

Article

Tribological Properties of PEEK and Its Composite Material under Oil Lubrication

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Abstract: PEEK (Poly Ether Ether Ketone) is a high-performance thermoplastic polymer with excellent mechanical, thermal and chemical stability. PEEK has good performance, and is widely used in hydraulic motors. However, there are few studies on the friction and wear properties of materials under the condition of oil lubrication with wide application. The modification of PEEK and the expansion of its application have become a hot research topic in the industry. This study focuses on the modification of the design of PEEK and explores the friction and wear characteristics of self-lubricating materials under different modification schemes. Friction and wear samples were prepared using PEEK-modification pelletizing and injection-molding processes, followed by fixed-condition friction and wear tests. The tribological mechanisms and wear properties of the materials under different modification schemes were analyzed, leading to the identification of several sets of improved reinforced materials. Experimental results demonstrate that modified materials can enhance surface tribological performance, with the best modification effect observed at an SCF filling rate of 15%. The modified PEEK material can better meet the requirements of specific applications, such as high-temperature environments, chemically aggressive environments, or applications requiring high strength and wear resistance.

Keywords: PEEK; friction and wear properties; modified materials; different content; friction reduction



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1. Introduction

PEEK is a widely used high-performance thermoplastic in engineering applications [1–3]. It has excellent mechanical properties and heat resistance, excellent electrical insulation [4], high fatigue strength [5], and stable chemical-corrosion resistance [6]. However, there are several main problems that exist in the practical application of PEEK. Firstly, the surface adhesion of PEEK is poor, leading to weak interface adhesion with fillers and poor thermal conductivity, which can result in thermal expansion, deformation, and fatigue. Secondly, its grinding performance still cannot meet the needs of engineering applications such as support materials, sealing rings, valves, etc. Lastly, PEEK has a melting temperature of 334 °C [7], and high melting viscosity and processing temperature, making its processing technology unstable. With advancements in technology across various domains come higher requirements for material properties. As a result, research on modifying PEEK and expanding its applications has become a hot topic in the industry. Currently available modification methods for PEEK include polymer blending modification; fiber reinforcement modification; whisker modification; inorganic-particle filling modification; and synergistic modification [8].

Carbon fiber (CF)-reinforced thermoplastic polymer composite materials have excellent properties [9,10], including high stiffness and strength, good processability, and low coefficient of thermal expansion. They are widely utilized in the automotive industry, and in biological and medical equipment, as well as other fields [11]. In order to enhance the

tribological performance of PEEK, researchers have incorporated CF into PEEK. Liang et al. [12–14] fabricated PEEK/CF composite materials and investigated their tribological properties in a seawater environment. The experiments were conducted using a friction and abrasion tester with an experimental load of 200 N at five different sliding speeds. The results revealed that pits on the surface of the samples generated hydrodynamic effects and trapped wear debris during the friction and abrasion tests, with the friction process primarily controlled by plowing mechanisms. The coefficient-of-friction range for PEEK composite material with 30% CF in the friction and abrasion tests was found to be 0.03 to 0.07, representing a decrease of 25–33% compared to unmodified PEEK. Glass fiber (GF), renowned for its unparalleled rigidity, modulus, and significant bearing capacity, finds its prime application as a reinforcement agent in polymer-based composites. In a recent study conducted by Li et al. [15], the frictional and abrasive characteristics of PEEK composite material bolstered by GF were meticulously examined under dry friction conditions and water lubrication. The study revealed a gradual uptrend in the friction coefficient and abrasion rate of both PEEK and PEEK/GF composites as the load intensity increased, culminating in a stable state. Specifically, when GF accounted for 30% of the composite under water lubrication, the friction coefficient and wear rate of PEEK/GF registered at 0.11 and $5 \times 10^{-5} \text{ mm}^3/(\text{N} \cdot \text{m})$, respectively. Notably, these values represent a reduction of 0.66% and 51% compared to their unmodified PEEK counterpart, highlighting the remarkable benefits imparted by GF reinforcement. Teng et al. [16] prepared and studied the properties of polytetrafluoroethylene (PTFE)/GF-reinforced PEEK composite material, in which the GF content was 1% and 5%, respectively [17,18]. The results revealed that the mechanical properties of the composite material can be significantly enhanced by adding GF [19]. The coefficient-of-friction and wear rate of PEEK composite material with 1%GF and 10%PTFE were 0.31 and $4 \times 10^{-6} \text{ mm}^3/(\text{N} \cdot \text{m})$, respectively, compared with pure PEEK, which decreased by 1.13% and 31% [20]. Although the addition of GF and CF can improve the mechanical properties of PEEK, the improvement in the interface compatibility between the fiber and the PEEK matrix needs to be further investigated in the future [21,22].

Wear-resistant particles usually have the characteristics of high hardness and high brittleness, such as ceramic particles such as ZrO_2 and SiO_2 . The mechanical and tribological properties of PEEK can be improved by filling metal and its oxides. Demirc et al. [23] researched the influence of the addition of ZrO_2 particles on the wettability and tribological behavior of PEEK composite coatings. The experiment was carried out under the condition of 25% new-born calf serum (NCS) lubrication. The results revealed that the friction coefficient of PEEK composite material coating filled with 5% ZrO_2 nanoparticles is the lowest, which is about 0.12, compared with pure PEEK, which decreased by 49% [24]. Compared with pure PEEK, the friction mechanism of PEEK zirconia composite coating is adhesive wear and mild-abrasive wear. The basic principle of blending is the similar miscibility principle, so the solubility value and surface tension of the blended materials must be similar [25,26]. The composite material prepared by blending PEEK with other polymer materials can have the comprehensive properties of the blend materials. Koike et al. [27] studied the wear mechanism of PEEK/PTFE composite material in rolling contact wear tests. The results revealed that, compared with pure PEEK materials, the abrasion rate of PEEK/PTFE composite material with 25% PTFE is 1/10 of the former. Potassium Titanate Whisker (PTW) as a reinforcement phase can improve the mechanical mechanism of the material [28–30].

Generally, research experts and scholars have carried out numerous valuable studies on PEEK and its polymers under dry-friction and water-lubrication circumstances. Even though research experts and scholars have conducted extensive research on the friction and wear characteristics of modified PEEK, oil lubrication is typically employed in the operating conditions of gear transmission, engines, and hydraulic transmission. There are scarce studies on the friction and wear properties of materials under the condition of oil lubrication, which has wide application. This paper mainly studies the friction and wear properties and wear mechanism of PEEK under the condition of oil lubrication with the

addition of various kinds of modified materials. And it compares the performance of PEEK modified by different materials.

2. Materials and Methods

2.1. Preparation of Raw Materials and Samples

It is indispensable to undertake the modification of pure PEEK since the conventional pure PEEK fails to fulfill the performance requirements of wear reduction and resistance, excellent self-lubricity, high strength, and favorable thermal performance, to enhance the physical properties of the self-lubricating layer and meet the demands of the inner-curve hydraulic motor under low-speed and heavy-load conditions. The modified design process is depicted in Figure 1.

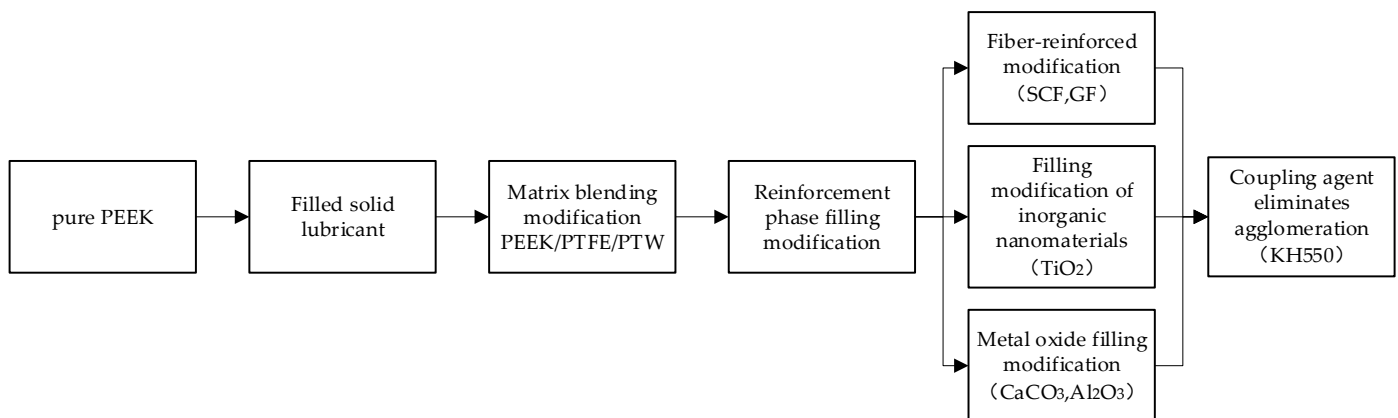


Figure 1. Flow chart of modified-material design.

Based on pure PEEK, it is essential to select an appropriate solid lubricant to enhance the self-lubricating performance of PEEK and facilitate the formation of the transfer film. Currently, among the existing solid lubricants, options such as PTFE, molybdenum disulfide (MoS_2), and graphite are available. Nevertheless, the lubrication performance of PTFE is the most superior, and thus PTFE is selected as the solid lubricant for the modified PEEK.

Once the appropriate solid lubricant is determined, the matrix design for PEEK modification should be carried out. Herein, polyphenylene ester (Ekonol) and PTW are selected for blending modification. The blending modification of PTW, PEEK, and PTFE has been well established, with the optimal ratio being 75% PEEK, 10% PTFE, and 15% PTW, for achieving the best overall performance. Hence, in the matrix group, the chosen proportion is PEEK/PTFE/PTW. The focus of the modification research in this group lies in the subsequent reinforcement filling modification.

Following the completion of the matrix composition design, the reinforcement phase design is necessary to enhance the mechanical properties of the modified PEEK, reduce the linear expansion coefficient, and further decrease the coefficient-of-friction and specific-wear rate. Three types of reinforcement materials are selected: fiber materials, inorganic nano-materials, and metal oxide materials. Specifically, short carbon fiber (SCF) and GF are chosen for fiber materials, titanium dioxide (TiO_2) for inorganic nano-materials, and calcium carbonate whisker (CaCO_3) and aluminum oxide (Al_2O_3) for metal oxides. The ratio of matrix PEEK/PTFE/PTW has been determined, allowing for the commencement of the reinforcement and modification design. The proportion of enhanced phase-modified materials still needs to be confirmed.

The principal advantages of fiber materials reside in their high strength and high stiffness. Nevertheless, an excessive proportion of fibers during filling modification might lead to the material being overly rigid and a decrease in its toughness. This occurs because the increase in fiber content makes the material more brittle, thereby reducing its capacity to resist impact and vibration. Hence, it is necessary to determine a suitable proportion to strike a balance between the strength and toughness of the material. According to the

literature review, when the mass filling percentage of SCF approaches 15%, the mechanical properties (such as micro-hardness, Young's modulus, elastic compression modulus, etc.) of the composite material begin to deteriorate [31]. When the mass filling percentage of GF approximates to 30%, the mechanical properties (such as micro-hardness, Young's modulus, elastic compression modulus, etc.) of the composite material also begin to decline [32]. With the rise in fiber content, the processing performance of the material may deteriorate. The addition of fibers may cause an increase in material viscosity, making the processing more challenging. Additionally, an excessive number of fibers may result in uneven mixing, influencing the quality of the final product. An excessive fiber proportion may lead to poor interface bonding, affecting the overall performance of the material. In this case, the filling ratio of SCF is selected to be 5–20%, and the filling ratio of GF is selected to be 15–35%.

The ultra-high surface area of nanoparticles is one of their most attractive features, as it helps to generate a large number of interface phases in composites, resulting in strong interactions between the low-loaded nano-fillers and the matrix under load. According to the relevant literature, when the mass filling percentage of nano-TiO₂ particles is close to 20%, the mechanical properties of composites (such as micro-hardness, Young's modulus, elastic compression modulus, etc.) begin to decline [33,34]. This is a major issue for all nanocomposites, as nanoparticles tend to form aggregates due to their high surface area. The dispersion state of nanoparticles in the polymer matrix has an important impact on the mechanical properties of composites. The uniform dispersion of nanoparticles is believed to help improve performance. Therefore, due to the strong tendency of nanoparticles to aggregate, the filling ratio of nano-TiO₂ should not be too high during the preparation of composites. In this case, the filling ratio of TiO₂ is selected to be 5–20%.

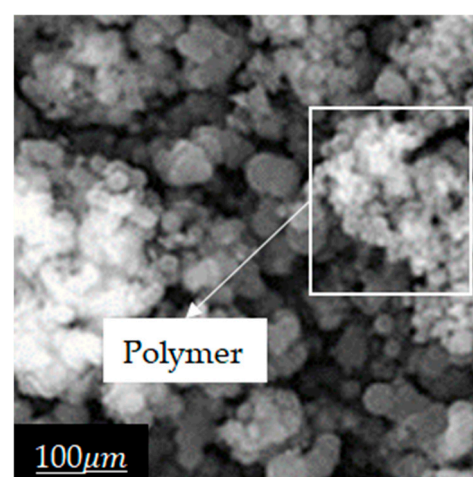
Whisker materials typically possess high strength and stiffness. However, an excessive number of whiskers can render the material overly rigid and diminish its toughness. This might lead to suboptimal performance of the material when exposed to impact or vibration. Thus, it is essential to identify a suitable proportion to strike a balance between the strength and toughness of the material. Based on the literature review, when the mass filling rate of CaCO₃ whisker approaches 20%, the mechanical properties (such as micro-hardness, Young's modulus, elastic compression modulus, etc.) of the composite material begin to deteriorate [35–37]. Likewise, when the mass filling rate of Al₂O₃ whisker approximates to 5%, the mechanical properties of the composite material also begin to decline [38,39]. As the content of whiskers increases, the processing performance of the material may deteriorate. The addition of whiskers may cause an increase in material viscosity, making the processing more challenging. Additionally, an excessive amount of whiskers might result in uneven mixing, influencing the quality of the final product. Excessive whiskers could exacerbate the incompatibility, thereby affecting the overall performance of the material. Hence, when filling and modifying whisker materials, it is necessary to comprehensively consider the proportion of whiskers in accordance with specific application requirements and performance objectives, in order to achieve the optimal balance between performance and cost-effectiveness. In this case, the filling ratio of CaCO₃ is selected to be 5–30%, and the filling ratio of Al₂O₃ is selected to be 1–5%.

The specific modification options are outlined in Table 1.

After finalizing the design of PEEK modification, it is essential to incorporate KH550 for the surface modification of the modified material, since various polymers are involved in the modification process. The blending of multiple polymers can cause aggregation, as depicted in Figure 2. Severe aggregation will lead to the deterioration in the friction and wear properties of the modified specimen. Hence, it is necessary to include the coupling agent KH550 in the modification scheme to mitigate polymerization.

Table 1. Design table of reinforcement and modification.

Modified Category	Enhancement Phase/wt%	Matrix Group/wt%
Fiber reinforcement	5% (SCF)	95% (75%PEEK/10%PTFE/15%PTW)
	10% (SCF)	90% (75%PEEK/10%PTFE/15%PTW)
	15% (SCF)	85% (75%PEEK/10%PTFE/15%PTW)
	20% (SCF)	80% (75%PEEK/10%PTFE/15%PTW)
	15% (GF)	85% (75%PEEK/10%PTFE/15%PTW)
	20% (GF)	80% (75%PEEK/10%PTFE/15%PTW)
	25% (GF)	75% (75%PEEK/10%PTFE/15%PTW)
	30% (GF)	70% (75%PEEK/10%PTFE/15%PTW)
	35% (GF)	65% (75%PEEK/10%PTFE/15%PTW)
Inorganic nano-filling	5% (TiO ₂)	95% (75%PEEK/10%PTFE/15%PTW)
	10% (TiO ₂)	90% (75%PEEK/10%PTFE/15%PTW)
	15% (TiO ₂)	85% (75%PEEK/10%PTFE/15%PTW)
	20% (TiO ₂)	80% (75%PEEK/10%PTFE/15%PTW)
Metal oxide filling	5% (CaCO ₃)	95% (75%PEEK/10%PTFE/15%PTW)
	10% (CaCO ₃)	90% (75%PEEK/10%PTFE/15%PTW)
	15% (CaCO ₃)	85% (75%PEEK/10%PTFE/15%PTW)
	20% (CaCO ₃)	80% (75%PEEK/10%PTFE/15%PTW)
	25% (CaCO ₃)	75% (75%PEEK/10%PTFE/15%PTW)
	30% (CaCO ₃)	70% (75%PEEK/10%PTFE/15%PTW)
	1% (Al ₂ O ₃)	99% (75%PEEK/10%PTFE/15%PTW)
	2% (Al ₂ O ₃)	98% (75%PEEK/10%PTFE/15%PTW)
	3% (Al ₂ O ₃)	97% (75%PEEK/10%PTFE/15%PTW)
	4% (Al ₂ O ₃)	96% (75%PEEK/10%PTFE/15%PTW)
	5% (Al ₂ O ₃)	95% (75%PEEK/10%PTFE/15%PTW)

**Figure 2.** Polymer blending.

2.2. Test Method

According to the modified design scheme of PEEK, the friction and abrasion specimens are processed. This is shown in Figure 3a, in which the upper-specimen material is GCr15SiMn and the lower specimen is modified PEEK.

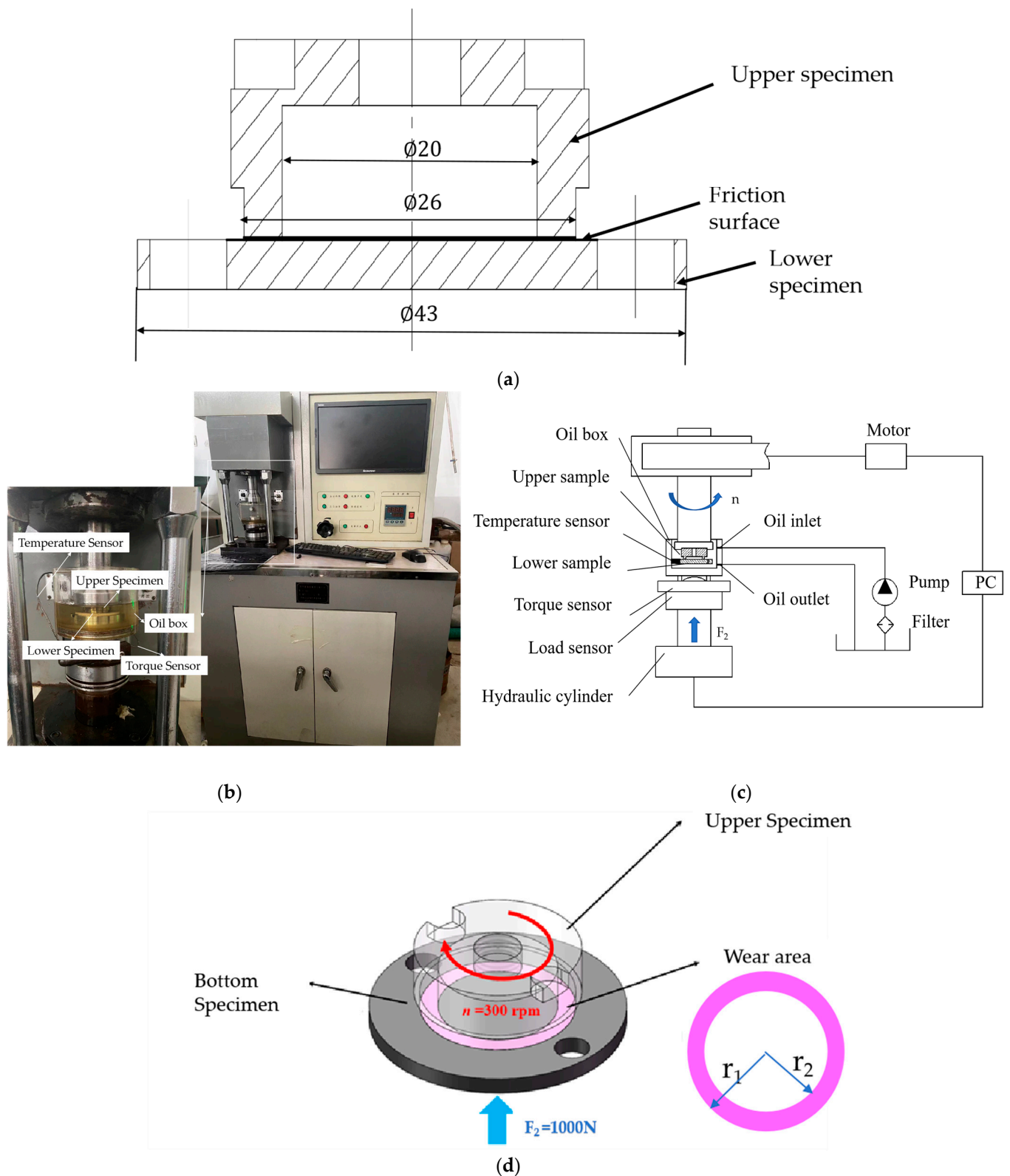


Figure 3. Friction and wear test chart: (a) friction and abrasion test, upper and lower specimens; (b) friction and abrasion tester; (c) test principle of the MMD-5A friction and abrasion tester; (d) friction and abrasion test specimen.

The test was performed on an MMD-5A friction and abrasion tester, as depicted in Figure 3b. The working parameters are presented in Table 2. The friction and abrasion test

is executed by varying the upper- and lower-specimen materials of the testing machine. The upper specimen is fastened to the motor spindle via the upper fixation block and screw. During the working process of the test, the motor drives the upper specimen to rotate, which constitutes the speed provided by the test. The lower specimen is installed on the piston of the hydraulic cylinder through the lower fixing block. The hydraulic cylinder supplies a loading force for the sample. During the test, the upper and lower specimens are immersed in a sealed fuel tank. The oil in the tank is provided by a small 8L oil pump to simulate the actual situation when the motor shell is filled with oil. The principle of the friction and abrasion test is illustrated in Figure 3c.

Table 2. Working parameter table of MMD-5A friction and abrasion tester.

No.	Property	Value
1	Test force range	50–5000 N
2	Test force accuracy	±1%
3	Maximum friction	300 N
4	Friction accuracy	±3%
5	Frictional-torque measurement range	0–15 N·m
6	Spindle motor power	3 kW
7	Spindle motor-speed range	1–300 r/min
8	Time setting	1 s–999 min

The lower specimen ascends under the driving force F of the hydraulic cylinder and comes into contact with the upper specimen, which rotates clockwise, driven by the motor. During this process, the lower specimen experiences a clockwise friction f exerted by the upper specimen, and a force sensor is positioned on the thrust bearing at the outer end of the hydraulic cylinder piston to detect this friction f when the upper die rotates clockwise. Simultaneously, measures are taken to prevent the lower specimen from rotating clockwise under the influence of the upper specimen, ensuring that torque balance is achieved between force sensor F_1 on the lower specimen and friction force f between both specimens. The force on thrust bearing is directly controlled by force F_1 according to feedback from sensors. Therefore, based on principles of torque balance, the coefficient of friction μ between the upper and lower specimens can be calculated by Formula (1)

$$\mu = \frac{2 \times F_1 \times r_3}{F \times (r_1 + r_2)} \quad (1)$$

where r_1 is the outer radius of the friction ring, r_2 is the inner radius of the friction ring, and r_3 is the outer radius of the thrust bearing.

The control variable method was employed for the test, ensuring quantitative and consistent loading force, speed, and test time. The material type and specimen ratio were treated as variables, with friction and wear tests conducted as depicted in Figure 3d. The loading force F was set at 1000 N, the speed n at 300 r/min, and the test duration at 3600 s. The wear morphology of the upper and lower samples was observed using a Scanning Electron Microscope (SEM). The wear depth of the upper and lower specimens were observed using a laser confocal microscope.

3. Results and Discussion

3.1. Tribological Properties of PEEK/SCF Friction Pairs

The coefficient of friction is an important target for considering the friction performance. Figure 4a presents the friction-coefficient curve after adding SCF. The figure demonstrates an increasing tendency in the friction coefficient of the matrix group. The friction-coefficient curves obtained by adding different contents of SCF initially tend to

increase and then decrease. Subsequently, the curve tends to reach a steady state. The friction-coefficient curve of SCF with 10% and 15% content is lower than that of the matrix group. When the SCF content is 15%, the friction coefficient is reduced by 70.4% at most. Regarding the coefficient of friction, these two modified blends are capable of enhancing the abrasion resistance of the specimens. The law of temperature variation of the specimen during the friction and abrasion test is also of great significance for the study of friction performance. Figure 4b presents the temperature curve subsequent to the addition of SCF. It can be discerned from the curve that all the temperature curves of the modified reinforced surface, with the exception of the matrix group, initially tend to ascend and subsequently reach a stable state. When the SCF filling ratio is 15%, the temperature drops significantly, with a decrease of 37.4%.

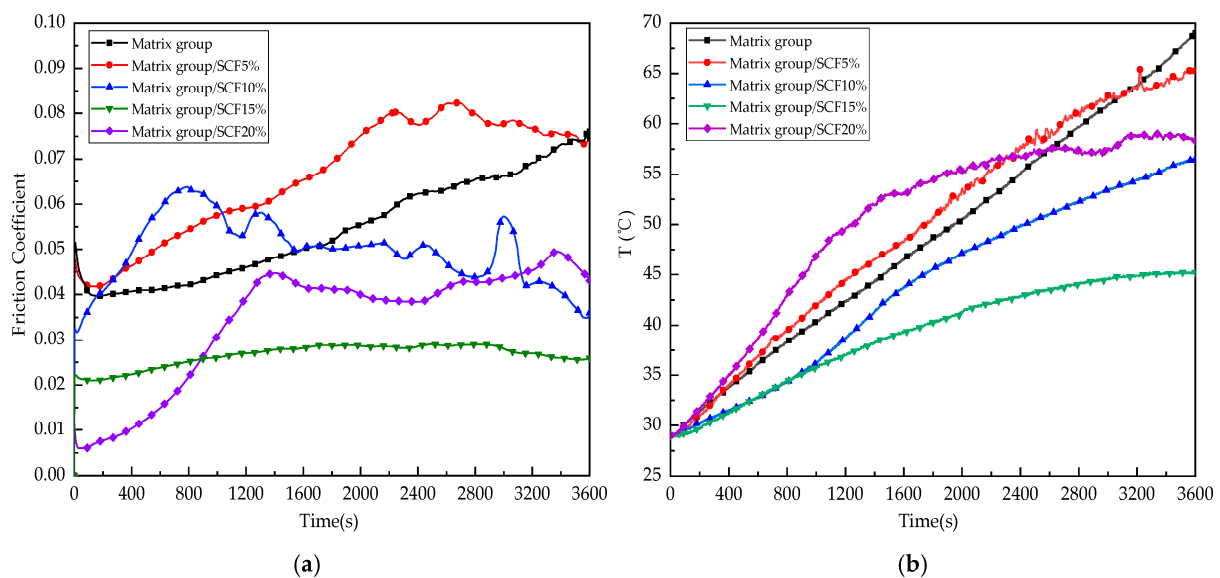


Figure 4. Friction-coefficient and temperature curve of modified reinforced (SCF): (a) friction-coefficient curve; (b) temperature curve.

The height values of the surface concave and convex peaks of the three sections were respectively taken by the laser confocal microscope. The three sections of the composite material are shown in Figure 5.

After taking the average value, the volume wear of each group of materials is calculated according to the Formula (2)

$$V_{wear} = \frac{h_{av} S_{wear}}{F x_{slip}} \quad (2)$$

where V_{wear} is the volume-wear amount of the lower specimen, h_{av} is the average difference of the height values of the surface convex peaks of the three sections, S_{wear} is the wear area of the upper and lower specimens, and x_{slip} is the wear and slip distance of the upper specimens.

$$S_{wear} = \pi(r_2^2 - r_1^2) \quad (3)$$

$$x_{slip} = 60 \times 2\pi r_2 n \quad (4)$$

$$V_{wear} = \frac{\pi h_{av}(r_2^2 - r_1^2)}{60 \times 2\pi r_2 n} \quad (5)$$

The volume wear of the matrix group was calculated to be $2.06 \times 10^{-4} \text{ mm}^3/\text{N} \cdot \text{m}$, and the volume wear of the other modified groups was calculated by the same method. Compared with the matrix group, the wear of the 5% SCF group was increased by 28.6%, and that of the 10% SCF group was increased by 42.8%. The wear of the 15% SCF group

was reduced by 36.4%, and that of the 20% SCF group was increased by 28.4%. Figure 6 shows the line chart of SCF wear of the matrix group and filling with different contents. Considering the wear amount, the wear amount of the SCF test group supplemented with only 15% content is lower than that of the matrix group, which has a positive effect on the wear resistance of the specimen. Therefore, it can be preliminarily judged that the modified enhancement group with 15% SCF content has the best modification effect.

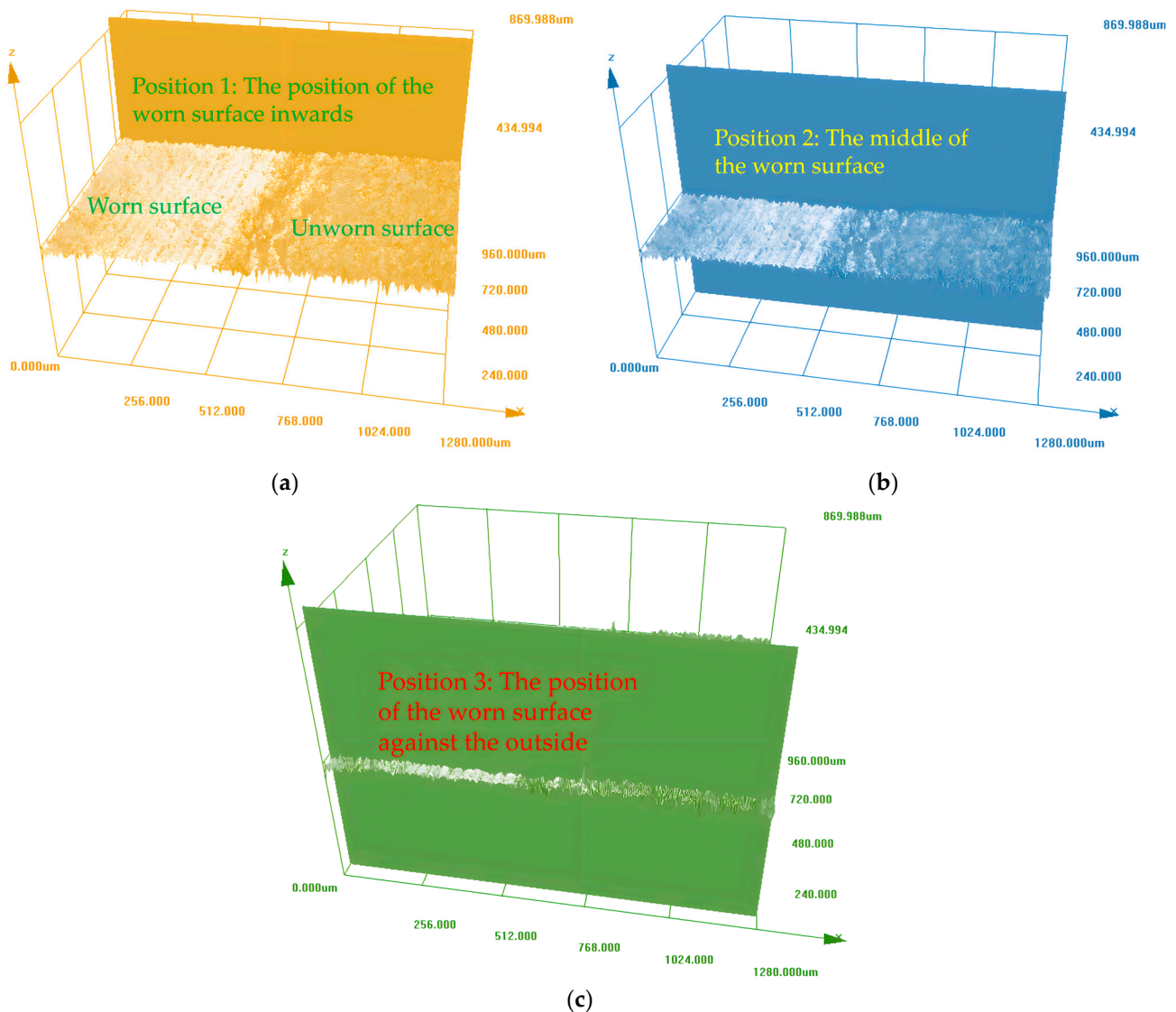


Figure 5. Three selected locations. (a) position 1. (b) position 2. (c) position 3.

The influence of the modified-material surface on friction is a complex mechanism, which is related to the contact characteristics, physical and chemical properties, geometric characteristics, working parameters, and other factors of the friction pair surface. Therefore, the type and content of modified materials will affect the geometric characteristics of the friction pair surface, thereby impacting friction. Figure 7 analyzes the worn surface of modified PEEK filled with 15% SCF. After blending pure PEEK with 10% PTFE and 15% PTW, and filling with 15% SCF, it is found that the worn surface of the lower pattern becomes smoother. Although there are a small number of wide wear marks present, the rest of the wear marks become compact and significantly narrower. At the same time, there is no longer an accumulation of modified PEEK debris around the furrow, in addition to this change in appearance being less serious than before. There are no wide wear marks in a large area because adding 15% SCF improves strength to a certain extent, while effectively

resisting hard particles' influence on lower specimen wear. Furthermore, no modified PEEK falls off onto wear marks; only a few parts fall off, indicating that shedding mainly occurs at initial stages due to strengthened resistance against plastic deformation after adding 15% SCF.

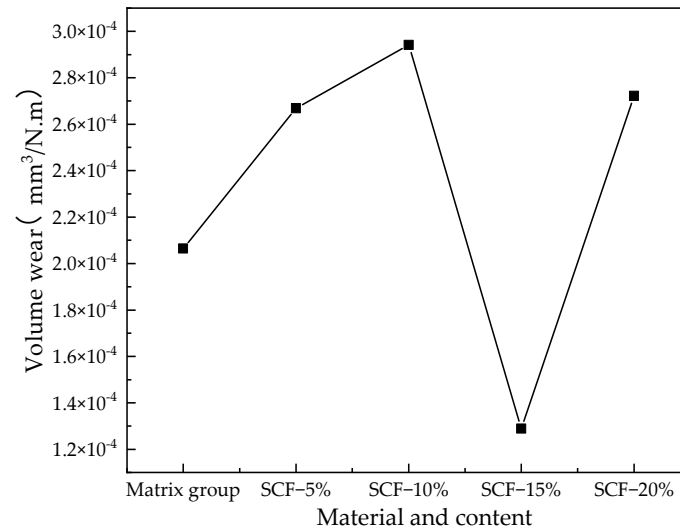


Figure 6. Wear loss of SCF materials with different proportions.

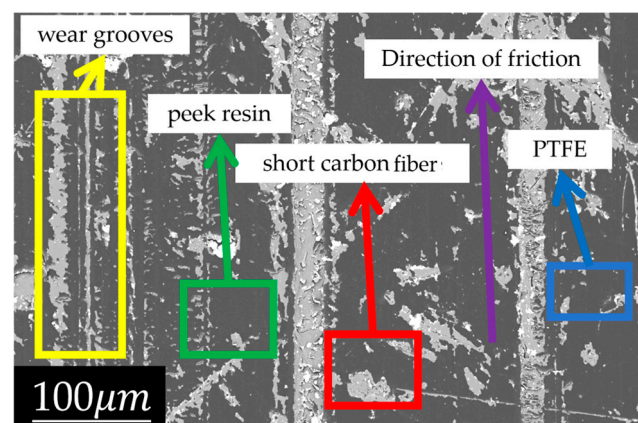


Figure 7. Wear diagram of 15% SCF-reinforced modification.

3.2. Tribological Properties of PEEK/GF Friction Pairs

Figure 8a depicts the friction-coefficient curve following the addition of GF. It is evident from the graph that the friction-coefficient curve for GF with 15% and 20% content eventually stabilizes and exhibits a lower friction coefficient. This suggests that the modified group with these two levels of content has the most favorable effect. When the GF content is 20%, there is a maximum reduction in friction coefficient of 82.4%. Figure 8b illustrates the temperature curve after adding GF. The temperature curve for the experimental group with 15% and 20% content initially increases and then becomes more consistent, whereas the temperature curve for other contents shows a continuous upward trend. In terms of temperature, the test group modified with 15% and 20% content of GF performs better, resulting in a maximum reduction in friction coefficient of 53.7% when the GF content is at 15%.

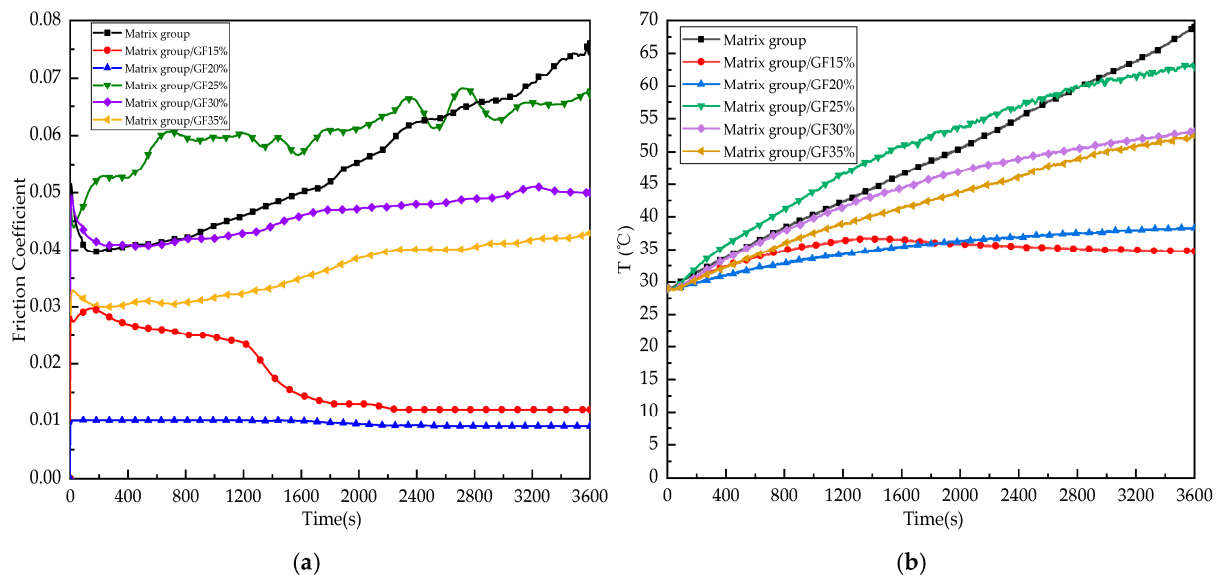


Figure 8. Friction-coefficient and temperature curve of modified reinforcement (GF): (a) friction-coefficient curve, (b) temperature curve.

The same method was used to calculate the volume wear of each group of materials, and, compared with the matrix group, the wear of the group with addition of 15% GF was reduced by 53.6%, the group with addition of 20% GF was reduced by 21.5%, the group with addition of 25% GF was reduced by 35.8%, and the group with addition of 30% GF was reduced by 28.6%. The wear of the 35% GF group was reduced by 10.8%. Figure 9 shows the line chart of GF wear of the matrix group and filling with a different content. Considering the wear amount, the wear amount of the GF test group supplemented with 15% mass fraction is lower than that of the matrix group, and the wear amount is reduced by 53.6% compared with that of the matrix group, which is the best optimization for the wear resistance of the specimen. Therefore, it can be judged that the modified-enhancement group with 15% GF content has the best modification effect.

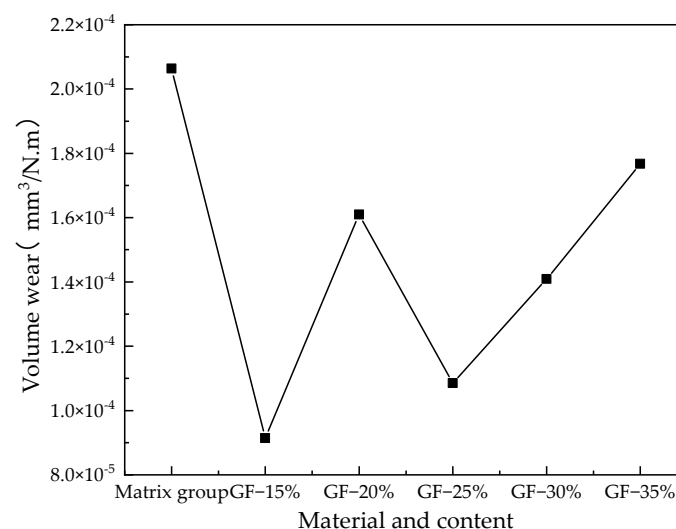


Figure 9. Wear loss of GF materials with different proportions.

Figure 10a,b presents the wear diagram of the modified specimen filled with 15% and 20% GF captured by SEM. Under the circumstance of oil lubrication, the frictional heat is dissipated promptly, due to the cooling effect of oil, and the frictional surface is in a relativistic or even glassy state, thereby significantly reducing the adhesive transfer to the

frictional dual surface of the composite material [40]. Simultaneously, the oil eliminates the wear particles from the friction zone, thereby reducing the wear. The protrusion of fiber fillers is distinctly visible on the wearing surface of GF-modified composites. During the process of friction, when under pressure, the fiber filler initially acts as the pressure-bearing element to prevent the metal friction pair from planing the matrix, and thus the worn surface plowing vanishes and the surface becomes smooth. There is a minor amount of wear debris on the worn surface of the composite material modified by GF, demonstrating abrasive wear and fatigue wear. After friction wear, there are furrows on the surface of the sample, indicating abrasive wear. Based on the comprehensive comparison of the diagrams, the wear of the modified group with 15% GF content is the slightest.

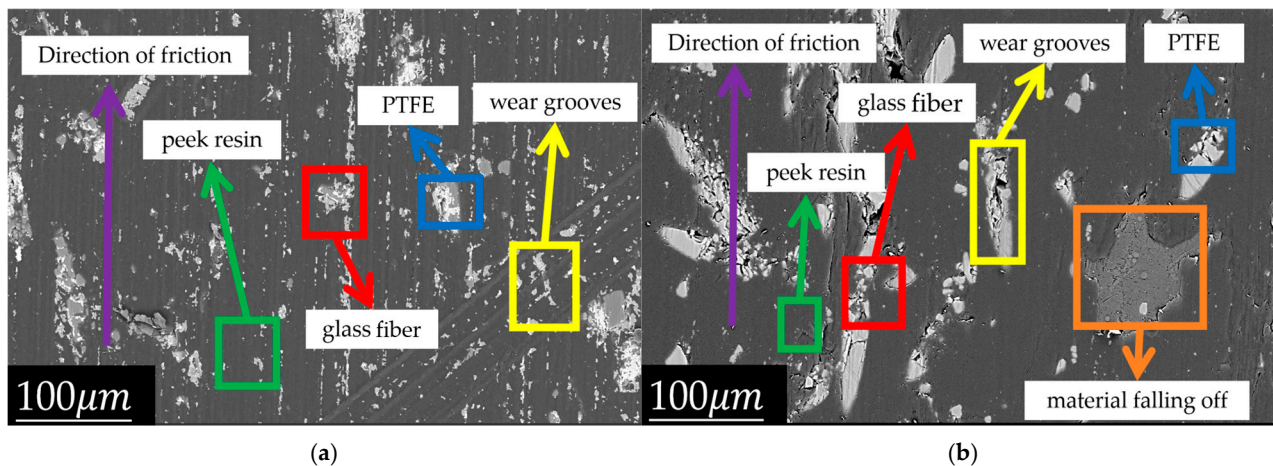


Figure 10. Wear diagram of 15% and 20% GF-reinforced modification. (a) mass fraction 15% GF. (b) mass fraction 20% GF.

The fiber filler is conspicuously visible on the abrasion surface of the glass fiber-reinforced composite material [41,42]. During the process of friction, when under pressure, the fiber filler initially plays the role of withstanding pressure to prevent the metal friction pair from planing the matrix [43,44]. Consequently, the grooves on the worn surface are eliminated, resulting in a smooth surface. The glass fiber-filled modified composites show a negligible amount of abrasive dust on their wear surface, giving rise to manifestations of abrasive wear and fatigue wear.

3.3. Tribological Properties of PEEK/TiO₂ Friction Pairs

Figure 11a depicts the friction-coefficient curve after the addition of TiO₂. The curve indicates that the friction coefficient of the composite material filled with TiO₂ is lower than that of the matrix. Specifically, the friction coefficient of the modified group with 5% and 10% content remained stable at 0.01, which was significantly lower than that of the matrix group. This suggests that the modified group with these two content levels has the most favorable effect. Furthermore, when the TiO₂ content is 10%, there is a maximum reduction in friction coefficient of 76.4%. Figure 11b illustrates the temperature curve after adding TiO₂. The temperature curve of the experimental group filled with TiO₂ initially increases and then tends to stabilize, exhibiting a distinct pattern compared to other modified groups. In terms of temperature performance, it is observed that the modified experimental group with 5% and 10% content of TiO₂ performed better. Notably, when the TiO₂ content is at 10%, there is a maximum reduction in friction coefficient of 52.1%.

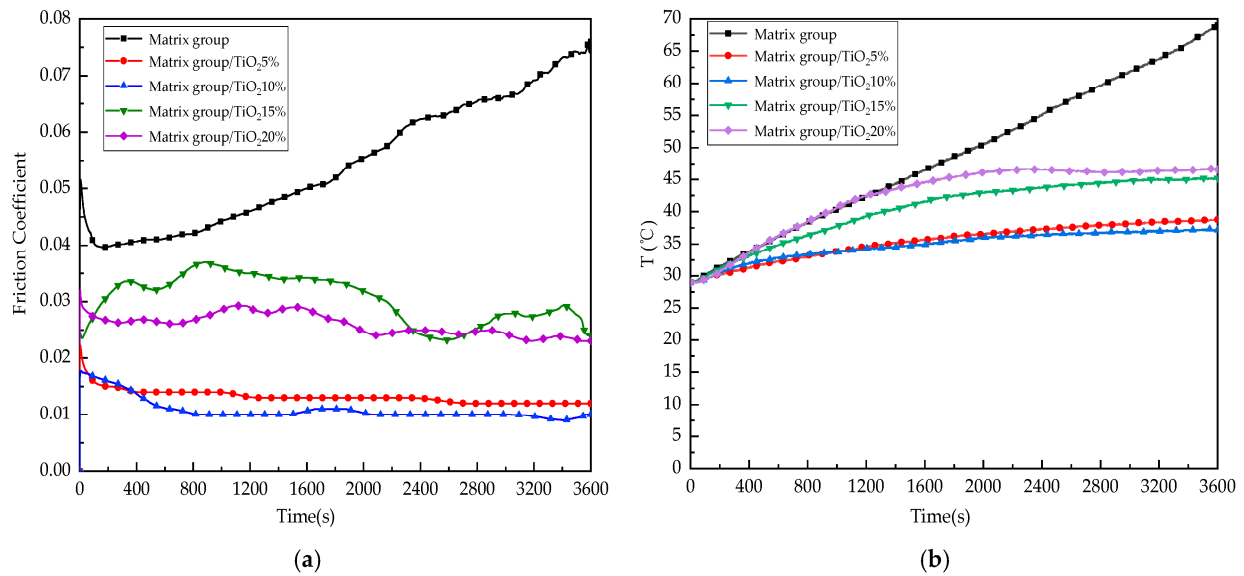


Figure 11. Friction-coefficient and temperature curve of modified reinforcement (TiO₂): (a) friction-coefficient curve, (b) temperature curve.

Compared with the matrix group, the wear loss of the group with added 5% TiO₂ decreased by 51.2%, the wear loss of the group added 10% TiO₂ decreased by 41.9%, the wear loss of the group with added 15% TiO₂ decreased by 21.5%, and the wear loss of the group with added 20% TiO₂ decreased by 32.2%. Figure 12 is a line chart of wear loss of the matrix group and the groups filled with different contents of TiO₂. In terms of wear loss, the experimental group with a mass fraction of 5% TiO₂ added has a lower wear loss than the matrix group, and the wear loss is reduced by 51.2% compared with the matrix group, which is the best optimization for the wear resistance of the specimen. Therefore, quantitative analysis shows that the modified-enhancement group filled with 5% TiO₂ has the best modification effect.

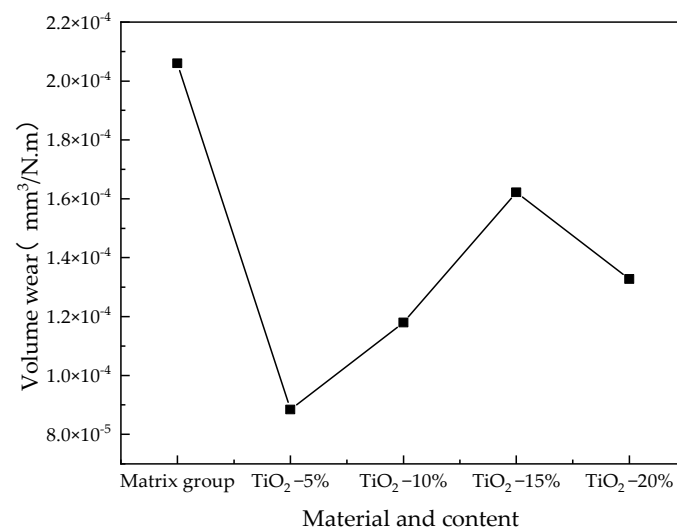


Figure 12. Wear loss of TiO₂ materials with different proportions.

Figure 13a,b present the wear diagram of the specimens modified by filling 5% and 10% TiO₂ and captured by SEM. TiO₂ particles function as “rollers”, reducing the actual friction contact area. Due to the extensive surface agglomeration, particles form large-sized wear debris, which is equivalent to a rough third body, and accelerates the wear of the material surface. The predominant wear mechanism is abrasive wear. It can be discerned from the diagram that there is conspicuous plowing on the surface of the sample after

friction, indicating abrasive wear. Based on the comprehensive comparison of the diagrams, the wear of the modified group filled with 5% TiO_2 was the slightest.

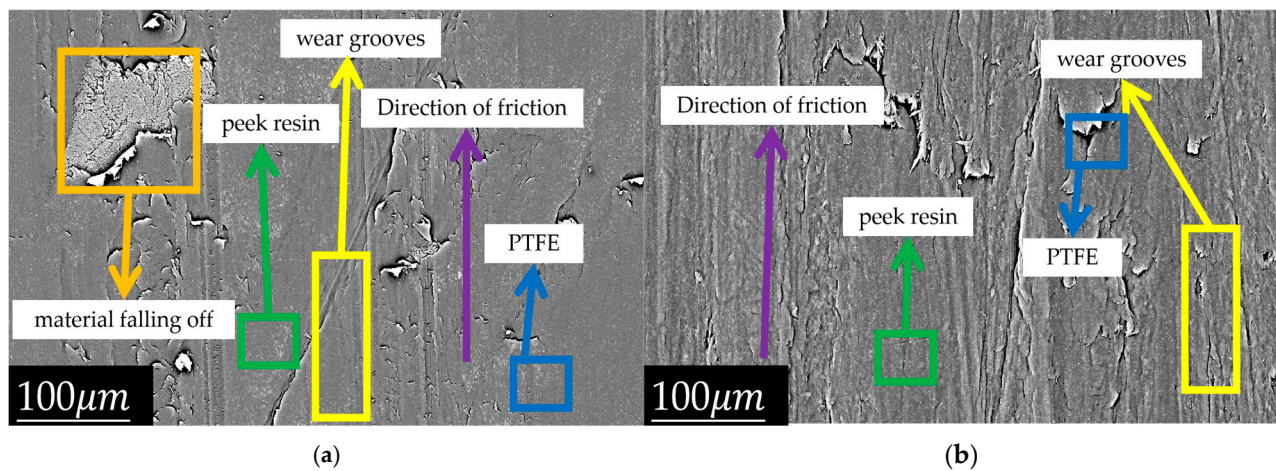


Figure 13. Wear diagram of 5% and 10% TiO_2 reinforced modification: (a) mass fraction 5% TiO_2 , (b) mass fraction 10% TiO_2 .

3.4. Tribological Properties of PEEK/ CaCO_3 Friction Pairs

Figure 14a shows the friction-coefficient curve after adding CaCO_3 . It can be found from the figure that the whisker filled with CaCO_3 can effectively reduce the friction coefficient of the material, and the friction coefficient decreases at first, and finally tends to be stable. With the increase in CaCO_3 whisker content, the friction coefficient of PEEK composite material decreased continuously. When the content of the PEEK whisker exceeded 10%, the friction coefficient increased slowly, and began to decrease again after reaching 25% content. When the CaCO_3 content is 25%, the friction coefficient is reduced by 80.4% at most. Figure 13b shows the temperature-change curve after adding the CaCO_3 whisker. As depicted in the graph, the surface temperature of each sample increases as the test progresses. The matrix group exhibits the highest rate of temperature rise, which then decelerates in the later stages of the test. The addition of CaCO_3 whisker reduces the heating rate, and a lower friction coefficient corresponds to a smaller heating rate. When the CaCO_3 filling ratio is 25%, the temperature drops significantly, with a decrease of 50.6%.

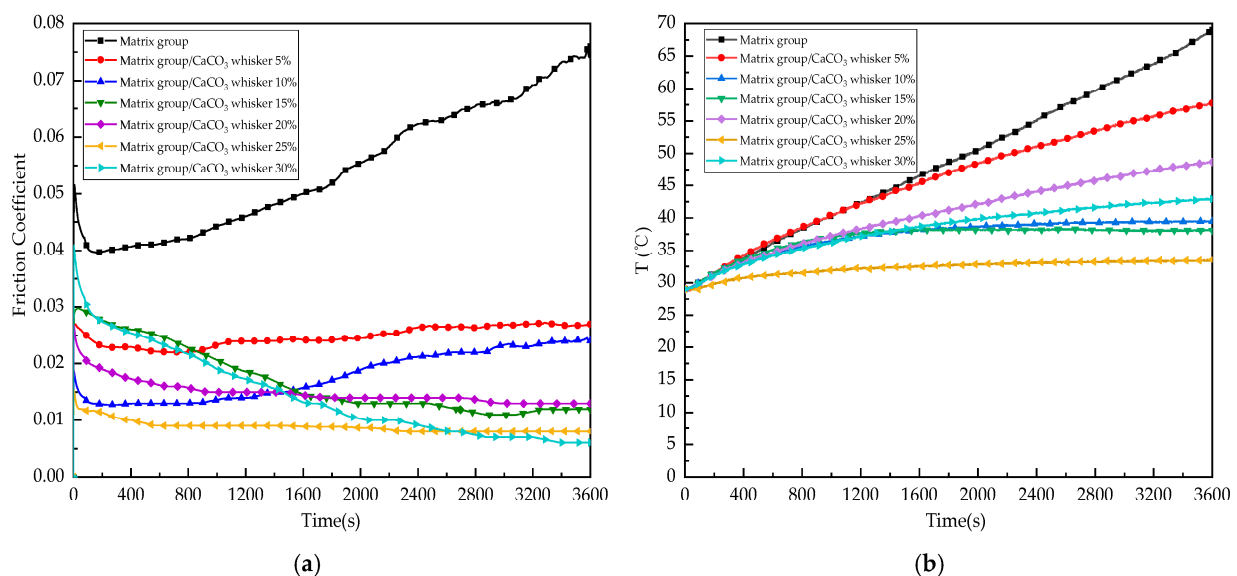


Figure 14. Friction-coefficient and temperature curve of modified reinforcement (CaCO_3): (a) friction-coefficient curve, (b) temperature curve.

The volume wear rate of the modified material with added CaCO_3 was calculated using the same method. The following conclusions were drawn: the wear rate increased by 49.1% in the 5% CaCO_3 group, decreased by 35.6% in the 10% CaCO_3 group, decreased by 49.3% in the 15% CaCO_3 group, decreased by 39.9% in the 20% CaCO_3 group, decreased by 42.9% in the 25% CaCO_3 group, and decreased by 7.2% in the 30% CaCO_3 group. The line graph is shown in Figure 15. As can be seen from Figure 15, except for the wear loss of composite material with 5% CaCO_3 whisker content, which increased slightly, the wear loss of other composite materials with different contents decreased significantly, and the wear loss showed a trend of fluctuation with the increase of content.

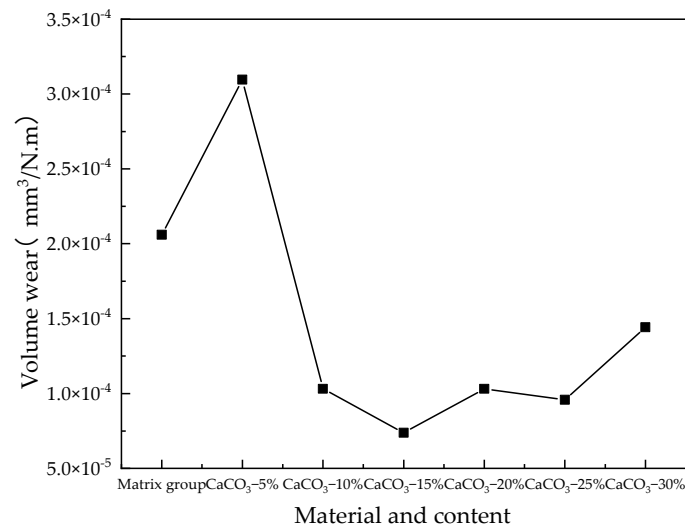


Figure 15. Wear loss of CaCO_3 materials with different proportions.

The diagram of the composite material with CaCO_3 whiskers was photographed under the SEM, as shown in Figure 16. It can be observed from the figure that the surface wear varies with the addition of different CaCO_3 whisker content, and the wear is mainly abrasive wear; some is more serious, some are slight scratches on the surface, and there is some serious wear and tear, such as extrusion spalling.

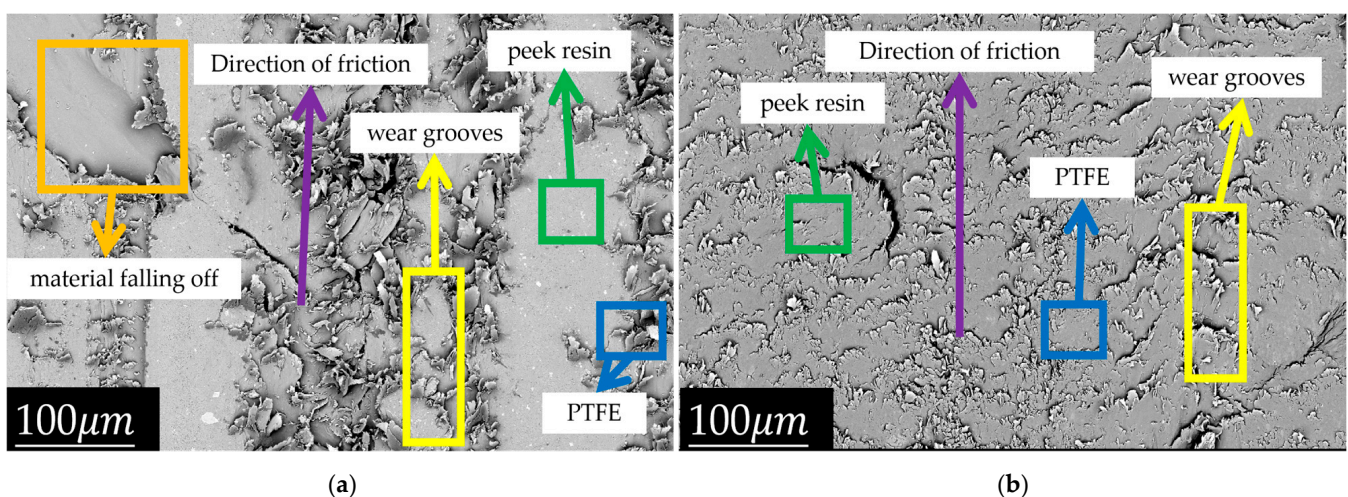


Figure 16. Cont.

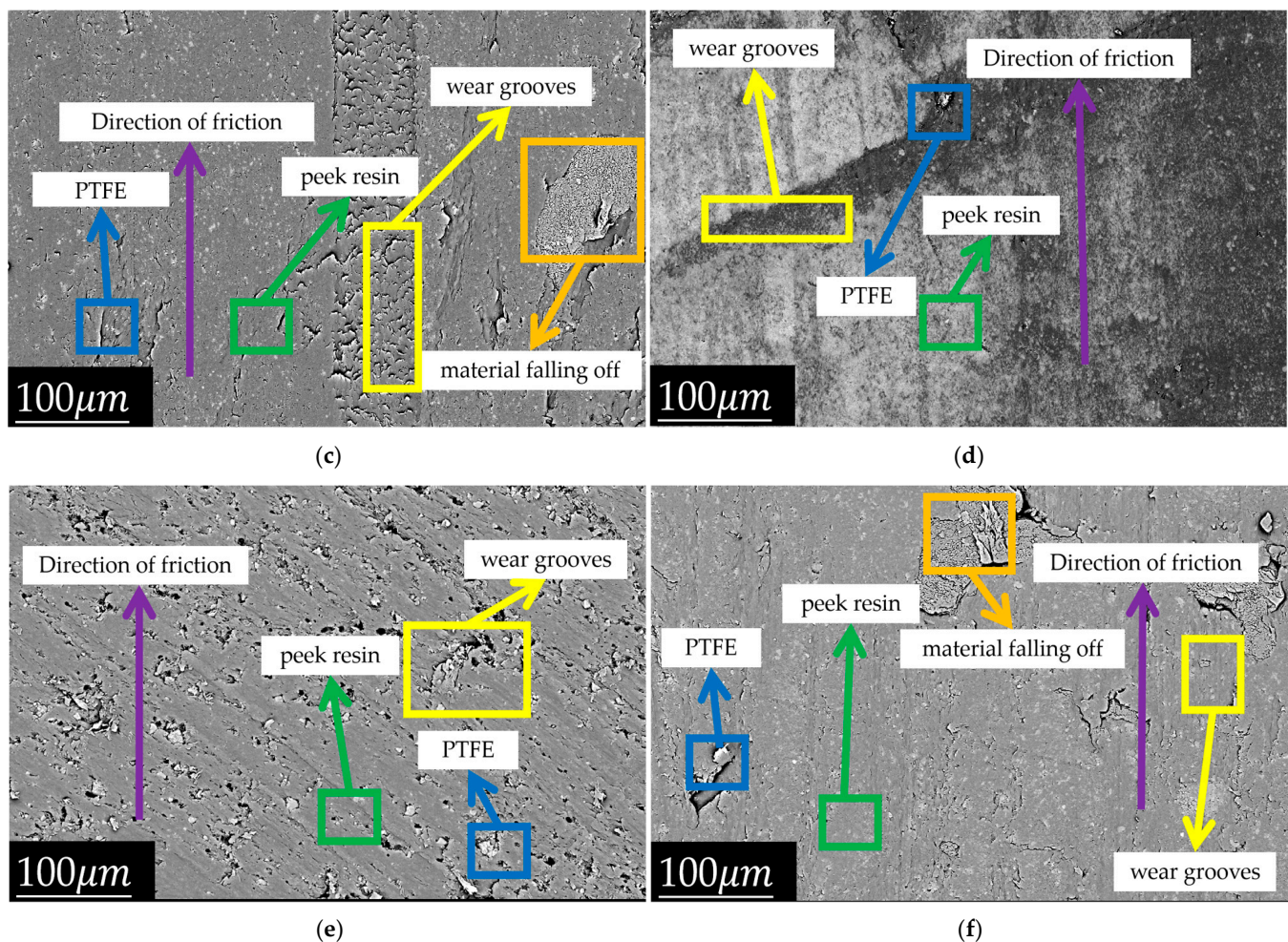


Figure 16. Wear diagram of CaCO_3 -reinforced modification: (a) mass fraction 5% CaCO_3 , (b) mass fraction 10% CaCO_3 , (c) mass fraction 15% CaCO_3 , (d) mass fraction 20% CaCO_3 , (e) mass fraction 25% CaCO_3 , (f) mass fraction 30% CaCO_3 .

3.5. Tribological Properties of PEEK/ Al_2O_3 Friction Pairs

Figure 17a illustrates the friction-coefficient curve following the addition of nano- Al_2O_3 particles. It is evident from the graph that incorporating Al_2O_3 particles effectively reduces the friction coefficient of the material. Only the composite filled with 2% Al_2O_3 particle content shows an upward trend in friction coefficient, while other contents tend to stabilize. The friction coefficient of PEEK composite material fluctuates with increasing or decreasing Al_2O_3 particle content. At the 5% Al_2O_3 content, the maximum reduction in friction coefficient reaches 82.1%. Figure 17b displays the temperature-change curve after adding Al_2O_3 particles. As observed from the figure, surface temperatures rise as the experiment progresses, with a slower rate of increase in later stages of testing. The matrix group exhibits the fastest temperature rise, indicating that adding Al_2O_3 particles reduces heating rates and that lower friction coefficients result in smaller heating rates. When the filling ratio is at 5%, there is a significant decrease in temperature of 43.7%.

Using the same method to calculate the volume wear loss of each group of materials and compare them with the matrix group, the wear loss of the group with 1% Al_2O_3 added decreased by 39.4%, the wear loss of the group with 2% Al_2O_3 added increased by 89.1%, the wear loss of the group with 3% Al_2O_3 added increased by 31.7%, the wear loss of the group with 4% Al_2O_3 added increased by 74.2%, and the wear loss of the group with 5% Al_2O_3 added decreased by 21.1%. The line chart is shown in Figure 17. As can be seen from Figure 18, the addition of nano- Al_2O_3 does not significantly reduce the wear loss of

composites. With the increase in Al_2O_3 particle size, the wear loss of composites shows a large fluctuation, and the change rule is not obvious.

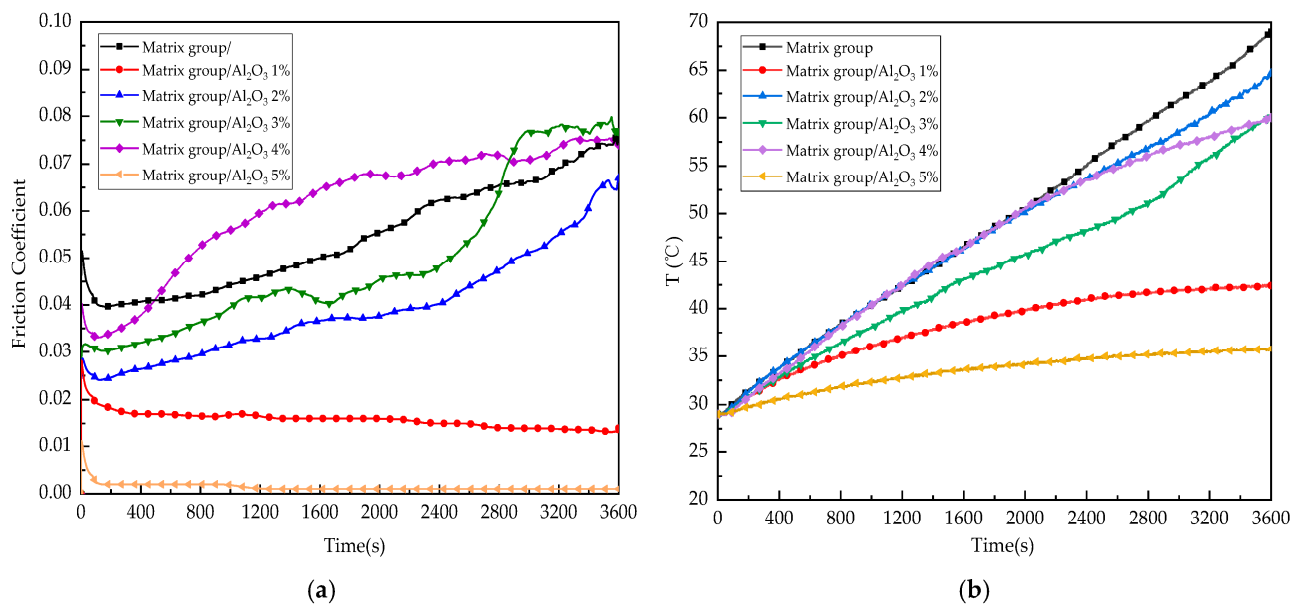


Figure 17. Friction-coefficient and temperature curve of modified reinforced (Al_2O_3): (a) friction-coefficient curve, (b) temperature curve.

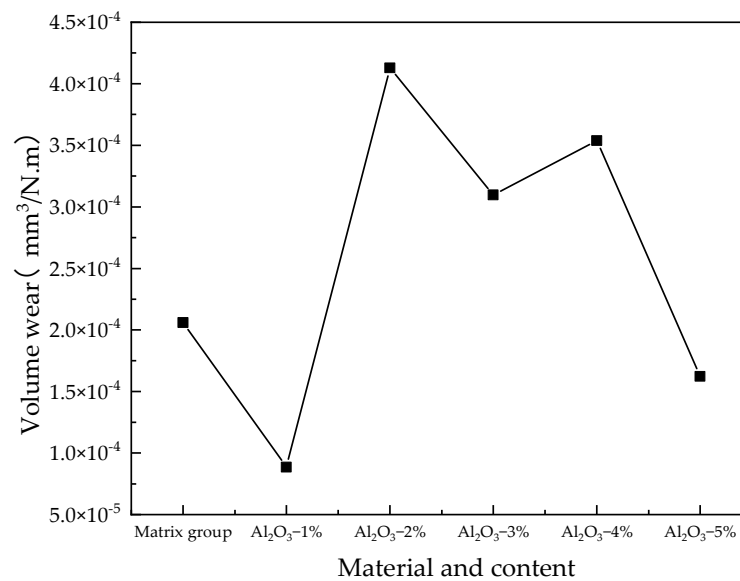


Figure 18. Wear loss of Al_2O_3 materials with different proportions.

The wear morphology of the composite material with Al_2O_3 addition is shown in Figure 19. It can be found that the surface wear is quite different with the addition of different Al_2O_3 particle-size content, and the wear is mainly abrasive wear.

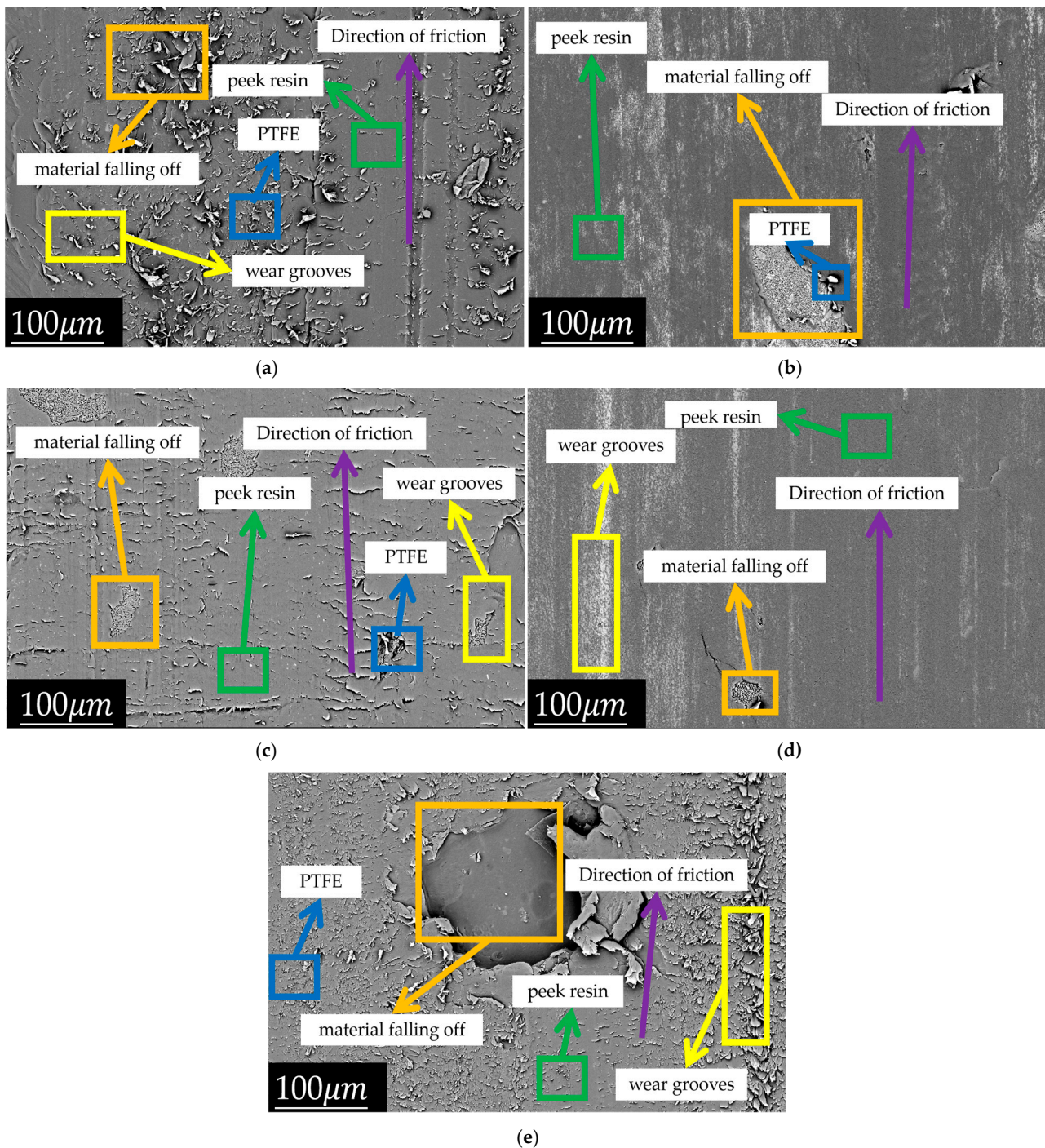


Figure 19. Wear diagram of Al_2O_3 -reinforced modification: (a) mass fraction 1% Al_2O_3 . (b) mass fraction 2% Al_2O_3 . (c) mass fraction 3% Al_2O_3 . (d) mass fraction 4% Al_2O_3 . (e) mass fraction 5% Al_2O_3 .

3.6. Comprehensive Comparison and Discussion

Figure 20a depicts the friction-coefficient curve subsequent to the addition of diverse materials. Figure 20b presents the temperature curve following the incorporation of different materials. Through the analysis of the friction coefficient and temperature-variation diagrams, it is observed that all the friction-coefficient curves of the modified reinforced surface initially tend to increase and subsequently decrease, with the exception of the friction-coefficient curve of the matrix group, which shows a gradual upward trend. Subsequently, the curves tend to reach a stable state. Except for the matrix group, all the temperature curves of the modified reinforced surface initially exhibit an increasing trend,

and then the curves tend to reach a stable state. The image indicates that the friction coefficient of the majority of the modified reinforced materials continuously rises, and fluctuates significantly within approximately 0–1600 s. The temperature continuously ascends. During the period of 1600–3600 s, the friction coefficient tends to stabilize and decline. The temperature curve tends to flatten. This is attributed to the micro-protuberances formed on the surface of the friction pair, which give rise to interface vibrations, resulting in fluctuations of the friction coefficient, an increase in the friction coefficient, and a continuous rise in the friction temperature. After a period of testing, the abrasive debris generated during the sliding process is captured by the modified material. The reduction in debris weakens the shear between the debris and the surface. With the smoothness of the micro-protuberances, a continuous oil film is formed between the friction pairs, and a hydrodynamic pressure effect is produced. Under the influence of hydrodynamic pressure, the contact between the two surfaces is reduced, thereby leading to a decrease in both the friction coefficient and temperature rise.

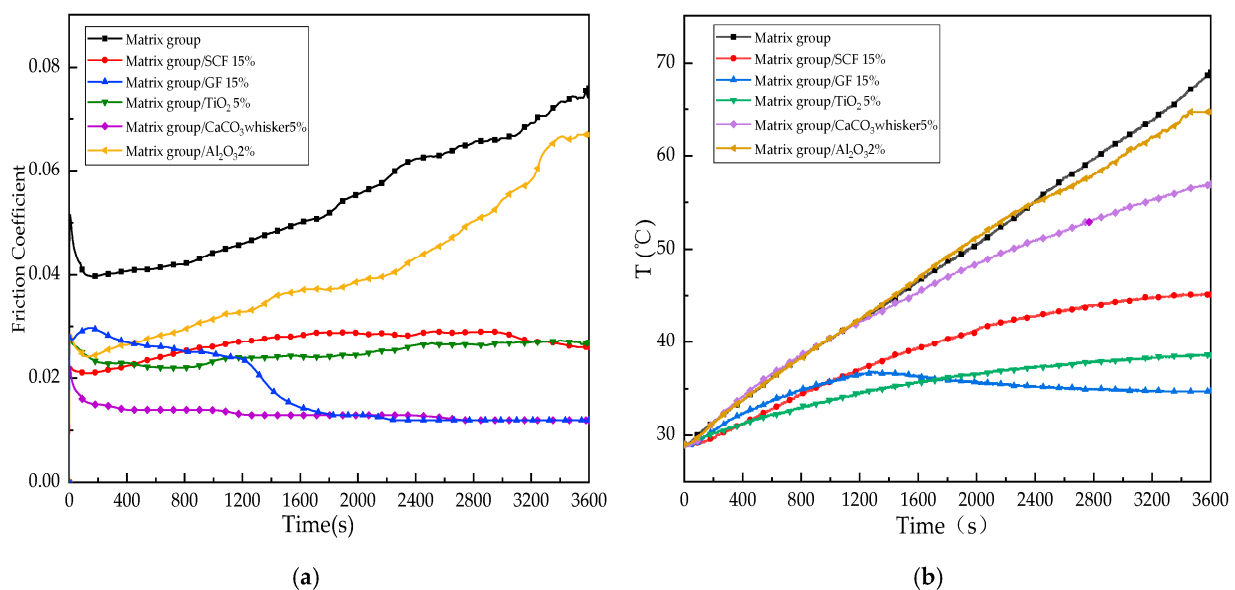


Figure 20. Friction-coefficient and temperature curve of modified reinforced (different materials): (a) friction-coefficient curve, (b) temperature curve.

Once the coefficient of friction and temperature reached the stable stage, the friction coefficient of all modified reinforced materials with varying contents of modified reinforcing materials decreased to different extents, compared with that of non-modified reinforced materials. This implies that the modified materials can enhance the tribological characteristics of the surface. Nevertheless, for instance, when the content of SCF amounts to 15%, the friction coefficient of the SCF-modified material drops significantly, in contrast to that of the unmodified reinforced surface. When the SCF content surpasses 15%, the friction coefficient of the SCF-modified material rises, compared to the reinforced surface when the SCF content is 15%. This is because the increase in roughness induced by the anti-wear effect of the material and the material morphology is counteracted, and a high content of modified materials will amplify the impact of surface roughness and stress concentration of the modified materials, and diminish the tribological properties of the samples. This indicates that a rational content of modified materials can guarantee that the materials can effectively reduce friction.

The original surface of the unmodified material sample shown in Figure 21a is almost worn. Deep scratches and grooves were observed, and severe wear was observed, indicating that mixed wear composed of abrasive wear and adhesive wear occurred during the test. This is because the ability of pure PEEK to resist plastic deformation is poor, and the effect of load and friction heat aggravate the plastic deformation of the lower style, resulting

in pure PEEK falling off and forming wear debris. Due to the formation of abrasive debris in the wear process, a large amount of abrasive debris accumulates on both sides of the furrow, which belongs to the furrow, and there is serious adhesive wear. For unmodified surfaces, it is difficult to trap debris and lubricants. The resulting wear particles will create secondary wear between the surfaces of the friction pair, leading to further wear of the smooth surface.

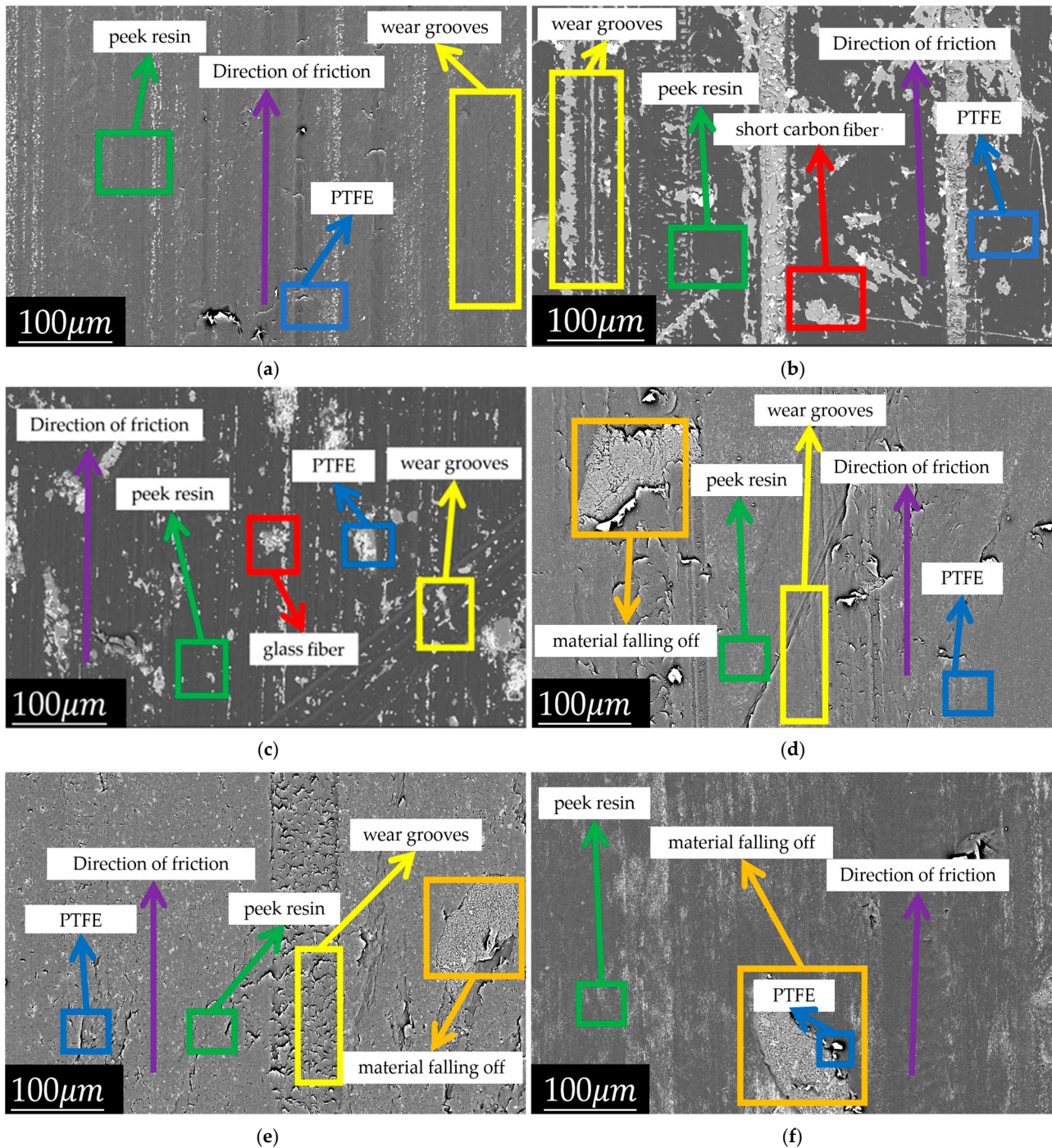


Figure 21. Wear diagram reinforced by different materials: (a) wear profile of unmodified material, (b) mass fraction 15% SCF-modified, (c) mass fraction 15% GF-modified, (d) mass fraction 5% TiO₂-modified, (e) mass fraction 15% CaCO₃-modified, (f) mass fraction 2% Al₂O₃-modified.

As shown in Figure 21b–f, after adding the modified material, it was observed that the wear surface of the subsequent styles became smoother. When the content of added materials is relatively low, it is found that the worn surface of the lower specimen becomes more smooth. Although there are a few wider scars, the rest of the scars become more compact and significantly narrower. Meanwhile, there is no accumulation of modified PEEK debris around the furrow, and the furrow is not very severe. This is because adding a small amount of material enhances the strength of the modified PEEK, to a certain extent, effectively resisting the scratching effect of hard particles on the lower specimen. Therefore, there are no large-area wide scars. At the same time, there is no peeling of modified PEEK on the scars, and only individual parts have peeling. As the filling ratio gradually increases, this phenomenon will gradually decrease, and the wear rate will also decrease, accordingly. When the content of added materials is relatively high, it is found that the furrows on the counter-face disappear, leaving only relatively close and narrow scars. This indicates that increasing the filling ratio can improve the plowing effect of hard particles on modified PEEK, until the mechanism is changed. However, it is found that when the filling ratio is excessively increased, there is a large-area material detachment phenomenon on the counter-face, and the surface becomes rough. This is because as the filling ratio increases, the strength of modified PEEK becomes too high, resulting in a decrease in creep resistance. Therefore, under the continuous action of load, modified PEEK peels off, which aggravates the wear of the counter-face. At the same time, this also explains why when the SCF filling ratio increases from 15% to 20%, both the friction coefficient and wear rate do not continue to decrease, but instead rise, to a certain extent. This relates to relatively severe adhesive wear.

During the polymer friction and wear test, the initial predominant wear mechanism is adhesive wear. As the coefficient of friction increases, the wear temperature gradually rises, and adhesive wear becomes more aggravated. Nevertheless, once the temperature reaches a certain threshold, adhesive wear actually diminishes. At this point, due to the intervention of hard particles in the upper specimen, into the polymer surface, the wear mechanism gradually shifts to abrasive wear. From the SEM images of both unmodified materials and materials with 15% SCF added, it can be observed that, due to the rapid temperature increase during the friction and wear processes, there are numerous hard particles on the surface of unmodified materials. The main wear mechanisms are scouring wear and abrasive wear. However, there are no considerable amounts of hard particles in the image of materials with SCF added, suggesting that the temperature rise has not yet reached a level that mitigates adhesive wear. At this point, it still demonstrates adhesive wear.

Simultaneously, we witnessed the formation of oil films on the surfaces of both the upper and lower specimens, which is associated with the hydrodynamic-pressure lubrication effect. This hydrodynamic-pressure lubrication effect is capable of withstanding external loads and minimizing friction and wear. Hydrodynamic-pressure lubrication is correlated with speed. Currently, our research pertains to the types and contents of modified materials. However, the study regarding the influence of rotational speed on the friction and wear performance of materials is not yet fully developed in this paper. In the future, we will persist in concentrating on this research aspect and continuously enhance the research on the effect of rotational speed on friction and wear performance.

4. Conclusions

The PEEK matrix composite was fabricated through blending modification. Subsequently, solid lubricants PTFE and PTW were incorporated, and, based on this, various modified materials with different contents were added. The friction coefficient and wear rate of the material under oil lubrication conditions were determined through experiments. After the friction test, the friction morphology of the wear area of the composite material was captured, and the tribological properties of the composite material under oil-lubrication conditions were analyzed. The principal findings of this study are presented as follows:

1. The friction-coefficient curves of the matrix group manifested a gradual upward trend, whereas those of the modified reinforced surface initially rose and subsequently declined. The majority of the modified reinforced materials displayed a continuous escalation of the friction coefficient in the early phase of the testing, followed by notable fluctuations and a temperature increase. In the latter stage of the testing, the friction coefficient tended to decrease steadily and the temperature curve flattened.
2. Once entering a stable stage, the friction coefficient of all modified reinforced materials at various modification levels decreased, in contrast to that of unmodified reinforced materials. This indicates that modified materials can improve surface tribological properties.
3. In comparison with unmodified material, the scratches and degree of wear on the modified material are smaller and less severe, suggesting an enhancement in surface tribological properties.
4. The increase in roughness resulting from anti-wear effects is counterbalanced by the material diagram. Nevertheless, a high content of modified material gives rise to an augmented influence on surface roughness and stress concentration, ultimately leading to a reduction in the tribological properties of the sample. Hence, maintaining a reasonable content of the modified material is of paramount importance for effectively reducing friction.
5. It can be inferred that the addition of 15% SCF material yields the optimal modification effect and the most superior friction and wear performance by comparing all the temperature curves, friction-coefficient curves and volume wear rate.

Author Contributions: Conceptualization, X.C.; methodology, X.C.; validation, Y.L. and X.H.; formal analysis, J.Z.; investigation, X.H.; data curation, Z.W.; writing—original draft, Z.W.; supervision, J.Z.; project administration, Y.L. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Abbreviations

f	friction force
μ	friction coefficient
F	driving force
F_1	testing force
r_1	outer radius of the friction ring
r_2	inner radius of the friction ring
r_3	outer radius of the thrust bearing
V_{wear}	the volume wear amount of the lower specimen
h_{av}	the average difference in the height values of the surface convex peaks of the three sections
S_{wear}	the wear area of the upper and lower specimens
x_{slip}	the wear and slip distance of the upper specimens.

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