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TECHNICAL ARTICLE

Hydrophobicity and Wear Resistance of Textured Carbon Fiber/Polytetrafluoroethylene Composite Coatings

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In the process of unconventional oil and gas resources exploitation, bit balling and life of polycrystalline diamond compact (PDC) bit. PDC bits are common problems to be solved. Polytetrafluoroethylene (PTFE) coating is suitable for PDC bit steel, but we need to use surface technology to further improve the hydrophobic and tribological properties of PTFE coating. In this study, carbon fiber (CF)-modified PTEF composite coating was prepared on 35CrMo steel substrate by one-step spraying method and was then laser textured. The effects of CF with different mass fractions (10, 20, 30%) of 200 μm texture diameter and 100 μm spacing on the contact angle, friction coefficient and wear rate of the composite coating were studied. When the mass fraction of CF is 30%, the hydrophobic property of the coating is the strongest. When the mass fraction of CF is 20%, the coating has good tribological properties under certain hydrophobicity. The research content of this paper has certain guiding significance for solving the problem of PDC bit balling and the service durability of composite modification layer.

Keywords CF/PTFE, composite coatings, friction and wear, hydrophobicity, texture

1. Introduction

In the exploration and development of petroleum and mineral resources, drilling rig is the main rock breaking tool, and the performance of the core bit of the drilling rig directly affects the drilling quality, drilling efficiency and drilling cost (Ref 1). PDC drill is suitable for shale oil and gas resource exploration and development. PDC bit in shale drilling process prone to "bit balling phenomenon" will not only hinder bit drilling and reduce mining efficiency, but also cause serious mining accidents (Ref 2, 3). A large number of studies have shown that the surface treatment of drill bit can effectively solve the problem of drill head mud pack. Sha et al. used boron (B)-coated diamond particles to spray on PDC bits to form a uniform boron carbide (B4C) barrier layer, inhibiting the oxidation of PDC bits.(Ref 4). Octavio et al. found that electroplated Ni-P coating has low surface roughness and can reduce mechanical adhesion. The electronegativity of the surface can weaken the electrochemical adhesion of the mud, thereby reducing the mud inclusion phenomenon of the PDC bit. However, the electroplating method has great pollution and edge effect, and the process repeatability of coating on the steel body of complex structure drill is poor (Ref 5). Therefore, it is of great significance to solve the pit balling problem of PDC bit by other surface treatment methods for improving drilling efficiency, avoiding mining accidents and saving mining cost. Texture treatment of drill bit surface is expected to become a new way to solve the problem of pit balling. Micro-nano structure was prepared on the surface of the drill to improve the hydrophobicity and anti-adhesion of the drill surface, so as to prevent the pit balling problem of the drill head. On the other hand, surface texture can also improve the wear resistance of the drill and the service life of the drill.

The usual preparation methods of surface texture include mechanical processing, reactive ion etching, electron beam etching, chemical deposition, sol-gel method and laser surface texture (Ref 6, 7). Among them, laser surface texture (LST) has the advantages of no pollution to the environment, simple fabrication, short time consuming, low cost, wide processing range, and can control the size and morphology of the surface texture. It has been widely used in a variety of material surfaces to improve their hydrophobicity and tribological properties (Ref 8-11). Qi et al. prepared round pitting LST on the surface of 1045 steel and studied the tribological properties of PTFE/Kevlar composite braids under dry friction conditions. The micro-protrusion structure reduces the stress concentration at the edge of the surface texture, captures and stores the lubricating material, and quickly forms a stable PTFE transfer film. The friction coefficient and wear rate are effectively reduced (Ref 12). Qin et al. constructed superhydrophobic PTFE surface by direct ablation of 355 nm picosecond laser. Under the condition of only changing the laser scanning interval, the contact angle can change from the original 112° to the maximum 167.9°, and the surface has an accurate and

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continuous adjustable water adhesion from ultra-low to ultra-high (Ref 13).

At present, the research on artificial hydrophobic coating shows that PTFE is used as the main raw material to prepare hydrophobic coating (Ref 14). PTFE is a fluoropolymer copolymerized with tetrafluoroethylene, which has the characteristics of high crystallinity, large molecular weight and no branched chain (Ref 15). Adding different kinds of additives to PTFE is an effective way to improve its hydrophobicity and tribological properties (Ref 16-18). Wan et al. prepared hydrophobic coatings by physical blending of SiO_2 and PTFE nanoparticles. It is found that the coating surface filled with hydrophobic SiO_2 has better uniformity than untreated SiO_2, and the maximum SCA measured on the composite coating surface is 163.1° (Ref 19). Yu K used aphanitic graphite and SiO_2 as modifier to improve the tribological properties of PTFE, and the wear resistance of the material was increased by 2.5 times(Ref 20). CF has high specific strength, high modulus, excellent wear resistance and creep resistance, which can be effectively used to enhance the mechanical properties of PTFE(Ref 21). However, due to the poor affinity between CF and PTFE and the low interfacial bonding strength between them, CF will appear uneven dispersion and poor performance in PTFE coating. Silane coupling agent KH550 can be used as a chemical medium between PTFE and CF to connect them firmly and improve the interface bonding strength. Yan et al., after treating CF/PTFE composites with different gases, found that argon plasma treatment of PTFE dispersion-coated CFs could increase the tensile strength and elongation at break of the composites by 49 and 100%, respectively, and decrease the wear rate by 56% (Ref 22). Wu et al. made a composite coating using CF-reinforced polytetrafluoroethylene. When the mass fraction of CF was 20%, water contact angles (WCA) reached 122.0 ± 2.0°, showing excellent hydrophobic properties (Ref 21).

At present, there are few studies on laser texture on the surface of the coating prepared by adding CF into PTFE. In this study, silane coupling agent KH550 was used to treat CF, so that it can be evenly dispersed in PTFE and well combined. CF-reinforced polytetrafluoroethylene composite coatings with different CF contents were prepared by one-step spraying method. Then, the surface textures of the composite coating were made by UV laser marking machine. The two-dimensional and three-dimensional morphologies of the coating surface were observed, and the static contact angles (SCA) of different coating surfaces were measured. In addition, the hydrophobic mechanism and wear mechanism of the composite coating were analyzed.

2. Experimental

2.1 Materials

PTFE emulsion (model FR301G) containing 60 wt.% of the PTFE resin was dispersed in deionized water, as obtained from Shanghai 3F New Materials Co. Ltd. CFs (diameter about 7 μm, aspect ratio 12:1, density 1.75 g/cm^3) are provided by Nanjing Zhining New Material Co., Ltd. Silane coupling agent KH550 (analytical pure) is provided by Shandong Yousuo Chemical Technology Co., Ltd. 35CrMo steel substrate (diameter 30 mm, thickness 5 mm) is provided by Beijing Binpeng Yinghao Technology Co., Ltd.

2.2 Preparation of Coatings

The matrix is 35CrMo steel (30 × 3 mm). After sand blasting, a certain amount of CF (10, 20 and 30 wt.%) and 1 wt.% silane coupling agent KH550 were added into the PTFE emulsion, and the mixture was uniformly dispersed by a magnetic mixer for 15 min. The mixture was loaded into the ANESTIWATAW-71 spray gun. The spray gun pressure was 0.3 MPa, the gun mouth was 20 cm away from the workpiece, and the spray angle was 45 degrees. Curing at 370 °C for 20 min, natural cooling in air to room temperature. The concave texture was prepared on the surface of composite coating by laser marking machine.

2.3 Contact Angle Test

At room temperature, the static contact angle was measured by HARKE-SPCA-X3 contact angle measuring instrument. The equipment was adjusted to the horizontal position, the sample was fixed, and the observation lens was adjusted. The droplets (about 5 μL) were dropped on the coating surface by a needle tube. The vibration of the equipment and the desktop was avoided in the measurement process. After the droplets were stabilized, the four random positions of each sample were measured, and each position was measured for three times. The average value of the 12 results was taken as the static contact angle of the sample.

2.4 Mechanical Property Measurement

The tensile properties were tested according to GB/T1040.1-2018, and the tensile speed was 10 mm/min. In Rockwell hardness test, the initial test pressure is 99 kN, and the main test pressure is 980 kN.

2.5 Friction and Wear Tests

The friction and wear tests of textured CF/PTFE composite coating were carried out on an MS-T3001 friction and wear tester. For the same sample, the average value is taken to ensure the reliability of the experiment. Rotating friction and wear tests were carried out in four positions on 35CrMo steel. The test parameters were set under the following conditions: 10 N load, 4 mm wear scar radius, 200 R/min rotation speed and 30 min test duration in air.

2.6 Characterization

After the rotating friction and wear experiment, an annular wear scar can be obtained. The top and bottom cross sections of the wear scar are selected to obtain the corresponding height contour curve, depth and cross-sectional area. After measuring the 12 positions of the wear scar, the average value of 12 measurements is taken to calculate the specific wear rate. K (mm^3 N^{-1} m^{-1}) as the specific wear rate, using the following equation:

$$K = \frac{S \cdot l}{L \cdot F}$$

where S is the cross-sectional area in mm^2, l is the wear track length in mm, L is the total sliding distance in m, and F is the applied load in N.

The surface morphologies of coatings before and after wear were characterized by Zeiss Supra™55 scanning electron microscope. Sputtering a layer of platinum on the coating surface can improve the resolution of scanning electron microscope (SEM). The wear morphologies of textured CF/PTFE composite coatings were characterized by confocal laser scanning microscope (CLSM, Olympus, ols4100). The worn surfaces of LST were analyzed by energy-dispersive x-ray spectroscopy (EDS).

3. Results and Discussion

3.1 Contact Angle and Coating Characterization

Figure 1 shows the SEM morphologies of the pure PTFE coating and three textured coatings of 10% CF/PTFE, 20% CF/PTFE and 30% CF/PTFE and the static contact angles of water on the corresponding coating surface. The diameter of textured CF/PTFE composite coating is about 200 μm, and the spacing is about 100 μm. The results show that with the increase in CF mass fraction, the contact angle increases, which are 114.4° ± 2.2°, 125.1° ± 2.9°, 136.9° ± 3.3° and 141.0° ± 2.5°, respectively. The 3D morphologies of the four coatings are shown in Fig. 2. By using laser texture technology, patterns with certain geometric morphology, size and distribution law can be machined on the surface of the coating. It can effectively improve the lubrication state of the surface contact mode of the friction pair. The roughness of the four coatings were 1.23 ± 0.15, 2.14 ± 0.21, 2.68 ± 0.26 and 4.03 ± 0.17 μm, respectively.

The cross sections of four kinds of coatings were cut by WEDM and then polished by sandpapers (400, 800, 1000, and 1500 #) in turn until the mirror surface was obtained. The cross-sectional morphologies of coatings were analyzed by SEM. The results are shown in Fig. 3. It can be seen that the cross sections of all coatings are uniform, and the thickness of the coating is about 40-50 μm, without obvious defects. The CFs evenly distribute in PTFE. There is no obvious cavity and large particle agglomeration, and the coating is well bonded with the substrate.

3.2 Hydrophobic Mechanism

The relationship between CF content, contact angle and surface roughness of the four coatings is shown in Table 1. The roughness and contact angle of textured CF/PTFE composite coatings are obviously higher than that of pure PTFE coating without any treatment. Therefore, the textured CF/PTFE composite coating shows better hydrophobicity than pure PTFE coating, and the hydrophobicity of the textured 30% CF/PTFE coating is the best, followed by the textured 20% CF/PTFE coating.

According to Cassie–Baxter, the contact between the water droplet and the composite coating can be regarded as composite contact. The liquid cannot fill the grooves on the surface, and some pores support the droplets in the middle. Apparent CA (θ_γ) can be estimated from the following equations:

$$\cos\theta_\gamma = f_1 \cos\theta - f_2$$
$$f_1 + f_2 = 1$$

where θ represents the inherent CA of droplets on the rough surface; f_1 is the fraction of the interfacial area between solid and liquid, and f_2 is the fraction of the contact between solid and air.

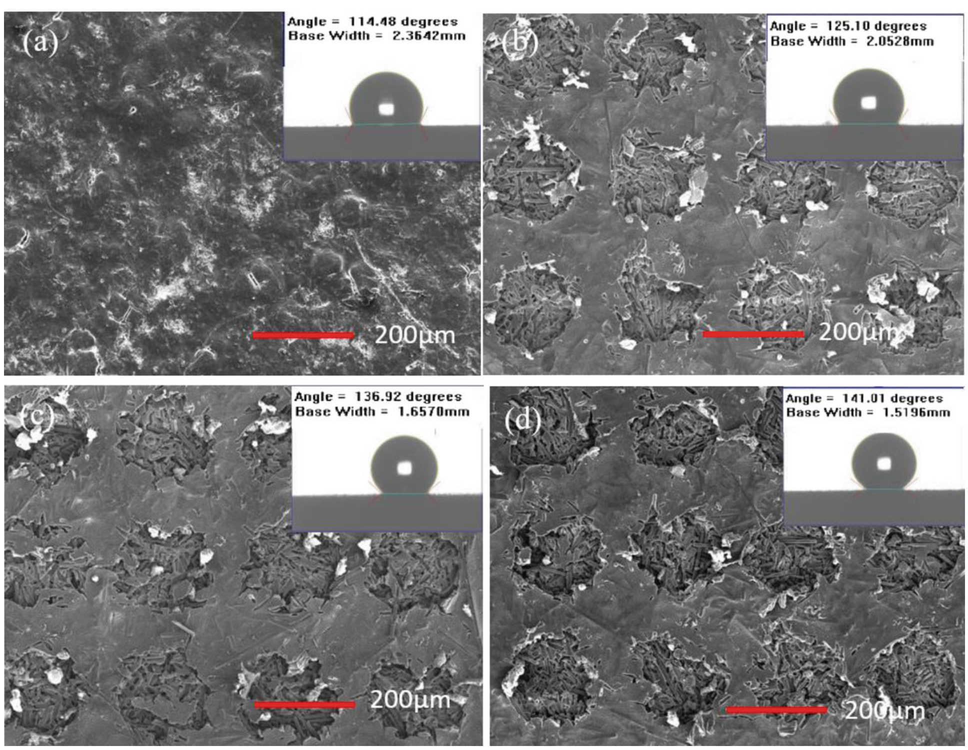

Fig. 1 SEM and SCA of (a) pure PTFE (b) textured coating of 10% CF/PTFE, (c) textured coating of 20% CF/PTFE and (d) textured coating of 30% CF/PTFE

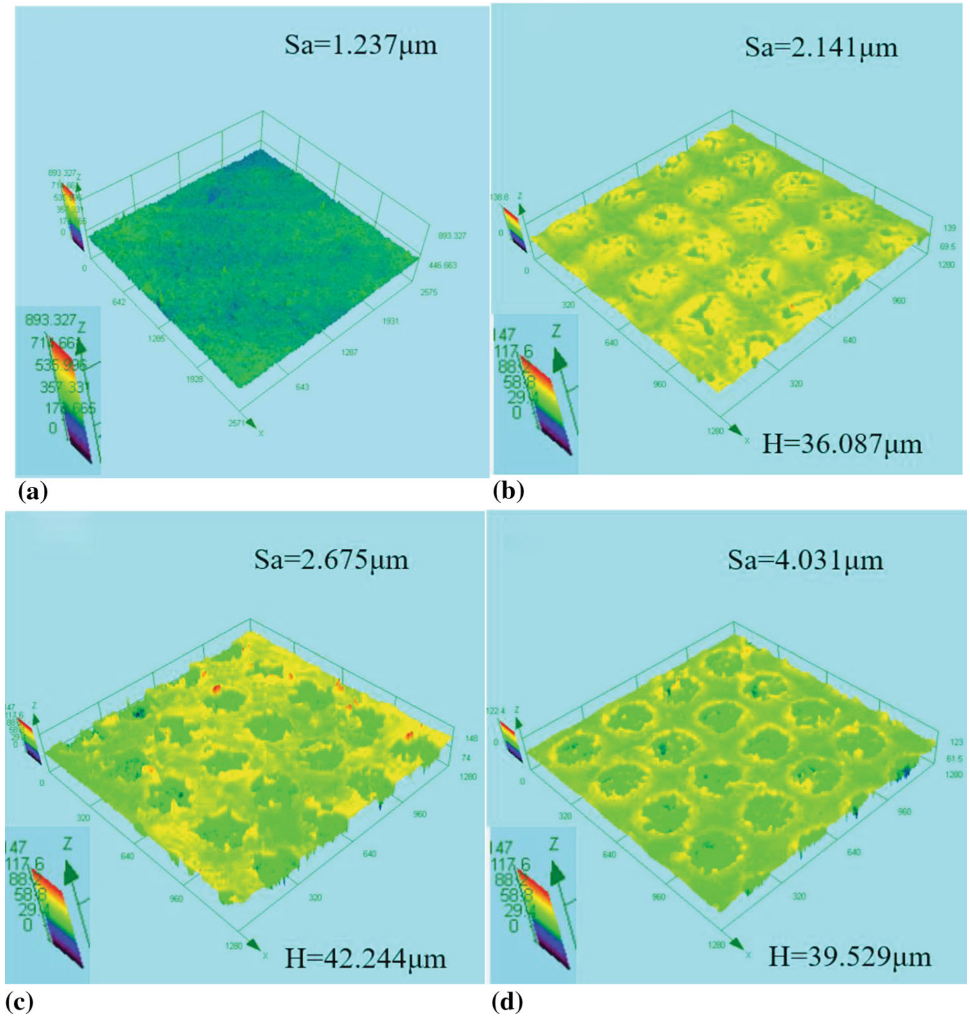

Fig. 2 3D morphologies of four coatings: (a) pure PTFE, (b) textured coating of 10% CF/PTFE, (c) textured coating of 20% CF/PTFE, (d) textured coating of 30% CF/PTFE

The weight percentage of each element specific to the four coatings is shown in Table 2. After wear, more iron content means more wear failure. The iron content of textured 20% CF/PTFE composite coating after wear is much lower than that of pure PTFE coating after wear.

In order to further analyze the influence of chemical composition on hydrophobicity of the composite coating, the cross section of 30% CF/PTFE textured composite coating with the largest contact angle was analyzed by EDS in backscattered electron imaging mode, as shown in Fig. 4. The yellow area is the distribution of F element, which accumulates on the surface of the coating, resulting in the decrease in the surface energy of the coating, which well explains the reason for the excellent hydrophobicity of the 30% CF/PTFE textured coating from the chemical composition (Ref 16).

3.3 Mechanical Properties

Table 3 is the mechanical properties of pure PTFE coating and textured composite coating with different CF content. It can be seen from the table that the elastic modulus of the coating increases greatly after adding CF compared with the pure PTFE coating. And with the increase in CF content, the elastic modulus of the composite coating increases continuously. It shows that the ability of the coating to resist deformation is effectively improved after adding CF. This is because CF dispersed in PTFE, when subjected to external force, can make different components together, effectively prevent the coating crack. After adding CF, the continuity of PTFE matrix decreases, and the effect of PTFE transfer stress is weakened, which leads to the decrease in tensile strength of composites. CF has high specific strength, high modulus, excellent wear resistance and creep resistance, which can effectively enhance the strength of PTFE and overcome the shortcomings of low hardness. When the CF content is 20%, the hardness can reach 68.5 HRE. However, due to the chemical inertia of CF, the affinity between CF and PTFE is poor, and the interfacial bonding strength is low. As a result, when the CF content continues to increase, the hardness of the composite coating decreases slightly due to the fact that excessive CF cannot better combine with PTFE coating.

3.4 Tribological Properties

Figure 5 shows the variation of friction coefficient of different coatings with time. In the initial stage of friction and wear experiment, the friction coefficient fluctuates greatly due to the insufficient smooth surface of the coating and the

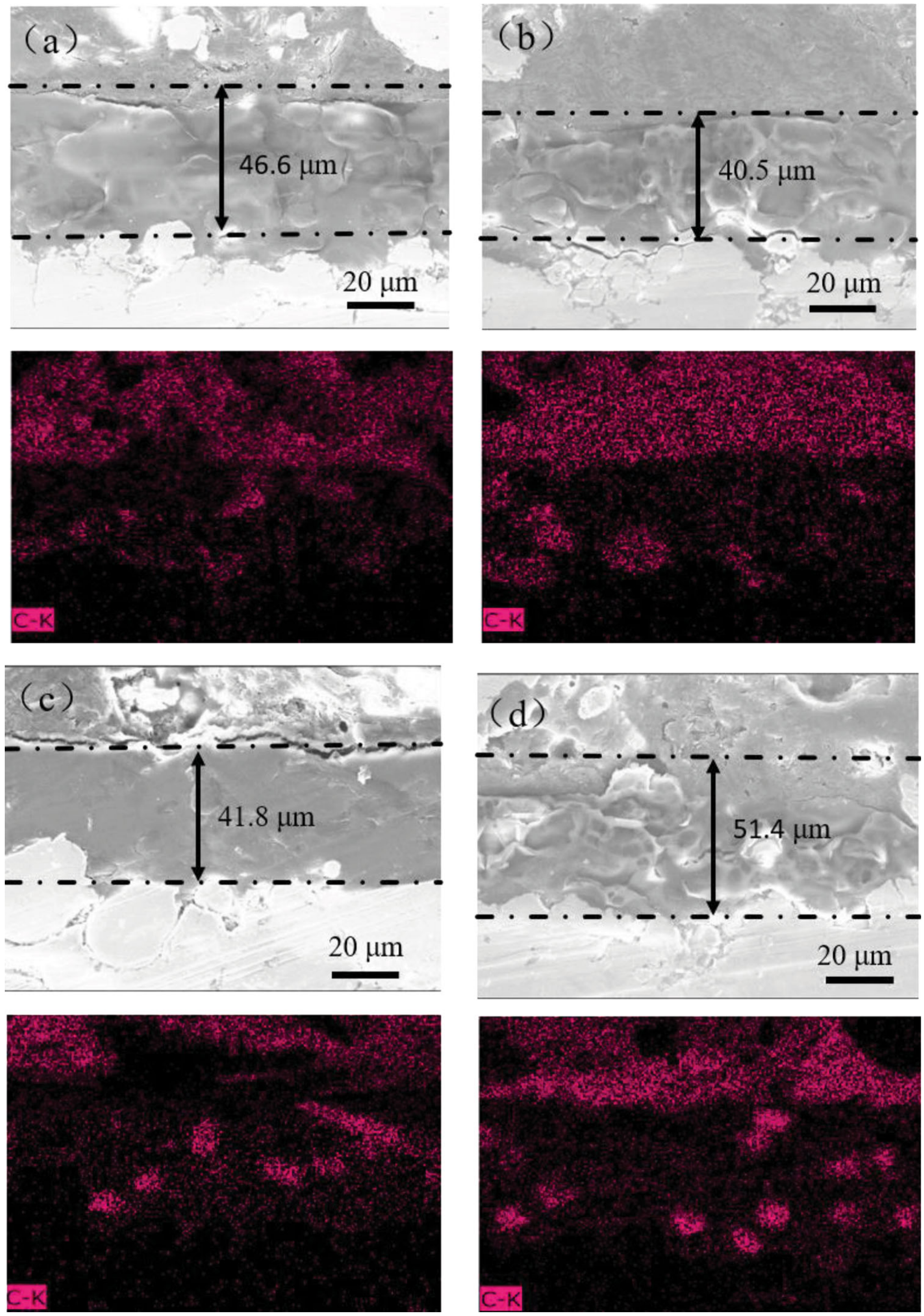

Fig. 3 SEM cross-sectional morphology of the coating: (a) pure PTFE, (b) textured coating of 10% CF/PTFE, (c) textured coating of 20% CF/PTFE, (d) textured coating of 30% CF/PTFE

Table 1 Contact angle and surface roughness of different coatings

Sample	SCA, deg.	Surface roughness, μm
Pure PTFE	114.4 ± 2.2	1.23 ± 0.15
Textured coating of 10% CF/PTFE	125.1 ± 2.9	2.14 ± 0.21
Textured coating of 20% CF/PTFE	136.9 ± 3.3	2.68 ± 0.26
Textured coating of 30% CF/PTFE	141.0 ± 2.5	4.03 ± 0.17

insufficient contact between the coating and the steel ball. The average friction coefficient of pure PTFE coating is 0.12. When the CF content is 10, 20 and 30%, the average friction coefficient is 0.13, 0.13 and 0.14, respectively. It can be concluded that the friction coefficient of textured CF/PTFE composite coating is slightly higher than that of pure PTFE coating, but the difference is not obvious, indicating that CF content and surface texture have little effect on the friction coefficient of the coating. It is a good phenomenon that the friction coefficient of the textured CF/PTFE coating cannot be increased too much.

Figure 6 shows the three-dimensional topography of worn surfaces. The wear trace depth of the textured CF/PTFE composite coating is shallow, indicating that mild wear occurs.

Table 3 shows the wear rate of different coatings and the change of wear rate with CF mass fraction is shown in Fig. 7. The wear rate first decreases and then increases with the increase in CF mass fraction, and reaches the lowest at the textured coating of 20% CF/PTFE. The wear rate is 8.70×10^{-4} mm^3/N·m, which is about 27.5% less than that of pure PTFE. One end of the dispersant KH550 is connected to the hydroxyl group of CF and the other end is connected to the polymer chain of PTFE, making each other firmly connected, strengthening the network structure of PTFE and improving the wear resistance of the coating. Because of its high strength and wear resistance, CF can be used as reinforcement material in composite materials. CF is uniformly dispersed in PTFE to

Table 2 EDS element analysis of four coating surfaces

Specimen	C, %	O, %	F, %	Fe, %	Total, %
Pure PTFE	16.46	6.23	57.82	19.49	100
10% CF/PTFE coating	23.53	5.55	56.86	14.06	100
20% CF/PTFE coating	37.61	4.21	53.32	4.86	100
30% CF/PTFE coating	32.98	5.16	54.10	7.76	100

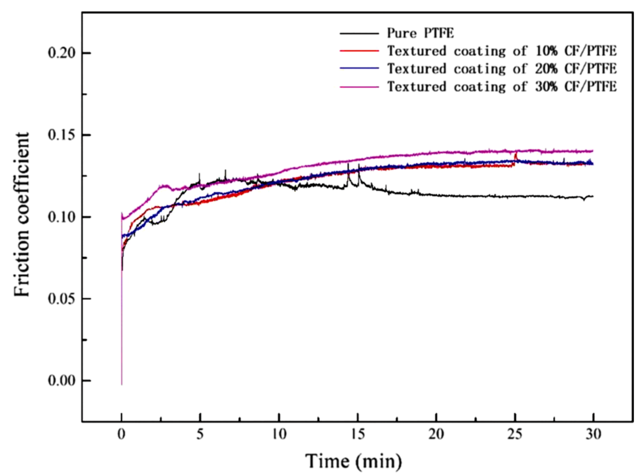

Fig. 5 Variation of friction coefficient of different coatings with time

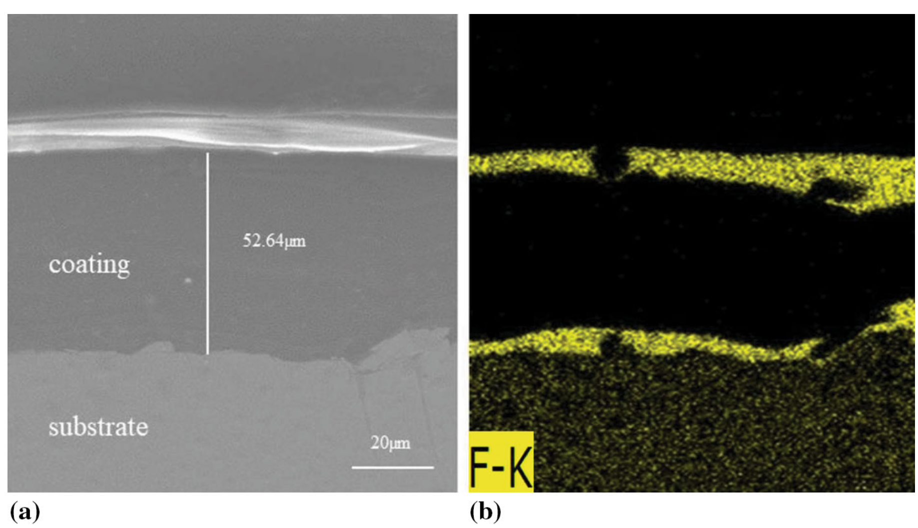

Fig. 4 SEM and EDS of 30% CF/PTFE textured composite coating

Table 3 Mechanical properties of different coatings

Sample	Elastic modulus, MPa	Tensile strength, MPa	Hardness, HRE
Pure PTFE	357	29.6	50.0
Textured coating of 10% CF/PTFE	722	27.2	63.8
Textured coating of 20% CF/PTFE	965	13.8	69.1
Textured coating of 30% CF/PTFE	1232	10.5	66.4

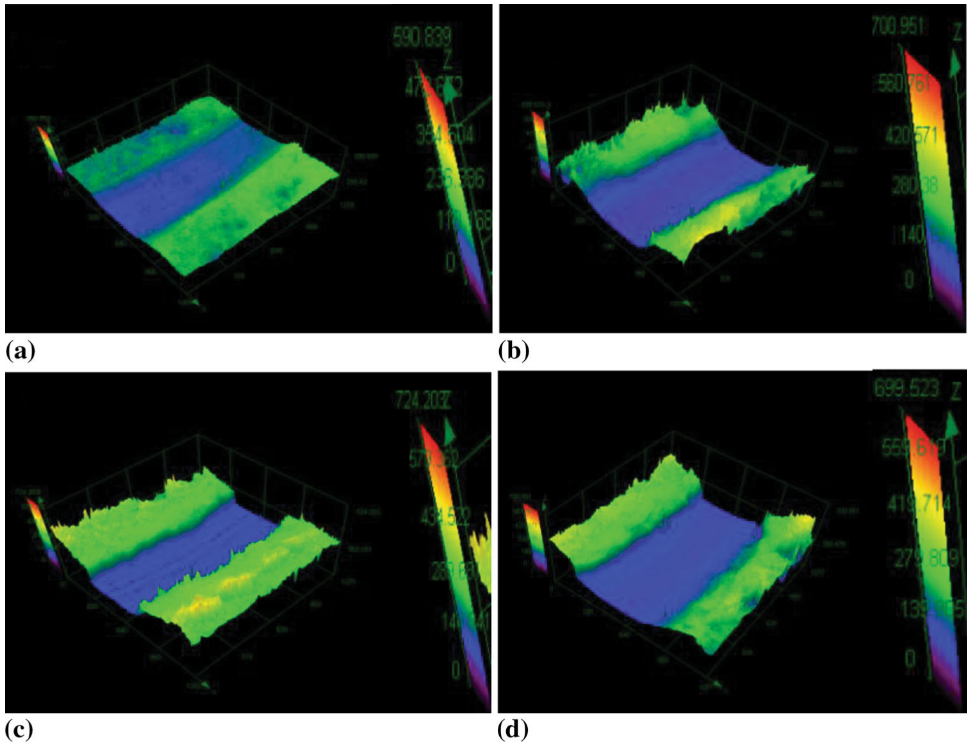

Fig. 6 Three-dimensional topography of worn surface: (a) pure PTFE, (b) textured coating of 10% CF/PTFE, (c) textured coating of 20% CF/PTFE, (d) textured coating of 30% CF/PTFE

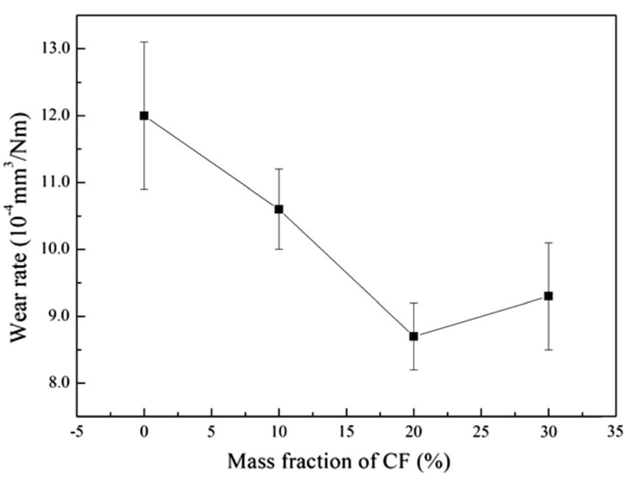

Fig. 7 Change of wear rate with CF mass fraction

transfer stress and bear most of the load during friction and wear. Therefore, the plastic deformation of PTFE is hindered to a certain extent and the wear of PTFE is delayed. Excessive CF cannot combine with PTFE well, so it is easier to peel off, which reduces the wear resistance of the coating. The addition of coupling agent can make CF evenly dispersed in PTFE, playing the role of stress transfer, and hindering the plastic deformation of the material to a certain extent, which can prevent the large-area damage of PTFE coating and delay the wear of PTFE (Ref 23). In addition, surface textures also play a role in reducing abrasive wear and play a continuous buffer role in the process of wear.

3.5 Wear Mechanism

SEM images of worn surfaces of different coatings and their corresponding EDS spectra are shown in Fig. 8. In Fig. 8(a), 35CrMo matrix was seriously exposed and delamination was serious. There were many obvious furrows and holes, and a lot of debris accumulation, which indicates that the abrasive wear and adhesive wear are very serious. In Fig. 8(b), many parts of the wear surface of 35CrMo matrix were exposed, and there were a certain amount of wear debris on both sides of the wear trajectory. The surface is stratified, and obvious furrows and pits appear. It indicates that abrasive wear and severe adhesive wear occur. The addition of CF and laser surface texture can improve the wear resistance of PTFE coating to a certain extent, but the CF content and texture size do not reach the ideal parameters, and the improvement is not obvious. In Fig. 8(c), a small part of 35CrMo substrate was exposed. There was no obvious delamination phenomenon, and the wear surface was relatively smooth and flat. There were slight furrows along the sliding direction, no obvious holes, and a small amount of wear debris. The above indicates that slight abrasive wear and adhesive wear have occurred, and the wear resistance of 20% CF/PTFE textured composite coating has been significantly improved. In Fig. 8(d), more 35CrMo matrix were exposed. There were slight delamination, some furrows along the sliding direction, and less debris and holes, indicating abrasive wear and slight adhesive wear occurred. The reason is that the excessive CF cannot combine with PTFE coating well to transfer stress, and the detachment of excessive CF may lead to the aggravation of abrasive wear.

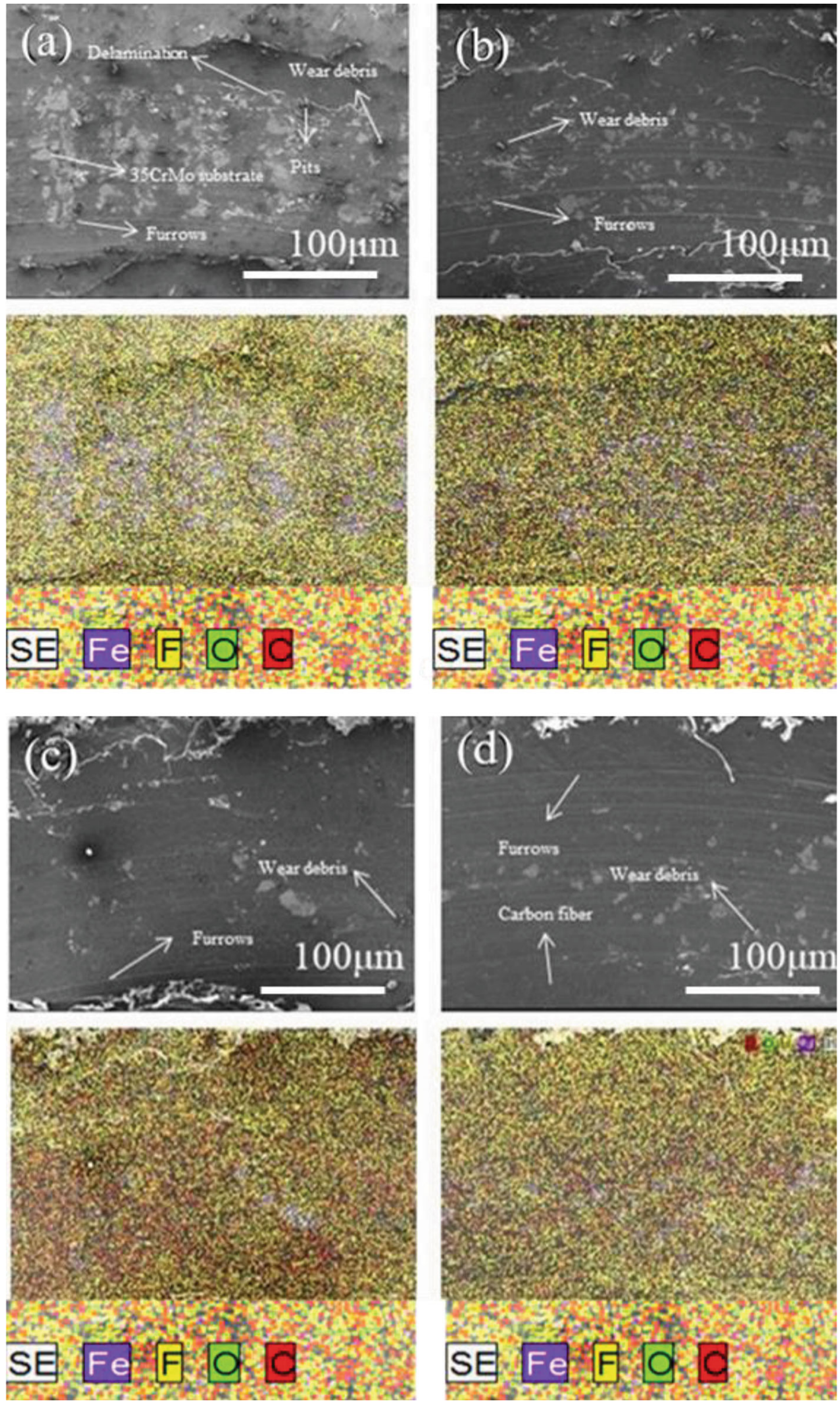

Fig. 8 SEM and EDS of four coatings: (a) pure PTFE, (b) textured coating of 10% CF/PTFE, (c) textured coating of 20% CF/PTFE, (d) textured coating of 30% CF/PTFE

4. Conclusions

The textured CF/PTFE coatings have better hydrophobic and tribological properties under dry friction conditions, compared with the pure PTFE coating. The results show that the contact angle of 30% CF/PTFE composite coating with texture diameter of 200 μm and texture spacing of 100 μm is 141.0° ± 2.5° and the surface roughness is 4.03 ± 0.17 μm, indicating excellent hydrophobic properties. The laser surface texture and addition of CF increase the surface roughness of the coating, which is the physical reason for the excellent hydrophobicity of the textured composite coating. The aggregation of F element on the coating surface reduces the free energy of the coating surface, which is the chemical reason for the excellent hydrophobicity of the textured composite coating. When the mass fraction of CF is 20%, the friction coefficient is 0.13, which is slightly higher than that of pure PTFE coating. The wear rate is reduced to 8.70×10^{-4} mm^3/N·m, which is 27.5% lower than that of pure PTFE coating. The addition of CF can transfer the stress in the PTFE coating, play the role of internal support and prevent large-area damage of the coating. The main wear mechanisms of composite coatings are abrasive wear and adhesive wear. The contact angle of the coating increased from $114.4° \pm 2.2°$ to $136.9° \pm 3.3°$, and the surface roughness increased from 1.23 ± 0.15 to 2.68 ± 0.26 μm, with a certain hydrophobicity. In general, the textured 20% CF/PTFE coating has the best comprehensive properties. Surface textures have two sides. It can collect debris and disperse stress. On the other hand, the surface microstructure will also increase the contact stress. How to make good use of the improvement brought by surface textures, reduce its negative effects, explore the influence of more size and shape texture, and the content span of composite layer additives need to be further studied in the future. This is of great significance to effectively extend the service life of the coating and improve the service durability of the bit.

Acknowledgments

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