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Wear behavior of Al(OH)$_3$-GF/epoxy composites in low velocity

Guirong Peng · Lijun Gao · Zaiji Zhan · Haifeng Wang · Wenguang Hao

Abstract Effects of fiber content, size, and weave form, and addition of particles on wear behaviors of epoxy composites are studied widely, while little investigation is paid on thermal effect in friction. In this study, effects of Al(OH)$_3$ powder on wear behavior of glass fiber reinforced epoxy composites are investigated. The experimental results show that within 6 wt%, the addition of Al(OH)$_3$ powder could decrease the friction surface temperature, friction coefficient, and wear mass loss of the composites. The decrease is attributed to the heat absorption when Al(OH)$_3$ powder decomposes. However, when the content of Al(OH)$_3$ powder increases to 9 wt%, the temperature, the friction coefficient, and the wear mass loss increase to nearly equal to those of pure epoxy resin-based composites. It is considered resulting from the decrease in mechanical property, which could lead to more serious fatigue wear. In a word, within a proper content, the addition of Al(OH)$_3$ powder in epoxy could increase the resistance to wear and friction.

Keywords Sliding wear · Polymer-matrix composite · Thermal effects · Al(OH)$_3$ · Epoxy · Tribophysics

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Introduction

Epoxy composites are a kind of materials with high strength and modulus, and show good corrosion resistance to most chemical reagents. However, their low resistance to wear limits their wider use. Lots of studies on the wear behavior of epoxy composites are concerned with effects of various kinds and weave form of reinforced fibers [1–7], and sliding condition [8] on the tribology behavior and wear mechanism of composites. Other investigations focus on the addition of rubber and graphite powder [9], UHMWPE [10], MoS_2 [11, 12], coordination of TiO_2 with short carbon fibers, Aramid and PTFE particles [13], nano particles [14–19], and carbon nanotube [20], and most of the results demonstrate that most of the inorganic particles can increase the wear resistance of the composites by increasing the mechanical properties and hardness [14–20], or by providing lubricating effects and decreasing the transfer of epoxy to counterpart [11–13]. While little investigation is on the thermal effects in friction, although the thermal effects in friction have serious influence on the tribology behavior of the epoxy composites for their low thermometric conductivity. We found in our experiments that typical flame retarder, $Al(OH)_3$ powder can decrease the wear of composites, but the mechanism of the wear resistance has not been comprehensively studied. In this article, typical flame retarder, $Al(OH)_3$ powder is added into the glass fiber (plain weave) reinforced epoxy composites (GF/EP) to study the effects of $Al(OH)_3$ on the temperature of the friction surface and thus on the wear resistance of the GF/EP composites.

Experimental

Average diameter of $Al(OH)_3$ powder is 19 μm with purity of 99%. The powder is used as received. The surface of the $Al(OH)_3$ powder is organic treated and the surface FTIR spectrum is shown in Fig. 2.

Al(OH)$_3$ powder was dried at 100 °C for 48 h and then was mixed with Bisphenol A of E-51. The mixture is mechanically stirred for 20 min, and then stirred with ultrasonic (100 kHz) for another 20 min. Then cure reagents of methyl tetrahydrophthalic anhydride and 2-methyl-4-ethyl imidazole were added into the mixture in weight ratio to epoxy resin of 80 and 1%, respectively. The mixture of epoxy resin was degassed under ultrasonic before preparing the composites.

The composites were prepared by hand lay up in mold of 150×240 mm and then cured in hot-press. The cure process is shown in Table 1.

Table 1 Cure process of GF/EP composites filled with different contents of micro-$Al(OH)_3$ particles

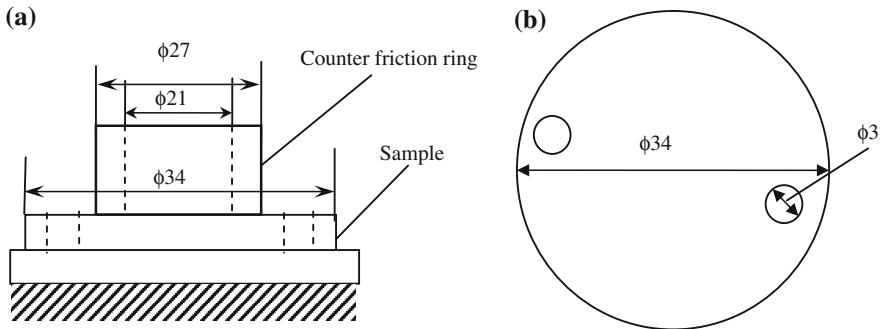| Cure temperature (°C)	Time (h)	Pressure (MPa)
90	1	3
130	2	5
150	2	5
180	2	0

(a)

(b)

Fig. 1 Schematic of the experimental set up (**a**) and the samples (**b**)

Low velocity friction was carried out on a ring-on-disk dry friction-wear tester of MMU-5G of Ji Nan Hua Yi Co. LTD with sample of Φ43 mm × 3 mm at 25 °C with humidity of 50% RH for 3 h. The load is 300 N and the friction velocity is 0.48 m/s. The counter friction ring is Φ27 mm and made of quenched 45# steel. Schematics of the experimental set up and the samples are shown in Fig. 1. The counter friction ring and the samples are treated with number 900 sand paper and washed with pure alcohol and ultrasonic.

Three point bending is carried out with samples of 60 mm × 15 mm × 2 mm, span distance of 40 mm and load rate of 2 mm/min. One data point is average of 6–10.

Surface morphology was observed with a KYKY-2800 SEM. A layer of Au was deposited on the surface of the samples. IR spectra were acquired with KBr pellets on Fourier transform infrared (FTIR) spectrophotometer of E55+FRA106 produced by BRUKER of Germany. The resolution of the spectrophotometer was 0.5 cm^{-1}.

DSC (STA449C of German NETZSCH) is carried out from room temperature to 700 °C in Ar air protection. The heating rate is 10 °C/min. Sample's weight is about 10 mg.

Results and discussion

Figure 2 shows the FTIR spectra of the samples with various content of Al(OH)$_3$. It can be seen that, the absorption of benzene ring and carbonyl between 1,500 and 1,750 cm^{-1} is changed little. Referred to the absorption at 1,510 cm^{-1}, the absorption between 1,000 and 1,100 cm^{-1} increases in intensity and new absorption at 1,090 cm^{-1} appears, which is not the absorption of Al(OH)$_3$. The absorption for Al(OH)$_3$ is at 1,020 cm^{-1}. While for Al(OH)$_3$, absorption of hydroxyl group between 3,400 and 3,660 cm^{-1} is strong, there is no obvious change for the sample with 6 wt% Al(OH)$_3$ in the FTIR spectrum between 3,300 and 3,650 cm^{-1}. All of these results indicate that the hydroxyl groups on the surface of Al(OH)$_3$ react with epoxy and form new ether group. However, when the content of Al(OH)$_3$ increases to 9 wt%, the absorption between 1,000 and 1,220 cm^{-1} changes insignificantly, but the absorption of hydroxyl of Al(OH)$_3$ is superposed on the epoxy and the
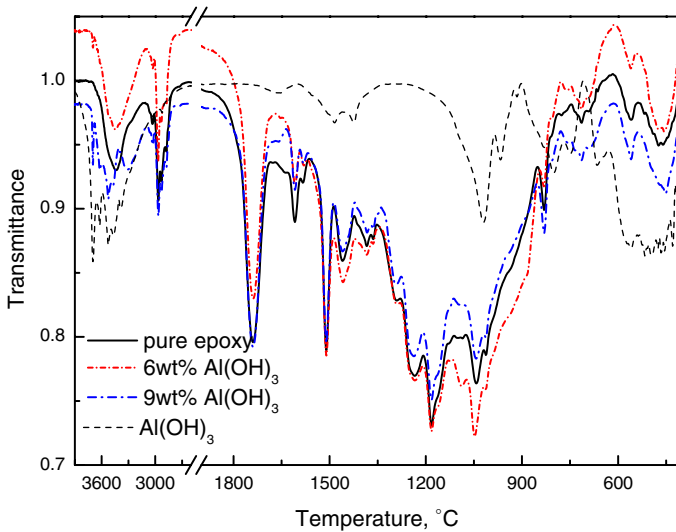
Fig. 2 FTIR spectra of Al(OH)₃ powder and GF/EP composites with various content of Al(OH)₃

absorption between 3,300 and 3,650 cm^{-1} is changed, which demonstrates that Al(OH)$_3$ could not react with epoxy to form new ether group, in other words, the compatibility of Al(OH)$_3$ with epoxy would decrease when the content of Al(OH)$_3$ increases to 9 wt%.

Figure 3 shows the DSC-TG plots of the composites with various content of Al(OH)$_3$. It can be seen that the decomposition temperature is increased with the addition of the Al(OH)$_3$, but changes erratically with the content of Al(OH)$_3$. The DSC plots show a significant peak at 385 °C, and the peak changes little with the variation of the content of Al(OH)$_3$ for all of the samples. However, there is a
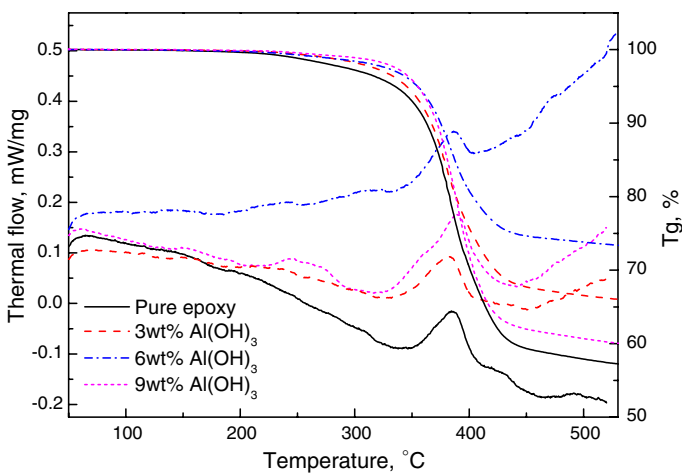

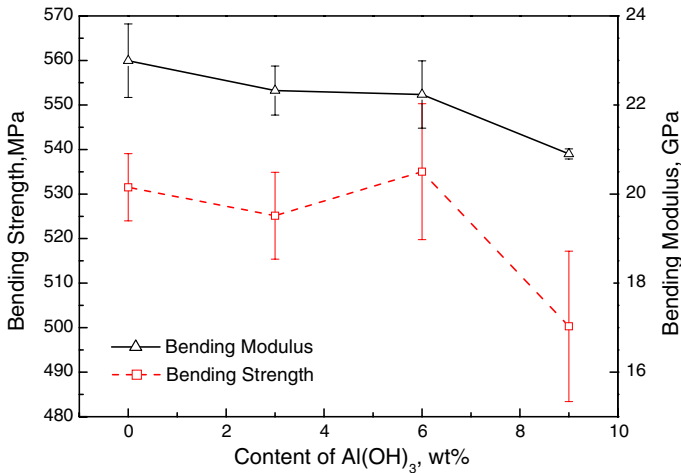**Fig. 3** DSC-TG curves for the GF/EP composites with various content of Al(OH)₃

Fig. 4 Bending strength and modulus of the GF/EP composites filled with various contents of micro-Al(OH)$_3$ powder

significant inflection between 200 and 300 °C in the DSC curves of the samples with Al(OH)$_3$, especially for the sample with 9 wt% Al(OH)$_3$, which might be for the decomposition of Al(OH)$_3$ to release water [21].

Figure 4 shows the bending strength and modulus of the GF/EP composites with different contents of Al(OH)$_3$ powder. The bending strength and modulus for the composites change insignificantly for the content of Al(OH)$_3$ within 6 wt%, and then decrease with the further increase of content of Al(OH)$_3$. When the content of Al(OH)$_3$ is higher than 6 wt%, the homogeneous dispersion is difficult for the higher viscosity, and block of Al(OH)$_3$ powder would act as weak points and result in decrease in strength.

Figure 5 shows the change of friction coefficient and friction surface temperature with content of Al(OH)$_3$. The average friction coefficient is calculated from 3,600 s to the final. It can be seen that within 6 wt%, the temperature and friction coefficient decrease with the content of Al(OH)$_3$, but increase to nearly a little higher than that of pure epoxy-based composite for the sample with 9 wt% Al(OH)$_3$. The curves of the average temperature and friction coefficient versus content of Al(OH)$_3$ particles give nearly same trends. Usually, the increase in temperature is resulted from friction thermal effects, thus the decrease of friction coefficient would certainly lead to the decrease of the surface temperature, and vice versa. The adhesive between the friction surfaces will increase the friction coefficient and produce wear debris, which would become more significant with the increase of the surface temperature.

Figure 6 shows the change of wear mass loss of GF/EP composites with content of Al(OH)$_3$. The wear mass loss is decreased, too, when the content of Al(OH)$_3$ is not higher than 6 wt%. However for the composite with 9 wt% Al(OH)$_3$, the wear mass loss is increased and even a little higher than that of pure epoxy composite.

The heat in micro zone on friction surface might be higher than hundreds of centigrade. The heat could result in mechanical property of composites decreased
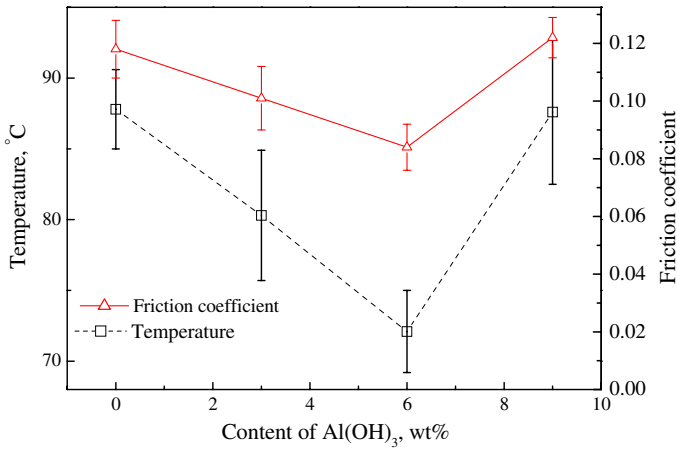
Fig. 5 Average friction coefficient and temperature of GF/EP composites versus contents of Al(OH)$_3$ powder
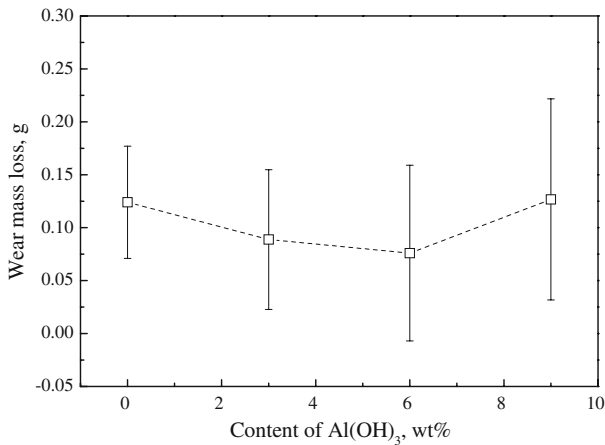

Fig. 6 Wear mass loss of GF/EP composites filled with micro-Al(OH)$_3$ powder

seriously and thus result in serious wear and increase of friction coefficients. However, Al(OH)$_3$ powder can begin to decompose at 200 °C [21] and lots of heat will be absorbed, therefore, for the composites the temperature on the surface could be decreased, and then the resistance to wear and friction would be increased. The DSC-TG curves do indicate the absorption (Fig. 3). However, when the content of Al(OH)$_3$ is higher than 6 wt%, the decrease in strength and modulus could eliminate the effects of temperature and even result in more seriously abrasive wear.

In the experiments, it is observed that lots of wear debris were produced on the surface and the wear debris gradually changes to smaller particles with the duration of friction. Some spots of transfer film were observed on the counter friction ring. All of these phenomena indicate that it is furrow friction at the beginning and then
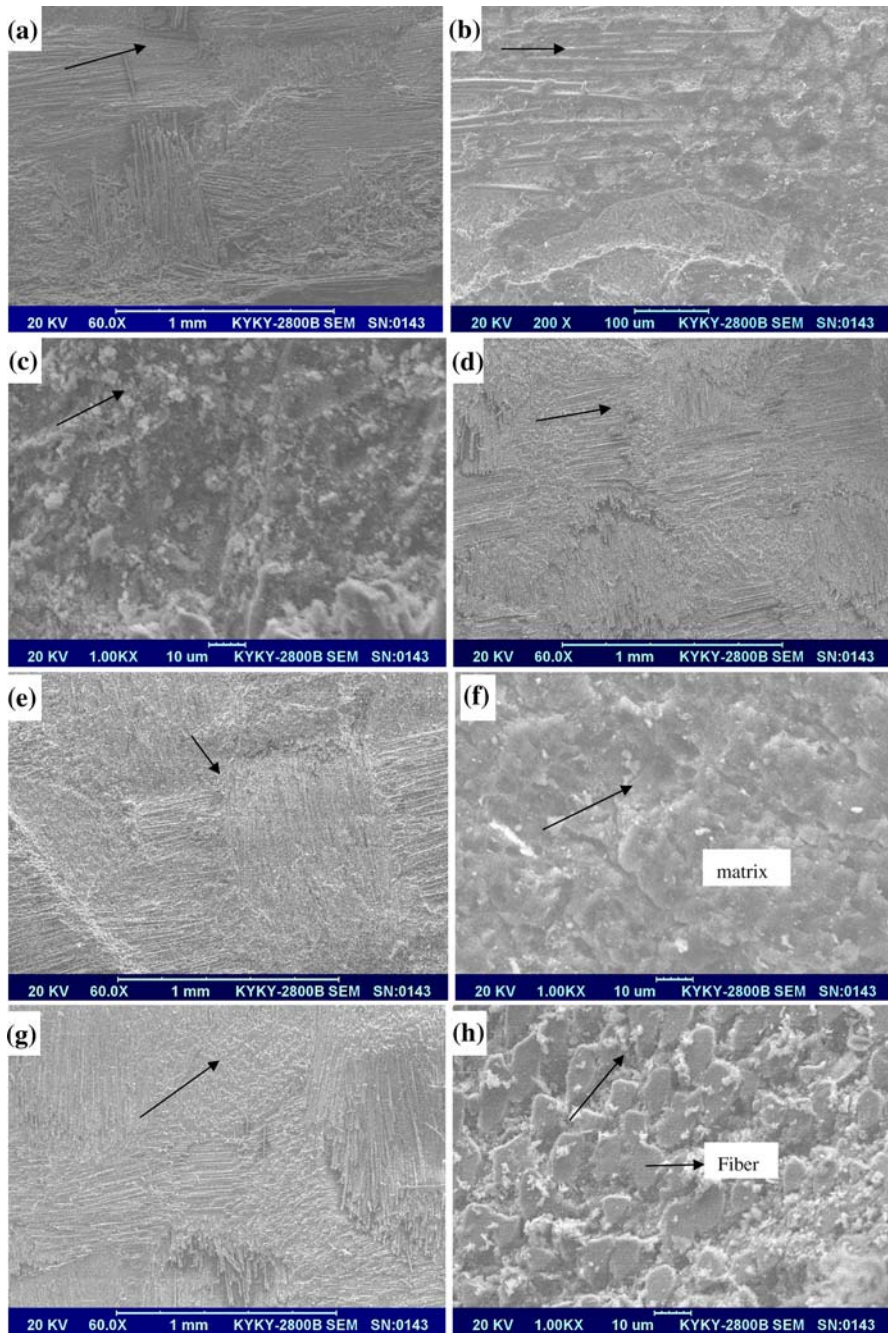
Fig. 7 SEM photographs of the worn surface of GFRE composites filled with different content of micro-Al(OH)$_3$. **a**, **b** and **c** GF-pure EP. **d** 3 wt% Al(OH)$_3$-GF/EP, **e** and **f** 6 wt%Al(OH)$_3$-GF/EP, **g** and **h** 9 wt% Al(OH)$_3$-GF/EP (The indicator indicate the sliding direction)

adhesive friction predominates in the wear mechanism. The surface of the pure epoxy-based composites (see Fig. 7 a, b, c) is typical adhesive wear morphology. Moreover, there are lots of concaves on the friction surface, part of which are long and round and might result from the fibers which are pulled out from the matrix. The other irregular concave might result from the fatigue breaking of the resin and fibers.

When increasing the content of $Al(OH)_3$ powder, the morphology changes are not obviously compared with those of the pure resin composites (see a, d, e, g in Fig. 7). However, more obvious cracks and concaves from fiber pulling off can be seen on the surface of the composites with 6 wt% $Al(OH)_3$ powder(f in Fig. 7). For the composites with 9 wt% $Al(OH)_3$ powder, lots of small particles can be seen on the surface, and the resin is abraded more seriously and even the breaking surface of the fiber is also changed to lean to friction direction (see h in Fig. 7). The particles might result from the $Al(OH)_3$ powder, which act as a kind of abrasive and result more serious abrasive wear. Furthermore, in previous section, the strength and modulus for the composites with 9 wt% $Al(OH)_3$ powder are decreased, which could lead to the decrease of fatigue strength and result in much serious fatigue wear. However, the effects of abrasive wear and fatigue wear only eliminate the heat absorbing effects, therefore, the friction coefficient and wear mass loss are nearly equal to that of pure epoxy composites.

Conclusion

1. When the content of $Al(OH)_3$ powder is not higher than 6 wt%, the bending strength and modulus are changed not obviously, but when the content of $Al(OH)_3$ powder is higher than 6 wt%, the bending strength and modulus decrease obviously, which is considered resulting from the compatibility of $Al(OH)_3$ with epoxy.
2. Within 6 wt%, the addition of $Al(OH)_3$ powder could decrease the friction surface temperature, friction coefficient, and wear mass loss. The decrease is considered for the absorption of heat by decomposing of $Al(OH)_3$ powder, which decreases the temperature and then increases the resistance of the composite to wear and friction.
3. When the content of $Al(OH)_3$ powder increases to 9 wt%, all of the temperature, friction coefficient, and wear mass loss increase to nearly equal to that of pure epoxy resin-based composites, which is considered resulting from the more serious fatigue wear and more serious abrasive wear.

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