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Study on Preparation and Tribological Characteristics of CF/PTFE/PEEK Composite

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Abstract: Carbon fiber and polytetrafluoroethylene were used as composite fillers, and CF/PTFE/PEEK composite materials were prepared by vacuum hot-press sintering technology. The Vickers hardness tester and compressive strength tester were used to test the microhardness and compressive properties of the composite; the friction and wear properties were investigated by a multifunctional friction and wear testing machine and scanning electron microscope. The influence of the content of composite filler on the tribological and mechanical properties of polyetheretherketone composites was analyzed, and the mechanism of anti-wear and anti-wear was discussed. The research results show that the hardness and compressive strength of the composite material increase with the increase of CF content, and decrease with the increase of PTFE content. The hardness and compressive strength of 10%CF/PEEK are the highest. They are 30.24HV and 110.4MPa respectively. As the CF content increases, the friction coefficient of the composite material decreases first and then increases. When the CF content is 10%, the friction coefficient of the material is the lowest; as the PTFE content increases, the friction coefficient of the composite material gradually decreases, when the PTFE content reaches 5% ,The friction coefficient remains stable after 10%, and 10%CF/10%PTFE/PEEK has good wear resistance. Considering the combined effect of CF and PTFE, 10%CF/10%PTFE/PEEK composite material has good comprehensive performance.

1. Introduction

As a fully aromatic and semi-crystalline thermoplastic engineering plastic, polyether ether ketone has a series of excellent properties, such as high temperature resistance, corrosion resistance, fatigue resistance, high mechanical strength, excellent fluidity, easy processing, etc., and also has good dimensions Stability, flame retardancy and insulation [1-2]. Wang Huaiyuan [3] and others found that 20% mass fraction of PTFE can play a good lubrication effect on PEEK composite materials, and can effectively reduce the friction coefficient and wear rate of the material. Wang Wancheng [4] showed that PEEK has a lower friction coefficient under water lubrication conditions, which can effectively extend the service life of the material. The results of Chu Tingting [5] showed that PTFE can reduce the friction coefficient of PEEK. When PTFE is added at 5%, the composite material has excellent comprehensive properties. Mana [6] found that the addition of CF can improve the tribological performance of CF/PEEK composites. CF bears the stress between the friction surfaces during the friction process. The experimental results of Qiu Xiaotao [7] showed that when the mass fraction of CF was 10%, the friction coefficient of PEEK/PTFE/CF composites was lower, and when the mass fraction was 20%, the surface wear rate of the composites was lower. Yao Guangdu [8] found that CF can reduce the friction coefficient of PEEK composites and increase the load-bearing capacity of the material, while PTFE micropowders can reduce the load-bearing capacity of the material. Literature [9] experiments prove that adding glass fiber or CF to PEEK resin can greatly improve the rigidity, dimensional stability, impact strength, tensile strength and bending strength of the material. The results of Khedkar J [10] showed that PTFE can improve the tribological properties of PEEK composites, but it will adversely affect the mechanical strength of the composites. Rasheva Z [11] experimentally found that in addition to PTFE, the addition of CF can not only improve the mechanical properties of PEEK, but also make the material have better friction and wear properties. Pei X [12] found that the friction and wear properties of unidirectional long CF reinforced PEEK composites are affected by the shape and size of PEEK particles.

In this paper, the vacuum hot-pressing sintering method is used to prepare the multi-element nano-particle-filled PEEK composite material, and based on the experimental study of the influence of the additive content on the friction and wear properties of the PEEK composite material under dry friction conditions, the composite mechanism of the multi-element nano-particle-filled PEEK composite material is discussed. Improve the comprehensive mechanical properties of PEEK composite materials, expand the scope of its use, provide theoretical basis and experimental basis.

2. Experiment

2.1 Experimental Materials

Polyetheretherketone (PEEK),100P; Carbon fiber (CF), average fineness 0.1mm; Polytetrafluoroethylene (PTFE), particle size 5um. Its main performance parameters are shown in Table 1.

Table 1 Main performance parameters of PEEK, CF and PTFE

	PEEK	CF	PTFE
Elastic Modulus (GPa)	3.55	230	1.3
Density(g/cm^3)	1.32	1.81	2.2
Poisson's ratio	0.3	0.39	0.4
Friction coefficient	0.45	0.4	0.15

2.2 Sample preparation

The ZT-70-20Y vacuum hot-pressing sintering furnace is used to prepare CF/PTFE/PEEK composite materials, and the composition is shown in Table 2. After the mixed powder is ball milled on a planetary ball mill for 4 hours (ball-to-material ratio 10:1), it is kept at 200°C for 6 hours in a drying box, and then cold-pressed with a servo hydraulic press under a pressure of 30 MPa, and the pressure is maintained for 10 minutes; the sample is formed Then put the mold together with the mold into the sintering furnace for vacuum pressure sintering, where the sintering vacuum degree is 10-1Pa, the sintering temperature is 330°C, the holding time is 60min, and the sintering pressure is 10MPa. After the holding is completed, the sample is cooled to room temperature with the furnace. A round sample of φ40mm×10mm was prepared.

Table 2 Distribution ratio of composite materials

Sample	PEEK/(wt.%)	CF/(wt.%)	PTFE/(wt.%)
1	90	0	10
2	85	5	10
3	80	10	10
4	75	15	10
5	90	10	0
6	85	10	5
7	75	10	15
8	100	0	0

2.3 Performance Testing

(1) Mechanical performance test

The theoretical density of the composite material is calculated based on the average effect theory of the composite effect of the material in the literature [13], and the calculation formula is:

$$\frac{1}{X} = \sum_{i=1}^{n} \frac{V_i}{X_i} \tag{1}$$

X is the density of the prepared composite material, g/cm^3; X_i is the density of each component added, g/cm^3; V_i is the volume fraction of each component of the composite material.

In the above formula, the conversion formula of volume fraction and mass fraction is:

$$V_i = \frac{m_i/\rho_i}{\sum_{i=1}^{n}(m_i/\rho_i)} \tag{2}$$

Whereρ_i is the density of each component of the prepared composite material, g/cm^3; m_i is the mass fraction of each component added.

The Wilson 402-MVD Vickers micro-hardness tester is used for hardness testing. Each sample is tested 5 times at 5 different positions and the average value is taken. The KY-D5205 servo hydraulic universal testing machine is used to test the compressive strength of the material.

(2) Friction and wear performance test

Use the RTEC MFT-50 multifunctional friction and wear tester to test the friction and wear performance under room temperature and dry friction conditions. The load is set to 20N, 35N, and 50N for the experiment. The reciprocating frequency of the main shaft of the tester is 2Hz, the stroke is 4.5mm, and the wear time It is 30min, and the material of the grinding ball is GCr15. The samples were ultrasonically cleaned before and after the start of each group of experiments.

The JA2603B electronic balance is used to measure the mass of the sample before and after the wear. The wear rate is calculated according to the empirical formula in literature [14]. The calculation formula is:

$$K = \frac{\Delta m}{\rho F L} \tag{3}$$

Where Δm is the quality difference before and after wear, g; ρ is the density of the composite material, g/cm^3; F is the experimental load, N; L is the relative sliding distance, m.

After the friction and wear experiment was completed, the sample was sprayed with gold, and the S-2500 scanning electron microscope produced by Hitachi was used to observe the surface morphology and section morphology of the sample before and after wear.

3. Results and discussion

3.1 Relative density

The theoretical density, actual density and relative density of the composite material are shown in Table 3.

Table 3 Theoretical density, actual density and relative density of composite materials

Sample	Theoretical density/ (g/cm^3)	Actual density/ (g/cm^3)	Relative density/ (g/cm^3)
1	1.39	1.35	0.971
2	1.41	1.36	0.965
3	1.44	1.39	0.965
4	1.45	1.38	0.952
5	1.38	1.36	0.986
6	1.41	1.36	0.965
7	1.45	1.38	0.952
8	1.32	1.30	0.985

It can be seen from Table 3 that the actual density of the composite material is lower than the theoretical density. This is due to the influence of the sintering process, the composite material will have certain pores during the sintering process; the relative density of the composite material decreases with the increase of the additive content, indicating The compactness of the material decreases with the increase of the additive content.

3.2 Hardness and compressive strength

Figure 1 shows the test results of the hardness and compressive strength of composite materials. It can be seen from Figure 1 that the hardness of the composite material increases with the increase of CF content, and decreases with the increase of PTFE content. The hardness and compressive strength of 10%CF/PEEK are both the highest, respectively 29.8 HV, 110.4MPa. This is related to the properties of the additive itself. CF has a larger elastic modulus and has better rigidity, while PTFE has a lower elastic modulus. It can be seen that CF can enhance the bearing capacity of composite materials, while PTFE can weaken it.

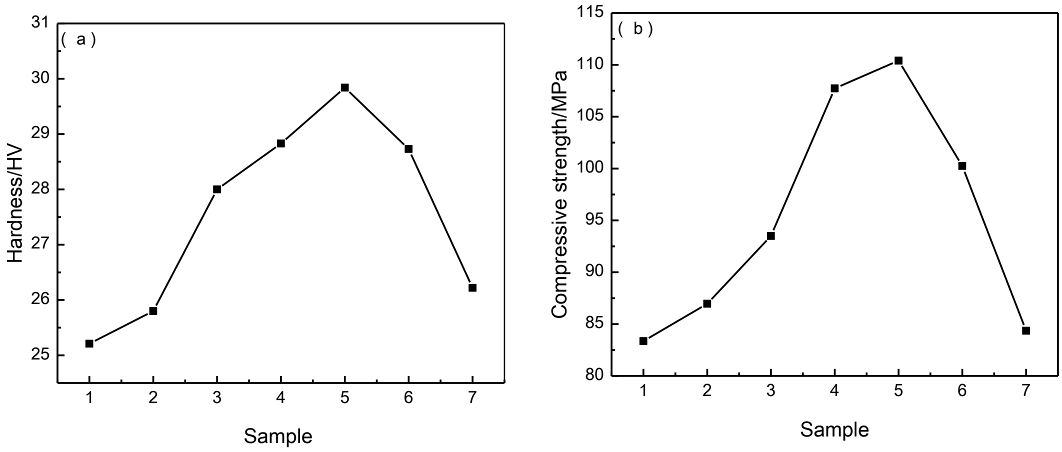


Figure. 1 Hardness (a) and compressive strength (b) of sintered sample

Fig.2 shows the compressed section of pure PEEK and 10%CF/5%PTFE/PEEK. It can be seen from Figure 2(a) that there are particles and cracks in the section of pure PEEK. It can be seen that the additive is well fused with PEEK, no unfused particles are seen, and carbon fibers are exposed on the section. It can be seen that the addition of CF can increase the internal bonding force of the material, and can bear the load, reduce the compressive stress of the PEEK matrix, and improve the compressive strength of the material.

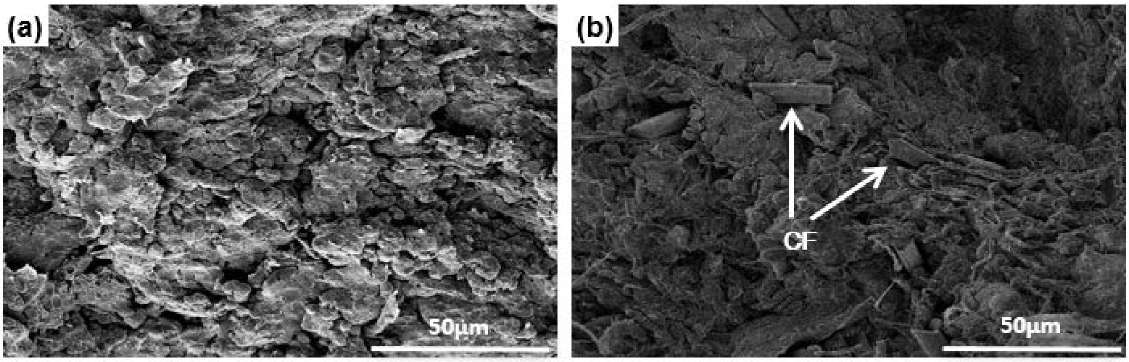


Figure. 2 Micromorphology of fracture surface of pure PEEK (a) and sample 3(b)

3.3 Friction and wear performance

Figure 3 shows the friction coefficient change curve and wear rate bar graph of pure PEEK, 10%PTFE/PEEK, 10%CF/PEEK and 10%CF/5%PTFE/PEEK under a pressure of 20N. Figure 4 shows the ultra-depth microscope friction and wear topography of PEEK, 10%PTFE/PEEK, 10%CF/PEEK and 10%CF/5% PTFE/PEEK. It can be seen from Figure 3 that the friction coefficients of pure PEEK, 10% PTFE/PEEK, 10%CF/PEEK and 10%CF/5%PTFE/PEEK all increase with the increase of the grinding time and gradually become stable. The coefficient of friction has the highest stable value. The friction coefficient of the samples with only PTFE and CF is significantly lower than that of pure PEEK (as shown in curves 2 and 3 in Fig.3), and the decrease is 22% and 25%, respectively, while the friction of the samples with CF/PTFE composite filler The coefficient is the lowest, with a stable value of about 0.1. It can be seen from Figure 4 that the wear scar depth of pure PEEK is deeper (Fig.4(a)), while the sample with CF/PTFE composite filler has the lowest depth of wear scars (Fig.4(c)). This indicates that CF and PTFE can play a synergistic effect on abrasion resistance and improve the abrasion resistance of composite materials.

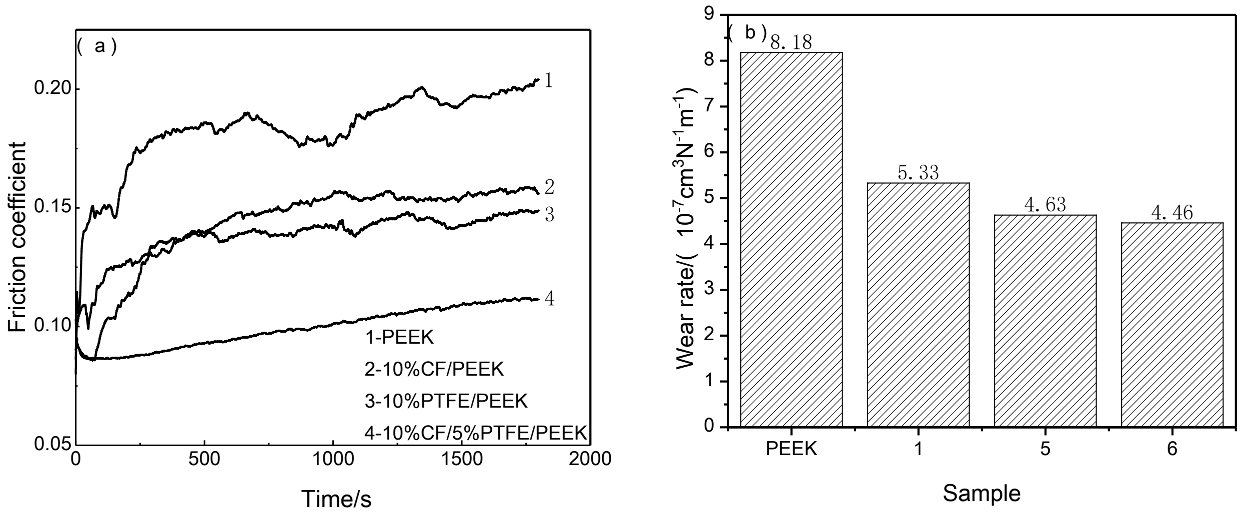


Figure. 3 Pure PEEK and 10%PTFE/PEEK, 10%CF/PEEK, 10%CF/5%PTFE/PEEK friction coefficient (a) curve with time and wear rate (b) histogram (Load: 20N)

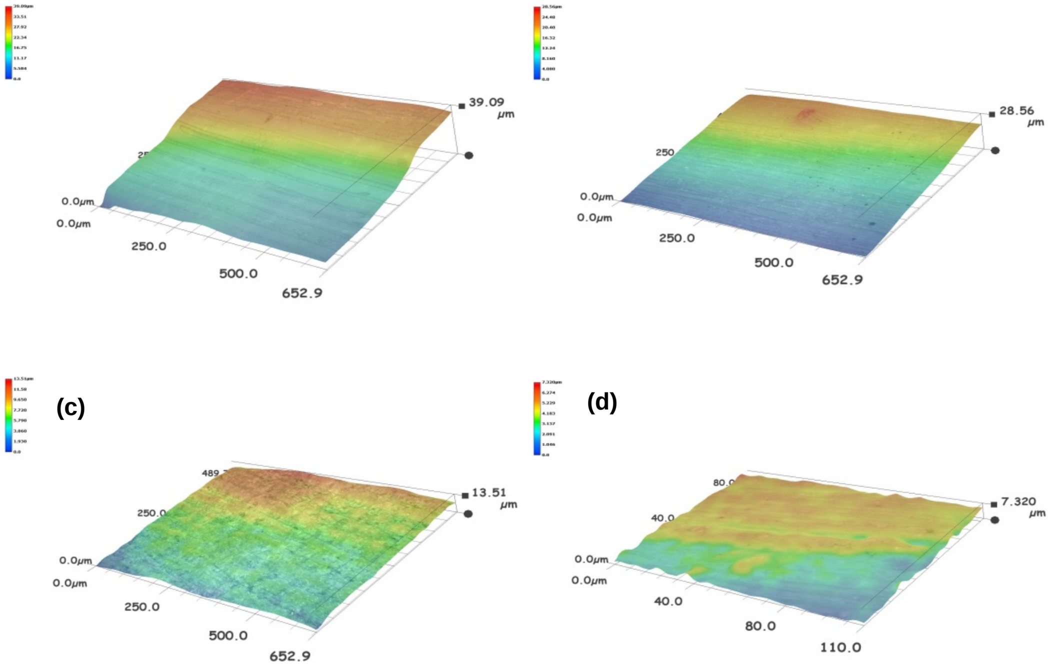
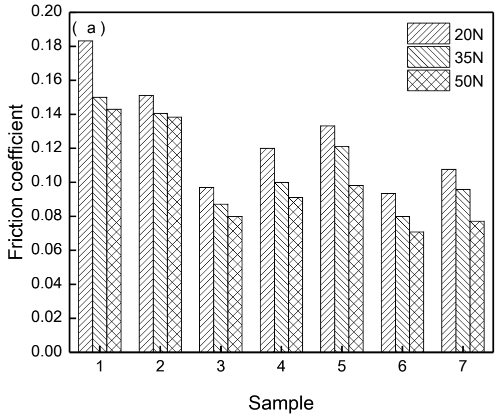


Figure. 4 Pure PEEK (a) ,10%PTFE/PEEK(b), 10%CF/PEEK(c) and 10%CF/5%PTFE/ PEEK(d) ultra-depth microscope friction and wear topography

Figure 5 is a histogram of the friction coefficient and wear rate of composite materials under different pressures. It can be seen from Figure 5(a) that when CF is added with 10%PTFE/PEEK as the matrix, as the CF content increases, the friction coefficient of the composite material gradually decreases. When the CF content exceeds 10%, the friction coefficient of the composite material gradually decreases. The constant increase of the composite material, the friction coefficient of the composite material gradually increases; As the friction pressure increases, the contact area between the material and the grinding ball increases, and the friction coefficient decreases slightly. The wear rate of the material and the friction coefficient show the same trend. When PTFE is added with 10%CF/PEEK as the matrix, as the content of PTFE increases, the friction coefficient of the composite material gradually decreases. When 5%PTFE is added, the friction coefficient reduction effect of the composite material is particularly significant, and when the content exceeds 5%, The friction coefficient of composite materials has a relatively small variation. It can be seen from Figure 5 that the friction coefficient and wear rate of 10%CF/5%PTFE/PEEK are the lowest. This shows that the addition of CF and PTFE can reduce the friction coefficient of the composite material. When only one additive is added, the composite material with 10%CF and 5%PTFE content has good wear resistance, and when CF/PTFE composite is added. When filling CF and PTFE have a synergistic anti-friction effect, and 10%CF/5%PTFE/PEEK has the best wear resistance.



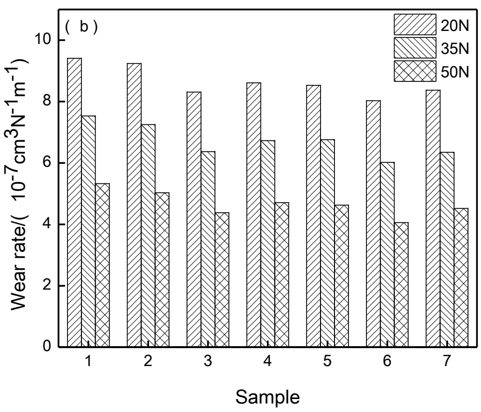


Figure. 5 Friction coefficient (a) and wear rate (b) of prepared composite materials

3.4 Micro morphology

Figure 6 shows the distribution of F and C elements obtained by X-ray energy spectrum analysis (EDS) on the surface of the 10%CF/5%PTFE/PEEK composite material. It can be seen from Figure 6 that the comparison of the distribution of F and C elements in the composite material Uniformity indicates that PTFE and CF have good dispersibility in the composite material without significant agglomeration.

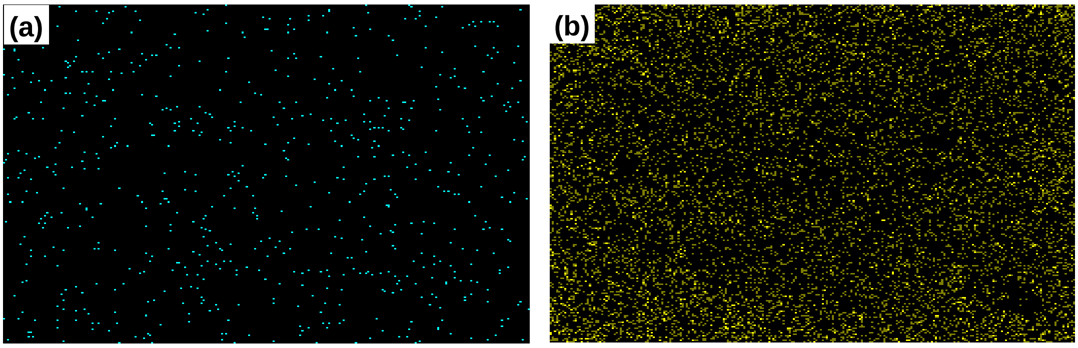


Figure. 6 Distribution of F(a) and C(b) elements in 10%CF/5%PTFE/PEEK

Figure 7 is the SEM image of the wear surface of the composite material with different CF content added with 10%PTFE/PEEK as the matrix. It can be seen from Figure 7 that when 5% content of CF is added, in the process of friction and wear, an incomplete transfer film is formed on the surface of the material, and adhesive wear occurs. When the amount of CF is increased to 10%, the surface of the material is more the complete transfer film has slight wear marks and shows relatively good wear resistance. When it is added to 15%, furrows appear on the surface of the material, showing obvious abrasive wear. This shows that the addition of carbon fiber can effectively improve the wear resistance of PEEK. The 10% content of CF is conducive to the formation of a transfer film on the surface and improves the wear resistance of the material. When the carbon fiber content exceeds 10%, friction and wear will occur. During the process, the surface will peel off, forming abrasive particles and aggravate surface wear.

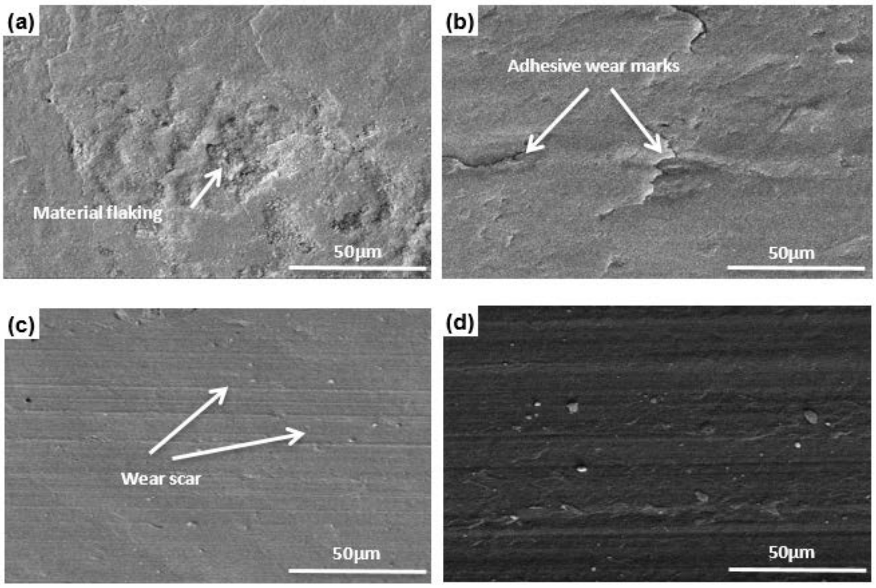


Figure. 7 SEM morphologies of composite materials with different CF content (a)0%CF;(b)5%CF;(c)10%CF;(d)15%CF

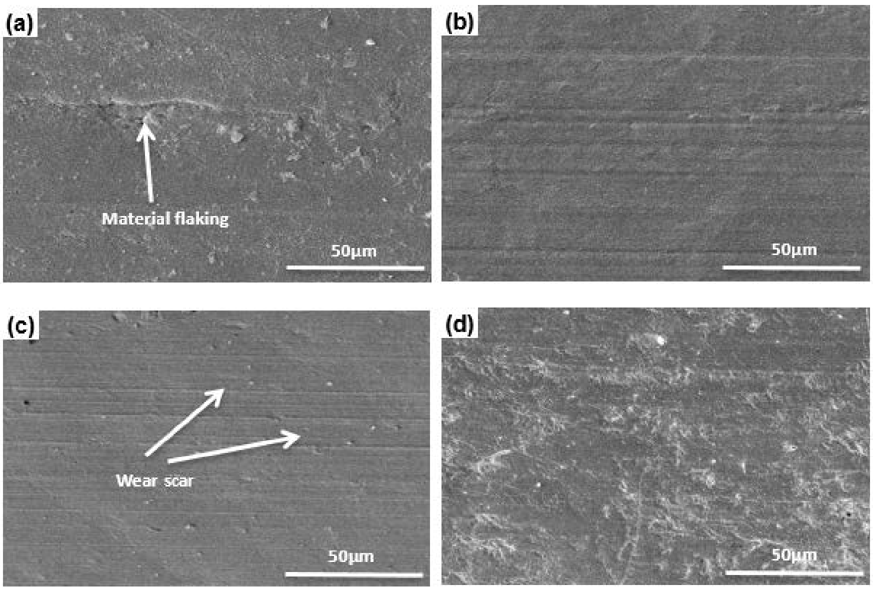


Figure. 8 SEM images of the wear surface of composite materials with different PTFE contents (a)0%PTFE;(b)5% PTFE ;(c)10% PTFE ;(d)15% PTFE

Figure 8 is the SEM image of the wear surface of the composite material with different PTFE content added with 10% CF/PEEK as the matrix. It can be seen from Figure 8 that when the PTFE content is added to 5% (Fig. 8(b)), a complete transfer film is formed on the surface of the material, and the material has good wear resistance. When the added content reaches 10%, Wear marks appear on the surface of the material, and when it continues to increase to 15%, obvious adhesion appears on the surface of the material. This shows that adding 5% of PTFE can make PEEK have good wear resistance, but because PTFE has a low elastic modulus and hardness, a higher content will reduce the bearing capacity of the material and make the surface of the material easier Adhesion occurs, so the amount of PTFE added should not be too large.

Figure 9 shows the SEM morphology of the wear surface of pure PEEK, 10%PTFE/PEEK, 10%CF/PEEK and 10%CF/5%PTFE/PEEK. It can be seen from Figure 9 that there are a lot of wrinkles on the surface of pure PEEK. When only PTFE is added, the surface of the composite material will peel off during the friction and wear process, and there are obvious adhesion marks (Fig.9(b)). When only CF is added, there are lighter adhesion marks on the surface of the material (Fig.9(c)). When CF and PTFE are added at the same time, a complete transfer film is formed on the surface of the material. The type of wear changes from adhesive wear to abrasive wear, with only slight the wear scar shows excellent wear resistance (Fig.9(d)). This shows that because PTFE itself has a low elastic modulus, it will weaken the mechanical properties of the composite material and is more likely to peel off during friction and wear. The higher elastic modulus of CF can enhance the internal bonding force of the material. Under the same conditions will not peel off. At the same time, adding CF and PTFE can play a synergistic effect on wear resistance, forming a complete transfer film on the wear surface, so that the composite material has better wear resistance.

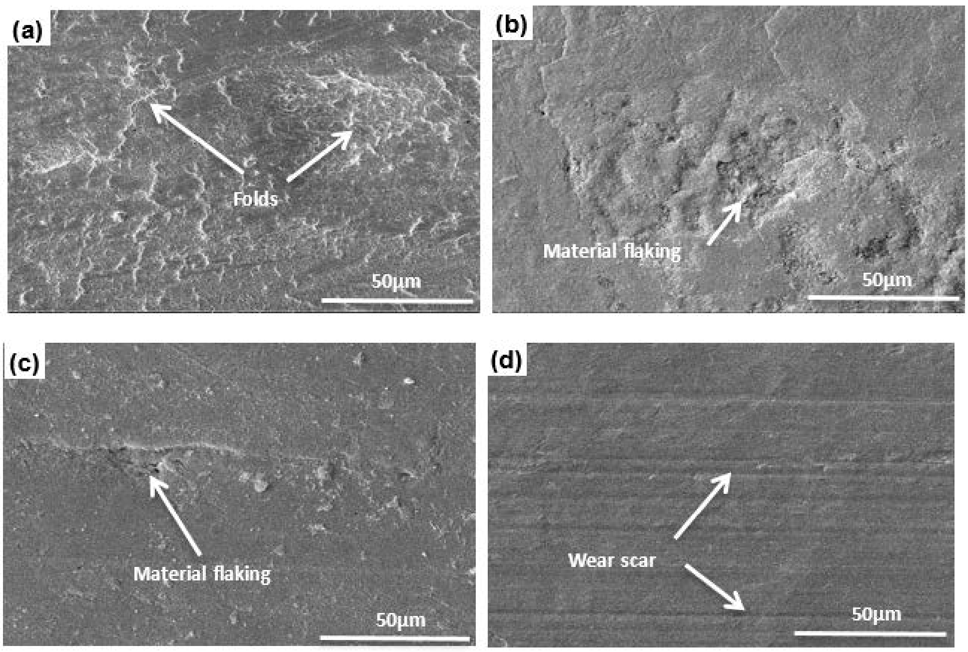


Figure. 9 Pure PEEK(a), 10%PTFE/PEEK(b), 10%CF/PEEK(c) and 10%CF/5%PTFE/ PEEK(d) wear surface SEM topography

4. Conclusion

(1) The hardness and compressive strength of composite materials increase with the increase of CF content, and decrease with the increase of PTFE content. Among them, 10%CF/PEEK has the best mechanical properties, with hardness and compressive strength of 30.24HV and 110.4MPa respectively.

(2) The friction coefficient of the composite material first decreases and then increases with the increase of the CF content. The friction coefficient and wear rate of the composite material are the lowest when 10% CF is added; the addition of PTFE can reduce the friction coefficient of the material. When 5% PTFE is added, the friction. The coefficient has the largest decrease. When it exceeds 5%, the coefficient of friction and wear rate decrease relatively low and are basically in a stable state. When CF/PTFE composite filler is added, CF and PTFE can play a synergistic effect on wear resistance and improve the wear resistance of composite materials. Considering the mechanical properties and wear resistance of composite materials, 10%CF/5%PTFE composite filler can be used. While the friction coefficient of the composite material is significantly reduced, the mechanical properties are only slightly reduced. It can be seen that 10%CF/5%PTFE/PEEK has the best comprehensive performance.

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