1:9), and the mixture was homogenized by magnetic stirring for 10 min. Then, the silica-coated whiskers were mixed into the solution and magnetically stirred at a speed of 700 r/min at 60 °C for 1 h. The samples were washed with absolute ethyl alcohol three times and dried to obtain whiskers grafted KH550. Finally, the whiskers and graphene were successively dispersed in ethanol solvent for ultrasonic treatment for 30 min and magnetic stirring at 700 r/min for 1 h. The PGMFs were obtained after thoroughly washing with ethanol and drying. According to different volume ratios (potassium titanate whisker/graphene) of 1:1 and 1:3, they were named PGMF1 and PGMF2, respectively.

2.3. Fabrication of PEEK Composites

The preparation process of the PEEK composites is illustrated in Figure 1. With PEEK powder as a matrix and the filler prepared by the above two methods as reinforcement material, PTW–GNP/PEEK composites with different adding concentrations were fabricated. The weighted filler and PEEK were stirred by magnetic force for 1 h to obtain a homogenous suspension, and the PTW–GNP/PEEK mixed powder was dried by vacuum drying oven at 100 °C. The PTW–GNP/PEEK mixed powder was pressed into the mold at 10 MPa, and then sintered at 350 °C for 2 h and cooled for 6 h with the sintering furnace to obtain PEEK composites. Different quantities of PTW–GNP fillers were added in order to achieve final concentrations of 5 and 10 wt% in the PTW–GNP/PEEK composites. The resultant composite designations are shown in Table 1.

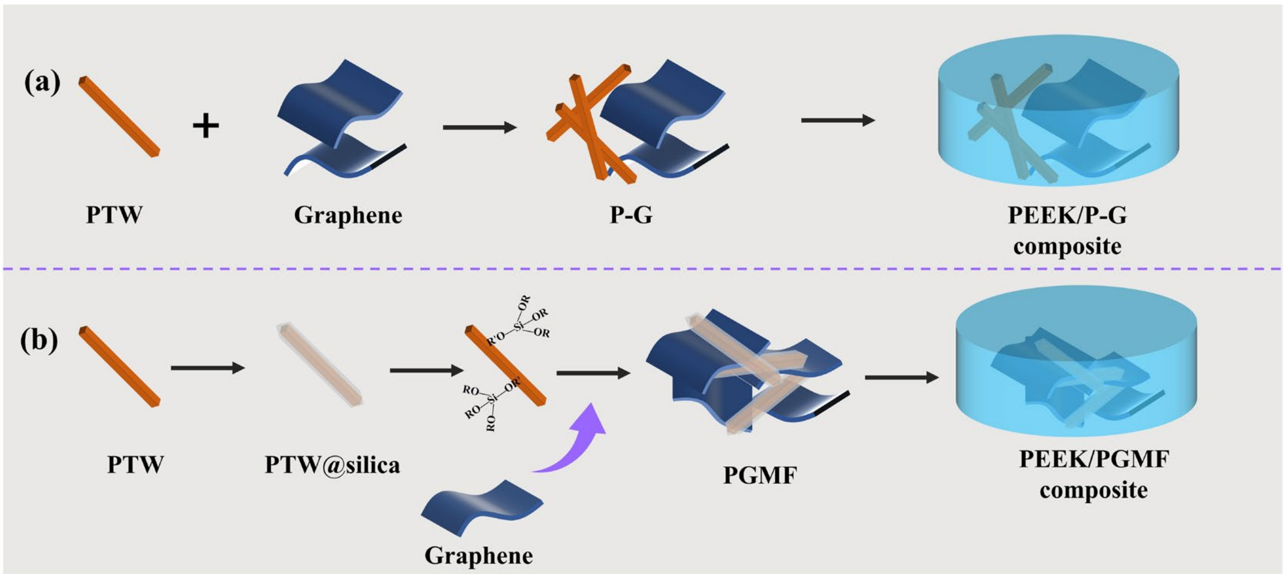

Figure 1. Schematic preparation process of the PEEK composites: (**a**) PEEK/P-G composite, (**b**) PEEK/PGMF composite.

Table 1. Composition of PEEK composites.

Sample No.	PEEK (wt%)	P-G1 (wt%)	P-G2 (wt%)	PGMF1 (wt%)	PGMF2 (wt%)
Neat PEEK	100				
PEEK/P-G1–5%	95	5			
PEEK/P-G1–10%	90	10			
PEEK/P-G2–5%	95		5		
PEEK/P-G2–10%	90		10		
PEEK/PGMF1–5%	95			5	
PEEK/PGMF1–10%	90			10	
PEEK/PGMF2–5%	95				5
PEEK/PGMF2–10%	90				10

2.4. Characterization

The tribological properties of the composites were determined using a reciprocating test rig (CFT-I). The PEEK composite disk with a diameter of 20 mm and a thickness of 5 mm is the lower sample. The steel ball (GCr15) with a diameter of 6 mm is the upper sample. The normal load is 10 N, the sliding velocity is 0.09 m/s, and the reciprocating stroke is 7 mm during the friction process. To ensure the accuracy of the test results, each sample was tested twice in the same condition. The wear rate of PEEK composites was calculated according to the following:

$$W = \frac{V}{FL} \quad (1)$$

where W (mm^3/Nm) is the wear rate, which is the wear volume of PEEK composite (V) per unit load per unit sliding distance. F and L are the applied load and the total sliding distance, respectively. Scanning electron microscopy (SEM) was conducted to analyze the morphologies of the fillers and the worn surface of the PEEK composites. A three-dimensional white light interference microscope (ZYGO NewView 5000, ZYGO, Middlefield, CT, USA) was used to observe the surface topography of the PEEK composites. Fourier transform infrared (FTIR) spectra were recorded with a Bruker Vertex 70 instrument (Bruker, Billerica, MA, USA) in a range between approximately 400 and 4000 cm^{-1}.

3. Results and Discussion

3.1. Microstructure Analysis of the Multi-Dimensional Hybrid Fillers

The morphologies of four types of fillers were studied by SEM analysis. As shown in Figure 2a, the SEM images showed that the surface of PTW was relatively smooth. When the volume ratio of PTW and graphene is 1:3 (Figure 2b), wrinkled graphene nanosheets can be clearly observed. Different from P-G1 and P-G2, which are prepared using a simple mixture, the PTW possessed a slightly rough surface and interconnected with graphene nanosheets to form a multidimensional structure in the PGMF1 (Figure 2c) and PGMF2 (Figure 2d). The silica layer and KH550 on the whisker surface could therefore offer groups and active sites, providing a critical bridging role between the whisker and graphene. Furthermore, it could be found that both the whiskers and graphene nanosheets are highly dispersed uniformly. The SEM results show the successful construction of the potassium titanate whisker/graphene multi-dimensional fillers via the grafting process.

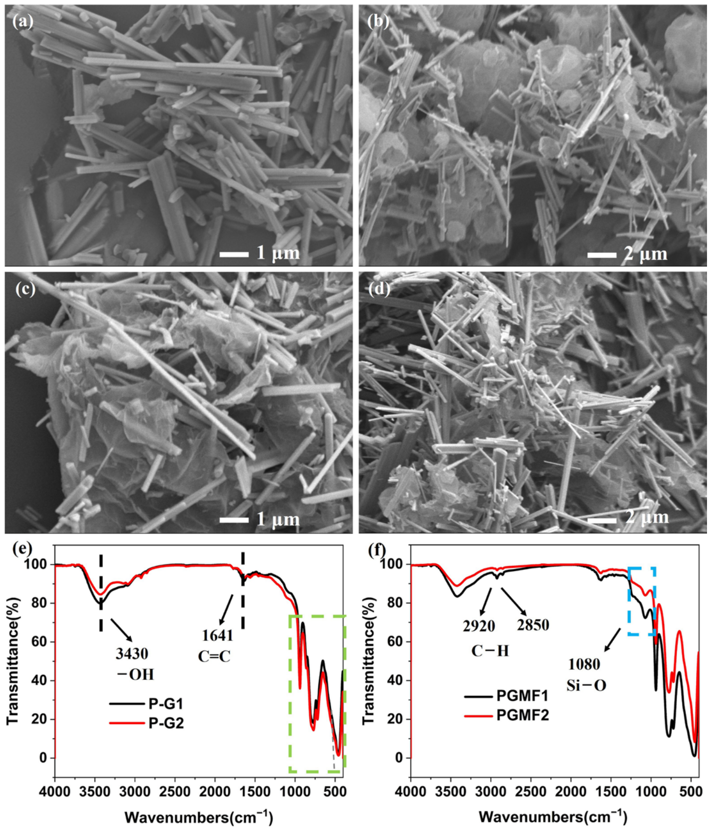

Figure 2. SEM images of P-G1 (**a**), P-G2 (**b**), PGMF1 (**c**), and PGMF2 (**d**) and FTIR spectra of the fillers (**e**,**f**).

To obtain more insight into the fillers, FTIR characterization was performed. The FTIR spectra of P-G1, P-G2, PGMF1, and PGMF2 are displayed in Figure 2e,f. The results proved the presence of O-H (at 3430 cm^{-1}), C=C (at 1641 cm^{-1}), and Ti-O (at 470 cm^{-1}) groups in all filler samples. Almost all the characteristic peaks in PTW and graphene can be observed in prepared fillers. Compared with P-G1 and P-G2, PGMF1 and PGMF2 exhibited a new absorption peak at 1080 cm^{-1}, which is assigned to the stretching vibration of Si-O in the silica and KH550. The silica in the surface of PTW is beneficial to grafting KH550 and further combining graphene. In particular, all the absorption peaks of PGMF2 become weak when the content of graphene increases, as shown in Figure 2f, indicating that the PTW was further wrapped by the graphene nanosheets and the interaction with each other enhanced. The FTIR results suggest the successful incorporation of PTW and graphene through the grafting of KH550, which is consistent with the SEM analysis.

3.2. Tribological Behaviors

The tribological properties of the PEEK composites are influenced by the filler structure and filler content [34]. Figure 3a shows the typical friction coefficient curves of PEEK composites with various fillers. It can be seen that the curves become smooth after adding fillers, indicating a more stable friction coefficient. This is mainly because the stick–slip behavior of the pure PEEK is eliminated by the addition of the fillers. Moreover, PEEK/PGMF2–10% exhibited the lowest friction coefficient and the average friction coefficient is 0.316 (Figure 3b). This is mainly due to the lubricating effect of graphene during friction, which has been proven by J.A. Puértolas et al. [12]. For PGMF2, the whiskers were wrapped in a large amount of graphene. Meanwhile, the results for wear tests are presented in Figure 3c,d. As shown in Figure 3c, the depth and width of the wear scar decrease significantly with the addition of the filler. In particular, the depth of PEEK/PGMF2–10% was only 4 µm. It indicates that the multi-dimensional fillers prevent the enlargement of wear scars effectively. Figure 3d shows the wear rates of PEEK composites. With the increase in filler content, the wear rate of all PEEK composites decreases just marginally. In addition, the wear rate of PEEK decreases by 54%, 44%, 58%, and 60% with the incorporation of 10 wt% P-G1, 10 wt% P-G2, 10 wt% PGMF1, and 10 wt% PGMF2, indicating a significantly positive influence of the multi-dimensional fillers on the resistance wear of PEEK matrix. It is also noted that the wear rates of PEEK/P-G exceeded that of PEEK/PGMF. This may be attributed to two reasons: one reason is the high interface bonding strength between the PTW and graphene via the grafting process, and the other reason is the synergistic effect of the load-carrying capacity of PTW and the lubrication capability of graphene. The PEEK/PGMF2–10% composite shows the best friction and wear performances, indicating that the excellent performances of PGMF2 made it an ideal potential filler for the PEEK composite.

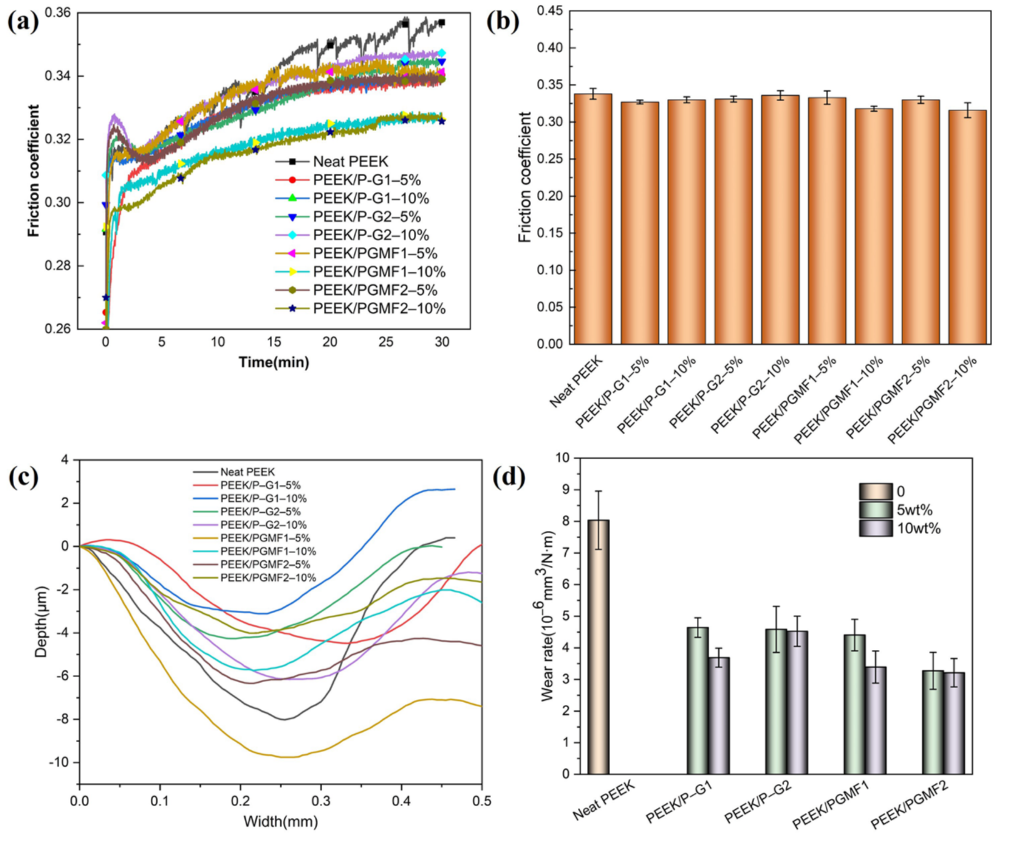

Figure 3. The friction coefficients versus time (**a**), the average friction coefficients (**b**), the profile of wear track (**c**), and the wear rates (**d**) of PEEK composites.

3.3. Wear Mechanism

To further elucidate the wear mechanism, the worn surfaces of PEEK composites after the friction process were analyzed using SEM. As shown in Figure 4, PEEK/P-G1–5% and PEEK/P-G1–10% show similar wear features, many clear holes and wear debris appear on the surface. The lower left corner of Figure 4a,b shows the high magnification of the wear area. The worn surface of PEEK/P-G1–10% presents slight furrows. It is suggested that the dominant wear mechanism is fatigue wear, accompanied by abrasive wear. With the increase in the graphene content in the filler, the furrows progressively disappear for PEEK/P-G2–5% (Figure 4c) and PEEK/P-G2–10% (Figure 4d), implying that the abrasive wear is suppressed. For PEEK/PGMF1–5% (Figure 4e) and PEEK/PGMF1–10% (Figure 4f), the amounts of furrows clearly increase, and the worn surfaces become rougher. This is principally because the PDMF1 filler, with strong mechanical strength and load-bearing capacity, was exposed to the worn surface. A small amount of graphene in the PDMF1 filler is not enough to provide lubrication. As shown in Figure 4g,h, the worn surfaces of PEEK/PGMF2–5% and PEEK/PGMF2–10% are clearly smooth without holes and wear debris. It is worth pointing out that the wear track of PEEK/PGMF2–10% is clearly narrow and shallow, which is consistent with the profile of the wear track. It is mainly attributed to the synergistic effect between PTW and graphene and excellent interfacial bonding between filler and matrix. On the one hand, the interlayer sliding of graphene and the strong load-bearing capability of the whisker take place at the contact interface, resulting in a significant decline in friction coefficient and wear rate. On the other hand, the 2D graphene nanosheet with a rough surface provides a larger contact area for the hybrid filler and matrix, as confirmed in previous studies [15,27,35].

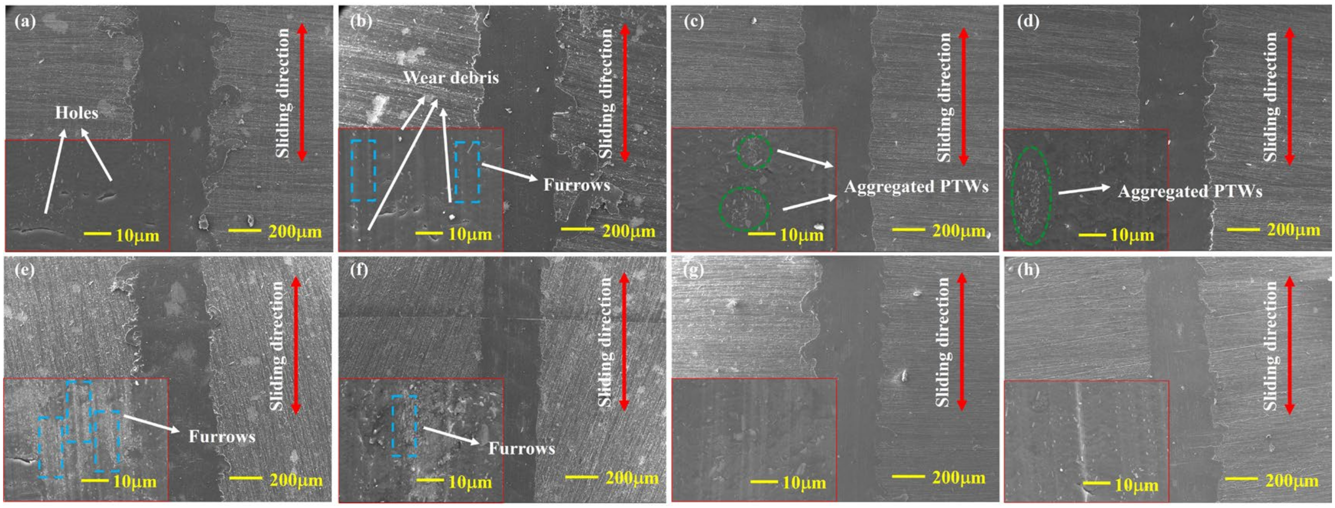

Figure 4. SEM images of the worn surface for the PEEK composites: (**a**) PEEK/P-G1–5%, (**b**) PEEK/P-G1–10%, (**c**) PEEK/P-G2–5%, (**d**) PEEK/P-G1–10%, (**e**) PEEK/PGMF1–5%, (**f**) PEEK/PGMF1–10%, (**g**) PEEK/PGMF2–5%, (**h**) PEEK/PGMF2–10%.

According to the results in Figure 4, the silica layer and KH550 play an important role in the connection between the whisker and graphene, thereby significantly improving the resistant wear of the PEEK composite.

Figure 5 shows the white light interferometry images of the neat PEEK and PEEK/PGMF composites after the friction test. The width and depth of the wear track of neat PEEK composites are greater than those of PEEK/PGMF1–5%, PEEK/PGMF1–10%, PEEK/PGMF2–5%, and PEEK/PGMF2–10%, indicating that the addition of the multi-dimensional hybrid fillers to the polymer matrix achieves a superior anti-wear performance. The main reasons for the low wear rate are the protection effect of graphene and the strong interaction between the fillers and PEEK matrix [36]. The protection effect alleviates pulling out of the whiskers from the matrix and the resultant distraction, contributing to the reduction in wear debris and the formation of a thin and stable transfer film. The neat PEEK presents an obvious pile-up of material at the sides of the wear tracks. The sizes of the chips in this region are relatively large. The pile-up at indent sides slightly decreased after adding fillers.

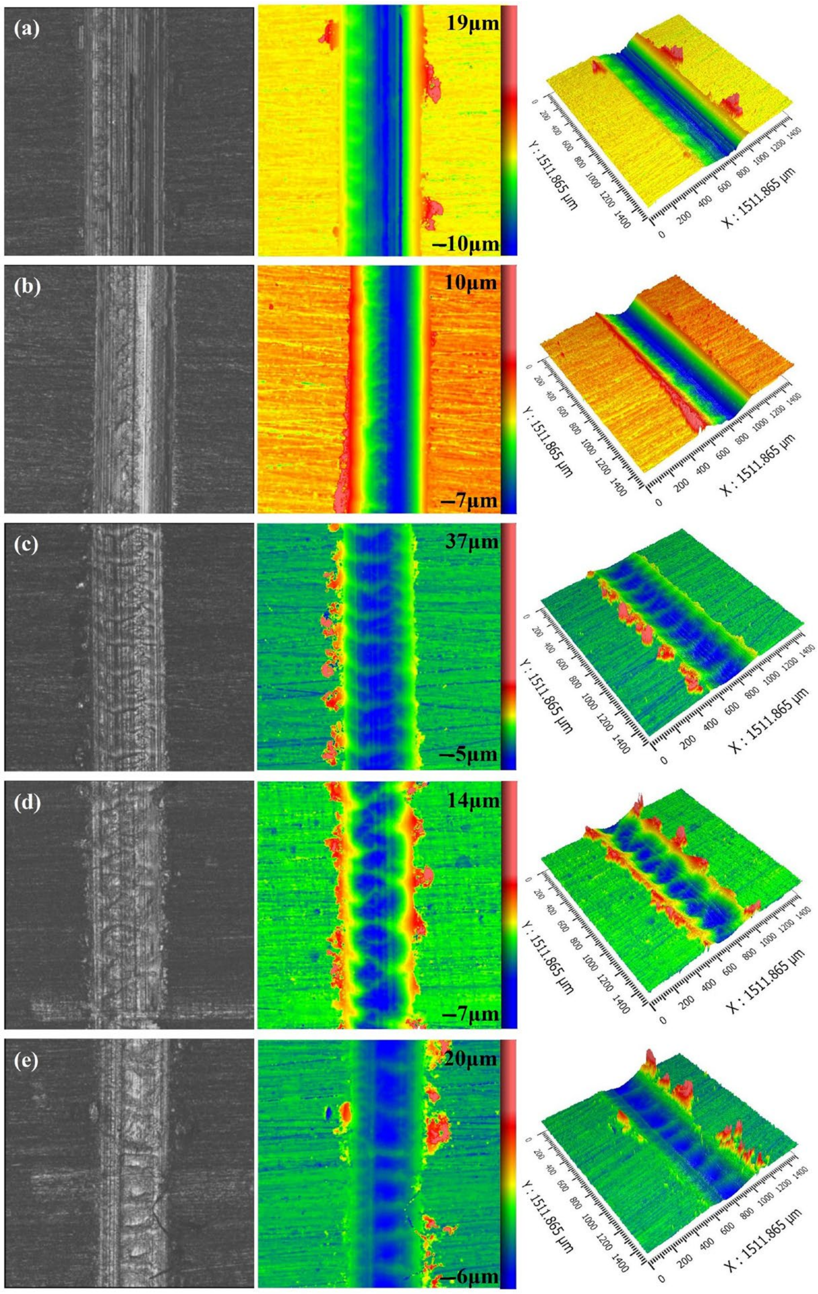

Figure 5. The white light interferometry images of the neat PEEK (**a**) and PEEK composites: (**b**) PEEK/PGMF1–5%, (**c**) PEEK/PGMF1–10%, (**d**) PEEK/PGMF2–5%, and (**e**) PEEK/PGMF2–10%.

Based on the analyses of the worn surface, the wear mechanism for the neat PEEK and PEEK composites is further investigated in the study. The schematic illustration of the wear mechanism is shown in Figure 6. After the friction, the PGMF exhibited good dispersibility, distributed uniformly PEEK matrix. It also reduced the abrasive wear to a certain extent. In comparison with neat matrix and P-G fillers, PGMFs with a significantly synergistic effect have greater load-bearing capability and form a uniform transfer film. The transfer film can play a crucial role in reducing the wear of the composite [37–39]. Moreover, the strong interaction between the graphene nanosheets and whiskers could restrain the increase in wear debris. Therefore, it could be concluded that the potassium titanate whisker/graphene multi-dimensional fillers effectively improve the wear resistance of PEEK composites.

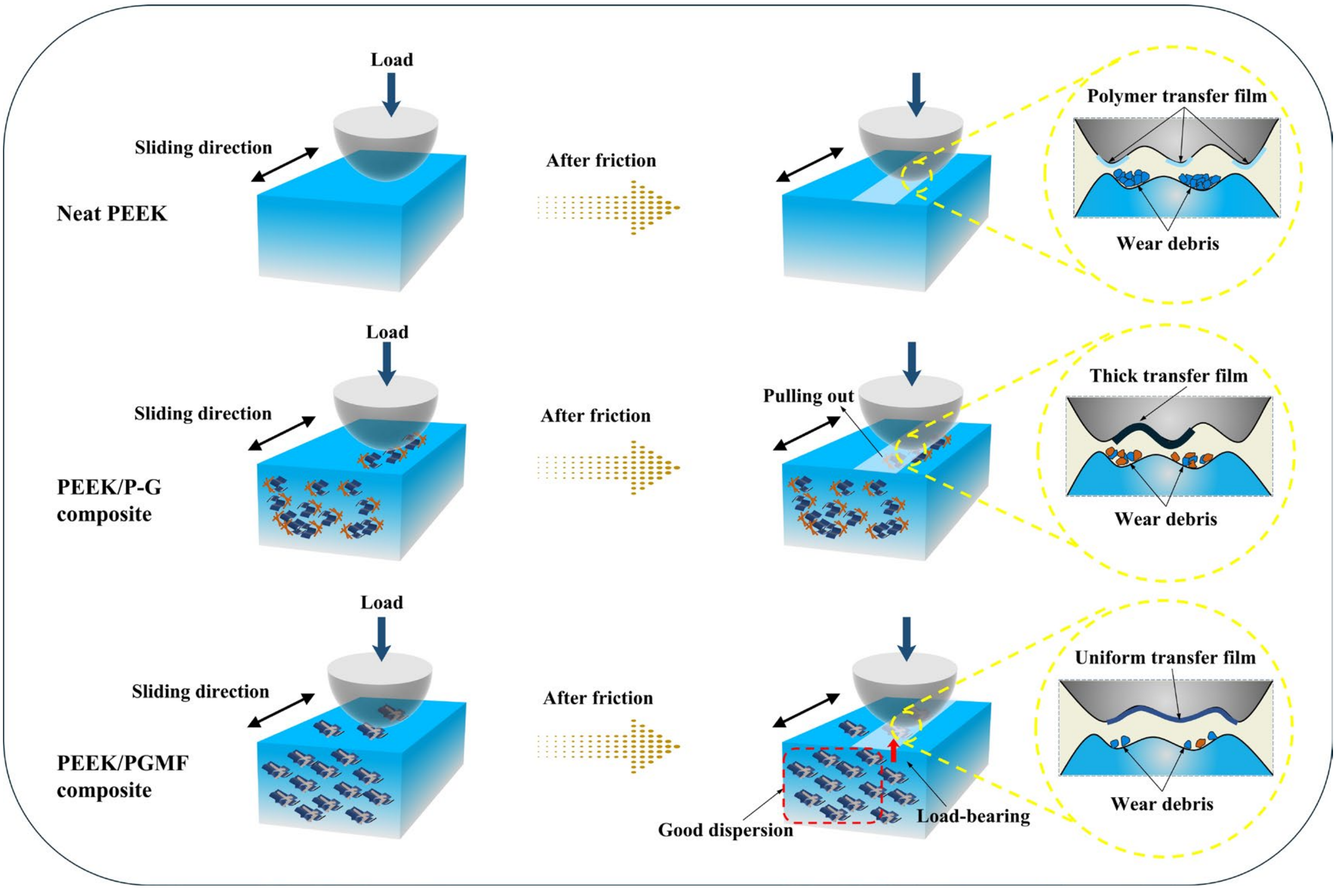

Figure 6. The schematic illustration of the wear mechanism for the neat PEEK and PEEK composites.

4. Conclusions

In the present work, four types of fillers were successfully fabricated and introduced into the PEEK composite. By combining the one-dimensional (1D) potassium titanate whiskers and two-dimensional (2D) graphene nanosheets, the dispersion of fillers in the PEEK matrix was significantly improved and the synergistic effect was successfully achieved. It can be found that compared to direct mixing, PTW–graphene multi-dimensional compound fillers significantly enhanced the wear resistance of PEEK. In addition, the wear rate of PEEK/PGMF2–10% composite was 3.214×10^{-6} $mm^3N^{-1}m^{-1}$, approximately 60% lower than that of neat PEEK. The main wear mechanism observed in the PEEK/PGMF composites was fatigue wear. Benefitting from considerable interface binding force and the multi-dimensional structure of the filler, it can effectively stabilize the friction coefficient and improve resistance wear. In addition, the optimized whisker/graphene volume ratio was 1:3. Therefore, this potassium titanate whisker/graphene multi-dimensional hybrid filler is expected to be a promising additive and to fabricate excellent wear resistance PEEK composite.

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Data Availability Statement: The raw data supporting the conclusions of this article will be made available by the authors on request.

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