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Tribological properties study of carbon fabric/ epoxy composites reinforced by nano-TiO_2

The carbon fiber fabric as reinforcement, carbon fiber fabric/epoxy composites are prepared with epoxy resin as the binder system, and this composite material, to which is filled with nano TiO_2 and nano modified composite materials are studied in the different loading conditions of friction and wear the effects of fillers, and the content of fillers on the tribological properties of the composites, the friction and wear mechanism are discussed. The results show that the nano TiO_2 can achieve good antifriction reducing grinding effect, nano TiO_2 and the optimum adding ratio of 3.0%. in this process, the wear of the composites by brittle spalling gradually turns to abrasive wear.

Keywords: Epoxy resin, carbon fabric, filling modification, friction and wear

1. Introduction

Polymer matrix composites are increasingly used in the field of mechanical friction because of their unique advantages of light weight, high strength, abrasion resistance, and resistance to load. The thermosetting polymer epoxy resin (EP) expresses an excellent adhesion due to the presence of epoxy groups in the structure that can interact with the active hydrogen groups on the surface of the metal material, and is often used as a protective coating applied to the surface of metal parts to improve their wear resistance. However, the simple epoxy resin is limited by its cross-linking structure of three-dimensional network, and has a large brittleness. The anti-peeling and frictional wear properties are inferior to those materials such as polyether ether ketone, PTFE, and nylon, and are difficult to satisfy the increasingly stringent performance requirements of friction materials in real operating conditions. For this reason, people usually adopt the method of compounding epoxy resin with other materials in order to obtain better friction reduction effect[1-4].

Carbon fiber fabric/epoxy composite (CF/EP) is a new type of composite material developed in recent years. This kind of material uses carbon fiber fabric as the base material and epoxy resin as the binding phase. It has the advantages of high modulus, high strength, light weight, wear resistance, good toughness, self-lubricating properties and epoxy adhesiveness. It can effectively adhere to the liner material or anti-wear coating that is attached to the surface of the material for the harsh friction environment, and has excellent process ability and good development prospects[5-8]. Up to now, research reports on such composite materials have mostly focused on the preparation methods of materials, and there are fewer studies on the tribological modification of composite fillers. For this reason, nano-TiO_2 was selected as the filler in this experiment, and the influence of filler content on the tribological performance of carbon fiber fabric/epoxy resin matrix under different friction conditions was investigated[9-13], and the friction and wear mechanism was analyzed.

2. Experimental part

2.1. Raw materials for experiment and main equipment

Epoxy resin (E-44, Zhenjiang Danbao Resin Co., Ltd.); low molecular weight polyamide (650, Zhenjiang Danbao Resin Co., Ltd.); butanone (AR, Xi'an Chemical Reagent Factory); toluene (AR, Tianjin Damao Chemical Reagent Factory); flat fabric of carbon fiber (Parameters are shown in Table 1, Jiangsu Tianniao High-tech Co., Ltd.); nanometer TiO_2 (25nm, Shanghai Chaowei Nano Inc, Anatase); silane coupling agent (KH550, Nanjing Dunning Coupling Agent Co., Ltd.); ultrasonic cleaner (KQ3200E, Kunshan Ultrasonic Instrument Co., Ltd.); magnetic stirrer (ZNCL-S, Zhengzhou Kaipeng Test Instrument Co., Ltd.); friction and wear tester (MM-200, Xuanhua Tester Factory).

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Table 1. The parameters of carbon fiber plane fabric

Type	Reinforcement Yarn		Weave	Fiber Count (10mm)		Weight (g/m^2)	Width (mm)	Thickness (mm)
	WWarp Yarn	Weft Yarn		Warp Ends	Weft Picks			
HY-1K-P	1K	1K	Plain	10.5	10.5	140	100	0.17

2.2. Sample preparation

The binder phase of epoxy resin used in this test contained both A and B components: in the A component, the base epoxy resin and the diluent butanone are formulated according to the ratio of m epoxy: v butanone = 1:2.4; in the B component, the curing agent polyamide and the diluent toluene are also formulated in the proportion of m polyamide:v toluene=1:2.4. The appropriate size of carbon fiber fabrics is cut and soaked in acetone solution, processed by ultrasonic cleaning at room temperature for 2h, and transferred to a constant temperature drying oven to be dried at 80°C for 24h, which is for standby application. According to the proportion of 2% of carbon fiber fabric/epoxy composite material, the appropriate mass of KH-550 silane coupling agent is weighed and formulated into the 90% ethanol solution. Nano TiO_2 are respectively added. After it is magnetically stirred for 60 minutes, the solution is evaporated to dryness at 120°C and cooled to room temperature to prepare a modified nanofiller. During adding component A, appropriate mass fraction of modified nanofiller is added. Then, the carbon fiber fabric is repeatedly impregnated and brushed in epoxy resin uniformly mixed with the B component in a mass ratio of 1:1 until the carbon fiber fabric accounted for 60% to 70% of the mass fraction of the composite material. Finally, the composite material is bonded to the surface of a 45# steel block and solidifiedat 80°C for 2 hours under a vacuum condition of 0.1 MPa to prepare a test sample.

2.3. Test method

The MM-200 ring-block friction and wear tester was used to evaluate the tribological properties of the material. The initial grind thickness of the carbon fiber fabric/epoxy resin composite was 1.5 mm, and the coated steel block size was 20 mm×8 mm×11 mm. The friction pair used quenched 45 # steel ring, whose the hardness is 40 ~ 50HRC and size is ø50 mm×10 mm. Before the friction and wear test, the surface of the dual steel ring is polished with 800# and 1200# water-based sand paper successively until surface roughness Ra is 0.2-0.45μm, and cleaned with acetone. During the test, the sliding friction linear velocity is maintained at 0.54m/s, the test time is fixed at 30min for each group, the applied loads were 150N and 200N in sequence, and the ambient relative humidity is 50±5%. All tests are dry friction test conducted at room temperature.

The friction coefficient μ of the material during the test can be calculated by reading the friction torque data on the tester and using the following formula:

$$\mu = M/(R \times P) \quad (1)$$

Where, M – friction torque in the formula, N * m; R – pair steel ring radius, m; P – imposed load, N.

After the end of the test, the wear depth of the carbon fiber fabric/epoxy resin composite is measured by using a digital micrometer with an accuracy of 0.001 mm. The friction wear life of the composite is calculated by dividing the sliding friction distance by the wear depth, and used to characterize the wear resistance of the material itself:

$$W = (V \times t)/\Delta H \quad (2)$$

Where, V – friction line speed, m/s; t – friction test time, s; ΔH – wear depth, μm.

A small amount of nano-TiO_2 is added to anhydrous ethanol for 30 minutes to fully be dispersed. A few drops of the suspension are sucked with a dropper and carefully dropped onto the surface of a copper mesh covered with a support film. After the solvent is dried, the surface morphology is observed under JEOL-2010 high-resolution transmission electron microscope (HRTEM).

The structure of nano-TiO_2 is characterized by D8 ADVANCE X-ray diffractometer (XRD) manufactured by Brux, Germany. The sequential notation scan method is used, where the tube voltage is 50 kV, the tube current is 40 mA, the scanning angle 2θ is from 10 to 90°, and the scan rate is 10°/min.

After the end of the test, the surface of the worn test specimen is sprayed with gold, placed under a JSM-5610LV scanning electron microscope for morphology analysis, where the acceleration voltage is 20 kV.

3. Experimental part

3.1. Structural analysis of nanofillers

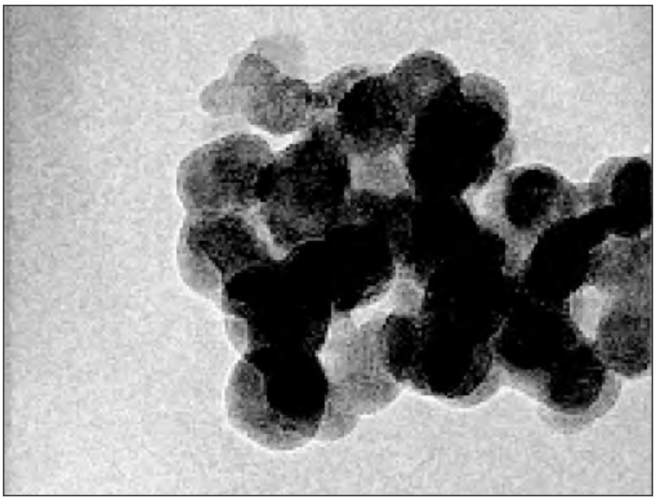
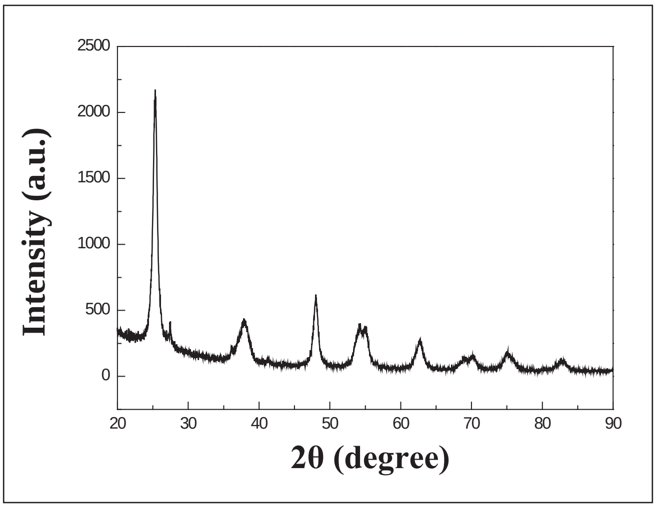
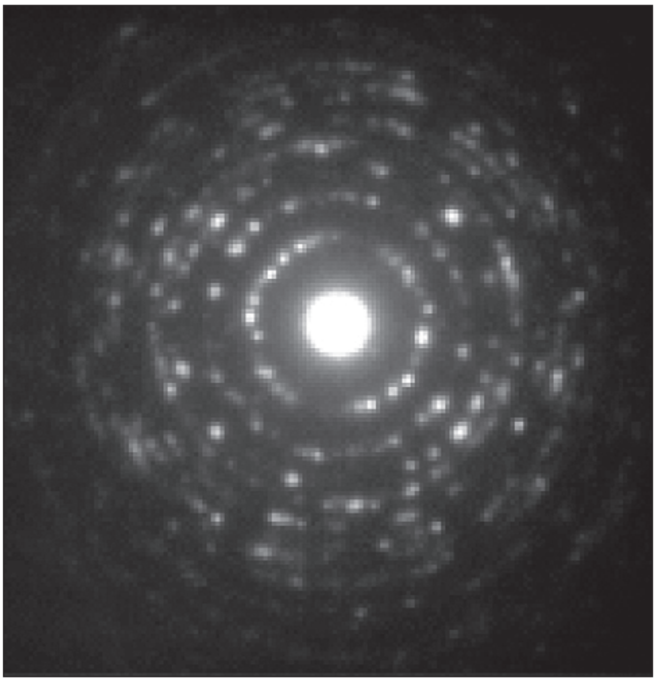


Fig. 1: TEM images of TiO_2 particles

It can be seen from Fig. 1 that the selected nano-TiO_2 particles have a relatively regular shape, most of which are between 20nm and 30nm in diameter, and the three-dimensional dimensions are within the nano-scale. XRD patterns and electron diffraction photographs (ED) of nano-TiO_2, are respectively shown in Figs. 2(a) and 2(b).



(a) X-ray diffraction spectrum of TiO_2



(b) ED diagram for TiO_2
Fig 2. The crystal structure of TiO_2 particles

As can be seen from Fig 2(a), there is a prominent main diffraction peak at $2\theta = 25.5°$ for nano-TiO_2. There are relatively strong characteristic diffraction peaks respectively at $2\theta = 38.05°$ and $2\theta = 48.06°$. Various characteristic peaks are generally more sharp, which proves that the nano-TiO_2 particles belong to the anatase TiO_2 with good crystallinity. At the same time, the characteristic bands of polycrystalline diffraction rings can also be seen in Fig. 2.(b), indicating that the selected nano-TiO_2 belongs to a crystal structure with good purity.

3.2 Friction and wear behaviour of carbon fiber fabric/epoxy resin composites filled with nano-TiO_2

Fig. 3 shows the change of friction coefficient of carbon fiber fabric/epoxy resin composite material under test conditions of 150N and 200N after nano-TiO_2 filling alone.

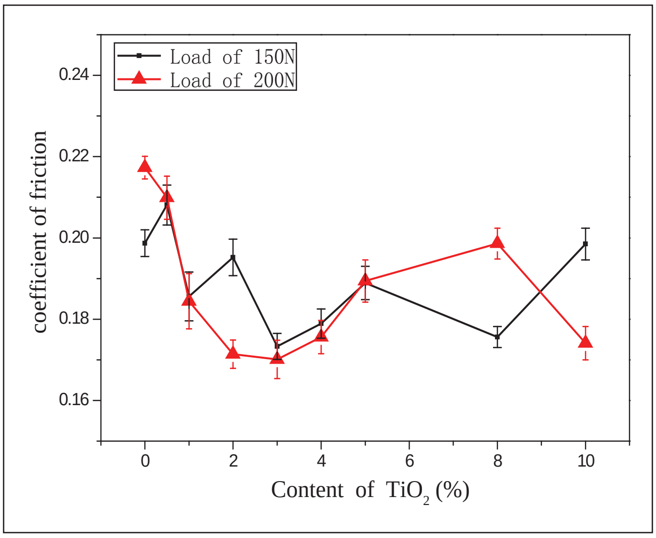
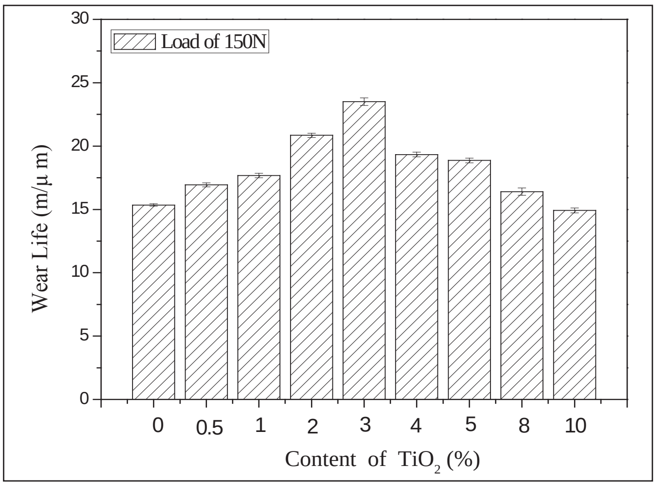


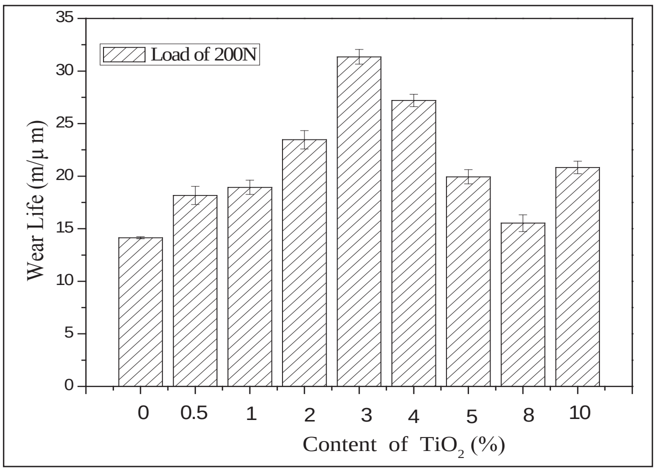
Fig. 3: Effects of addition amount of nano-TiO_2 on the friction coefficient of CF/EP composites under different loads

It can be seen from the figure that the friction coefficient of the pure carbon fiber fabric/epoxy resin composite is stable between 0.18 and 0.23, and the friction coefficient increases slightly with the increase of the load; under the condition of 150N load, the addition of different proportions of nano-TiO_2 has little effect on the friction coefficient of the composites. The friction coefficient shows a fluctuated trend with the increase of the amount of filler added. When the amount of filler added is 3%, the friction coefficient of the composite material reaches the lowest value of 0.1733, which is 12.78% lower than that of the pure carbon fiber fabric/epoxy resin composite; under 200N loading, the friction coefficient of composites first decreases and then increases with the increase of nano-TiO_2 addition. When the filler addition ratio is 3%, the friction coefficient of the composite material takes the lowest value of 0.1701. Compared to the pure material, the friction coefficient is reduced by 21.72%. And the improvement effect is significant.

Figs. 4(a) and4(b) respectively show the relationships of friction and wear life of carbon fiber/epoxy composites under test conditions of 150N and 200N after nano-TiO_2 filling.



(a) Load of 150N



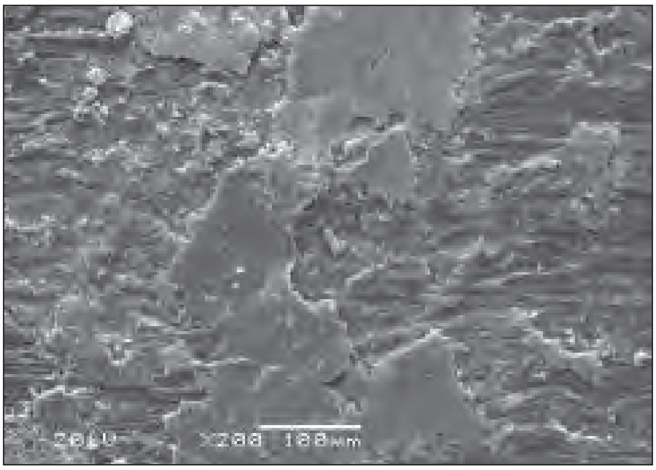
(b) Load of 200N

Fig 4. Variation of wear life of CF/EP composites with different nano-TiO_2 contents under

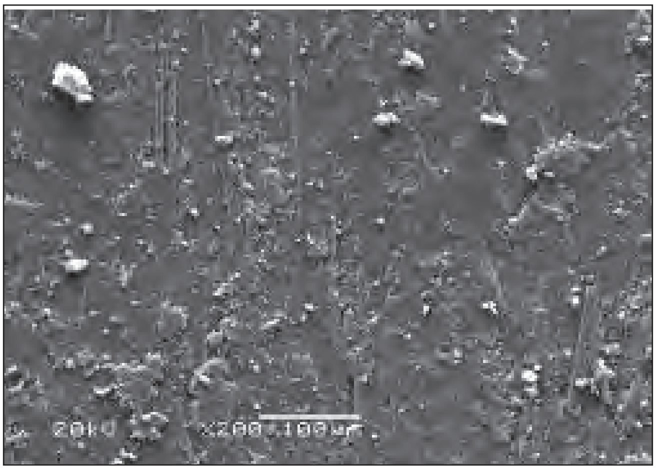
From Fig. 4(a), it can be seen that under 150N load conditions, with the increase of the content of nano-TiO_2 added, the friction and wear life of CF/EP composites first increases and then decreases. When the filler loading is 3%, the friction wear life of the modified composite is 23.51m/μm, which is 53.22% higher than that of the unmodified CF/EP material. When the content of nano-TiO_2 is more than 3%, the wear properties of the composites begin to deteriorate.

When the load condition is increased to 200N, as shown in Fig. 4(b), the change trend of the wear life of the composite material is larger than that of the 150N load condition, and the overall trend of the curve appears more "steep"; with the increase of the content of nano-TiO_2 from 0% to 3%, the tribological wear life of the composite changes from 14.15 m/μm to 31.35m/μm, which increases 121.55%; among them, when the load increases from 150N to 200N, the enhanced wear life of the pure CF/EP material is reduced due to the external force shearing. The friction and wear life of CF/EP composites modified by nano-TiO_2 slightly increased. It can be speculated that the anti-wear enhancement effect of nano-TiO_2 on CF/EP substrates can be fully exerted under higher loading conditions.

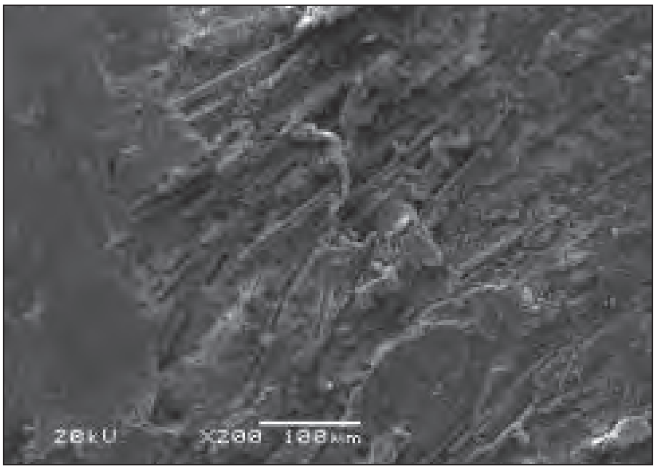
Scanning electron microscopy is used to further observe the wear morphology of unmodified CF/EP materials and CF/EP composites modified by 3% nano-TiO_2 respectively under the condition that loads are at 150N and 200N, as shown in Fig. 5.



(a) 0% TiO_2 + CF/EP under load of 150N



(b) 3.0% TiO_2 + CF/EP under load of 150N



(c) 0% TiO_2 + CF/EP under load of 200N

(d) 3.0% TiO_2 + CF/EP under load of 200N

Fig 5. (a-d) SEM micrographs of the worn surfaces of composites

From Fig. 5(a), it can be seen that a deep groove-like scratched appears on the wear surface interface after a certain period of friction and wear tests on unmodified CF/EP substrate materials under a load of 150N. The bulk of the binder resin phase is destroyed and exfoliated. And the only part of the resin substrate expressed severe extrusion plastic deformation, and are intermittently distributed like islands on the surface of the material, and made many cut fibers exposed. Brittle flaking and fatigue wear morphology are expressed on the whole; after being filled with 3% nano-TiO_2, the peeling of the adhesive resin on the worn surface was effectively suppressed, the wear scar is relatively shallow, and a large amount of fiber is not exposed. As shown in Fig. 5(b), when the load is increased to 200N, the unmodified CF/EP matrix material is subjected to a more severe shearing action. A large number of fibers in the surface layer are pulled out and cut off, and the interface is uneven, as shown in Fig. 5(c) which expresses a more severe fatigue wear pattern; Fig. 5(d) shows the wear morphology of CF/EP composites modified by 3% nano-TiO_2 under 200N load. It can be seen that the structure of the material remains better. The surface layer is scattered with some clusters of abrasive particles, which, however, did not cause severe plough marks. The overall surface is relatively flat, showing the morphology of patchy and adhesive transfer along the sliding direction.

From the above characteristics, it can be seen that the epoxy resin in the surface layer of the unmodified CF/EP material is brittle. Under the effect of the shearing force of the mating part, it is prone to cracking and peeling, and the fiber reinforced layer in the depth of the surface is also partially pulled out and cut off. Because the abrasion resistance of carbon fiber is much better than that of epoxy resin, it can be inferred that the structural component that plays a major role in the friction process is still the carbon fiber fabric layer. However, although carbon fiber has the anti-wear effect and self-lubrication due to its high strength and high mode, it is a material with high brittleness. In the harsher friction environment, once the carbon fiber wears thin, it is easy to generate high-hardness debris residue, which in turn causes severe abrasive plunge cutting of the epoxy substrate. Until the carbon fiber begins to break, it will also mean that the anti-wear effect of the material loses efficacy.

After adding a certain amount of nano-TiO_2, due to the small size effect and dispersion strengthening effect of nano-particles, the friction and wear properties of CF/EP materials have been correspondingly improved. When the filling content of nano-TiO_2 is 3.0%, the CF/EP substrate has a better friction reduction effect. However, as a whole, with the increase in the amount of filler added, the friction coefficient still shows a tendency to change, and there are still some brittle exfoliation pits on the worn surface. It is speculated that this may be related to the agglomeration and loss of nano-TiO_2 fillers in CF/EP substrates.

4. Conclusions

a. Filling the carbon fiber fabric/epoxy composite with an appropriate content of nano-TiO_2 can play a good role in friction reduction and wear reduction. The optimal addition amount of nano-TiO_2 is 3.0%. What is more, under higher load conditions, the composite material has a lower friction coefficient and higher wear life.

b. Unmodified carbon fiber fabrics/epoxy composites mainly express wear patterns of fatigue wear and brittle exfoliation. With the increase of the loading amount of nano-TiO_2, the wear patterns of materials gradually change to the adhesive wear and abrasive wear.

Acknowledgements

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