

Friction behaviors and wear mechanisms of multi-filler reinforced epoxy composites under dry and wet conditions: Effects of loads, sliding speeds, temperatures, water lubrication

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ABSTRACT

Polymer composites have been extensively used as marine engineering structures under extreme environmental conditions for their mechanical/thermal stability and special self-lubricating performance. In the present paper, the multi-fillers reinforced epoxy composites (MFREC) had been prepared and analyzed. The mechanical, thermal properties, friction behaviors and wear mechanisms of MFREC were systematically investigated. The research results showed that the increase (98.46%) of MFREC tensile toughness was mainly attributed to improvement of tensile strength (9.56%), tensile modulus (22.07%), and elongation at break (39.09%) due to multi-fillers reinforcement and its compatibility with the epoxy matrix. Then, increases in loads and temperatures reduced frictional coefficients at the expense of wear rate and line roughness of MFREC due to the change in contact state (from elastic to plastic state). Additionally, effects of sliding speeds and water lubrication on MFREC were not obvious compared to loads and temperatures owing to the formation of transfer layer and lubrication of water film. Furthermore, among the several influencing factors studied for tribological changes, MFREC was the most sensitive to the load effect due to the higher shear force of applied-load steel-ball by a tangential displacement, temperature second, sliding speed third and water lubrication last because of the lubricating effect and heat dissipation function of water film.

1. Introduction

In the recent years, with the depletion of continental energy sources, the scientific and technological powers have taken the development of marine economy as a new strategic priority. However, the ocean has severe environmental conditions such as scouring of seawater splashing zone, high loads, high wear and even high temperature, which puts forward higher requirements for the applicability of marine engineering materials [1,2]. Furthermore, friction and wear is a widespread phenomenon in engineering equipment, which forces the development of materials with better tribological characteristics [3]. Additionally, the wear and corrosion of metal materials are extremely prominent, which greatly reduces the long-term life of marine engineering structures [4,5]. Therefore, there is an urgent need for high performance composites with lightweight, high strength, friction and wear resistances.

Epoxy resin shows excellent applicability owing to its outstanding mechanical, adhesive, and insulated properties in civil engineering, automotive manufacturing and aerospace [6,7]. This is because its molecular structure contains a variety of polar groups and large active epoxy groups, which has a strong interfacial bond with a variety of polar materials such as metals, cement, wood and plastics [8,9]. However, the low fracture toughness and poor anti-wear characteristics of epoxy resin obviously limit their development in harsh tribological service environments [10,11]. Furthermore, the addition of nano-/micro-inorganic particles into the epoxy can effectively enhance the mechanical and tribological properties of composites by improving the particle distribution and utilizing the coupling effects between functional fillers [12, 13]. Therefore, the modification (mechanical and tribological performances) of epoxy resin is necessary for the preparation of high performance anti-wear composites. For example, Tian et al. [14] reported the

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Table 1

Physical and mechanical parameters of additives.

MFREC	Particle size (μm)	Tensile modulus (GPa)	Density (g/cm^3)
Nano-TiO ₂	0.3	182.0	4.23
Graphite	20.0	9.8	2.25
SCFs	7.0 (diameter) 60.0 (length)	240.0	1.70
ZnS	0.5	/	4.09

friction behavior and wear mechanism of the epoxy matrix before and after nano-particle TiO₂ modification. The results showed that the improvement of friction and wear resistances of composites was mainly attributed to the fact that the functional particles could improve the mechanical toughness by blocking the propagation direction of cracks in the resin, and then enhance the wear resistance. Additionally, for the tribological improvement of filler-modified epoxy resin (FMPE) according to Alajmi et al. [15] and Alrashdan et al. [16], the graphite filler could improve the anti-friction/wear performances by forming a third-body between the friction pairs, decreasing wear track roughness of composites. Li et al. [17] reported that CNTs/ZnS hybrid could significantly enhance the tribological properties of epoxy resin due to their great dispersion and synergism. However, the influence and coupling effects of external environmental conditions on tribological behavior of FMPE have been not analyzed and summarized. The tribo-responses of FMPE subjected to the applied loads, sliding speeds, elevated temperatures and water lubrication, as well as the associated wear mechanisms are not clear. Hence, it is a necessary step to establish the relation between the friction evolution behaviors and wear response mechanisms served for the above harsh environmental conditions.

With the deterioration of the different actual environments, the service characteristics and failure behaviors of FMPE are obviously different. During the wear process, the FMPE undergoes a series of changes such as creep→yielding→twisting→deformation, which eventually manifests itself in debris turnover or removal [18]. This mechanism of physical material removal constitutes an extremely complex thermo-mechanical process, which is a function of number of parameters such as the polymer matrix properties, functional filler types, environmental conditions and interfacial physical/chemical reactions [19,20]. In particular, the frictional contact may lead to the formation of a transfer film at the worn furrows, which ultimately plays a key role in determining the overall tribological behavior at the steel-ball/composite interface. Therefore, the characteristics of friction film have been considered as signature distinctiveness for particular fillers and resin matrix under different service environments [19]. In order to give clear semi-quantitative interpretation to the friction film, the concept of line roughness (LR) is introduced, which considers its morphologies along a given wear trajectory. This introduction of friction film roughness provides a new insight regarding the deformation of materials in tribo-contacts, which is beneficial for the understanding of friction behavior and wear mechanism of FMPE. In view of that, it is important to have a clear understanding of the friction characteristics and wear mechanisms of FMPE under high load, fast sliding speed, elevated temperature and water-lubricated friction applications.

So far, the research on the friction behavior and wear mechanism of FMPE in the harsh served environments is still limited. This is because the material performances (component structure and functional filler types) and external environmental factors (loads, sliding speed, temperatures and water lubrication) can affect or even control the friction behaviors and wear morphologies of FMPE. For example, Qi et al. [21] found that incorporating SCFs into PEEK/graphite composites degraded the tribological properties, and this was because the SCFs ploughed from the surface increased the roughness of the composites/counterpart interface, led to the severe friction and visible wear tracks in the friction pair. Chen et al. [22] reported that the CF/PEEK composite had better tribological performances under sea water lubrication than under dry

and pure water lubrication, because the presence of seawater would affect the chemical composition of the counter surface. Therefore, in order to establish a relation of material component and environment factors against friction coefficient and wear rate, we prepared a new composite through multi-filler reinforced epoxy-based matrix. Where, for multiple fillers, TiO₂ and SCFs were used as the mechanical reinforced phases due to toughening effect and mechanical skeleton, and graphite and ZnS were acted as frictional lubricated phases because of the formation and repair of dense friction film. The reciprocating friction and wear tests of the as-prepared composites were carried out under different loads, sliding speeds, temperatures and water lubrication conditions in order to explore its friction behaviors and wear mechanisms.

In the present study, the multi-fillers reinforced epoxy-based composites (MFREC) were prepared and analyzed combined with the mechanical reinforcement and frictional lubrication phases. The mechanical tensile, flexural, thermal and tribological tests under the harsh service conditions were systematically investigated to characterize the mechanical/thermal reinforcement and friction/wear resistance of MFREC. Furthermore, the microstructure tests (scanning electron microscope, super-depth microscope, line roughness) were comprehensively conducted to reveal the tribological evolution mechanism. Finally, the anti-friction sensitive factors and wear mechanisms of MFREC were analyzed and compared in details, which would provide a guideline for the actual applications.

2. Experimental details

2.1. Raw materials and sample preparation

Ts (Bisphenol A type) was used as the epoxy matrix, and the corresponding B component and HTDA (4-methylcyclohexane-1, 3-diamine) were selected as the curing agent, which ratio was 100:16.3:10.9. Epoxy resin (Dagong Composite Company (Linyi, Shandong, China)) had a density of 1200 kg/m³. Nano-TiO₂ and SCFs were produced by the Badu metal material Company (Hebei, China) and Zhongsen Pilot Technology Co. LTD (Shenzhen, China), respectively. Additionally, Graphite and ZnS were purchased at the Pipi Chemical Raw Materials Co. LTD (Qingdao, China) and the Nano Chemical Technology Co. LTD (Guangzhou, China), respectively. Some detailed parameters of the above four additives are shown in Table 1, including the size, tensile modulus and density. It was noted that the each optimal-filler formulation was selected based on the previous report [10], including 4 wt% TiO₂, 7 wt% graphite, 10 wt% SCFs, 5 wt% ZnS.

The fabrication procedure of MFREC is depicted in Fig. 1. Firstly, Nano-TiO₂ particles were added into ethanol solvent and stirred continuously for 5 min, then polyvinylpyrrolidone (PVP) dispersant (Tianjin Kemeiou chemical reagent co., LTD, China) was added and stirred for another 5 min, and Ts was added into the above solution and stirred by sonication for 30 min. After mixing evenly, the corresponding weights of graphite, SCFs and ZnS were added in turn, stirring for 6 h by a high-speed mechanical stirrer (JRJ300-SH, Shanghai Huxi co., LTD, China) with the speed of 3000 rpm. Secondly, to remove organic solvents from the mixture, the above solution was heated and stirred in an oil bath at 120 °C for 2 h. After cooling to the R.T., the corresponding curing agent was added and stirred at a speed of 5000 rpm for 5 min. Finally, the above solution was prepared by a three-roll grinding (ZYTR-120E, Shenzhen Zhongyi technology co., LTD), and multi-fillers in the mixture were finely dispersed by the friction force and extrusion pressure between rollers. The pre-treated solution was vacuumed again for 20 min to remove air bubbles. Mechanical, thermal and tribological samples were prepared by pouring the mixture into designed silicone rubber molds. All samples were pre-cured at R.T. for 24 h and then maintained at 60 °C for 24 h for post-curing.

It was noted that the feasibility and applicability of MFREC would be verified from the perspectives of mechanical, thermal and tribological

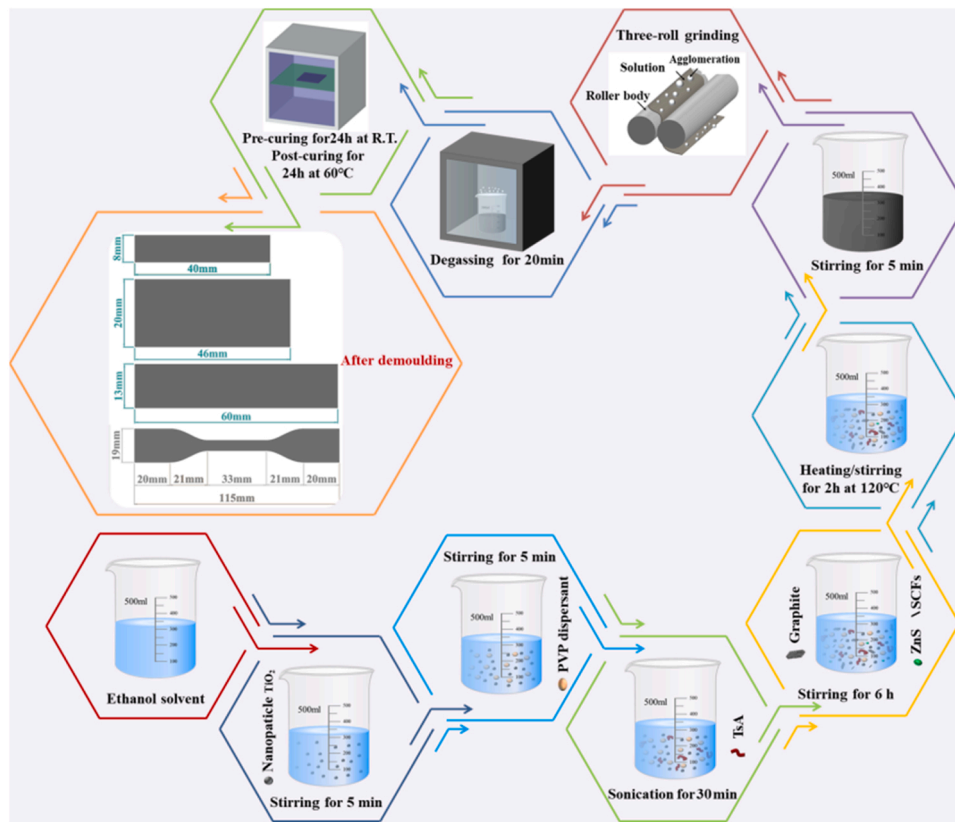


Fig. 1. Schematic procedure of MFREC fabrication.

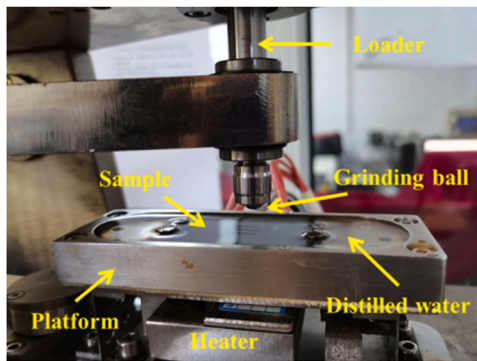


Fig. 2. Reciprocating friction and wear tester.

properties. Additionally, MFREC was enhanced in the two aspects compared to the conventional epoxy-based composites. Functional fillers could be divided into mechanical reinforced phase and frictional lubricated phase. Nano-TiO₂ and SCFs fillers have the effect of cracking resistance and three-dimensional skeleton support, respectively, which could improve mechanical toughness and strength-modulus of MFREC. The graphite particles have the excellent natural lubrication properties due to the C-C individual bond and relatively weak van der Waals forces between the laminates, and ZnS fillers can form the friction film on the wear track surface because of its weak stiffness and low shear strength, which could enhance friction and wear resistances of MFREC. Therefore, it was necessary to systematically study the friction behaviors and wear mechanisms of MFREC under harsh service environments to provide a guideline for the actual applications.

2.2. Mechanical property testing

Tensile tests of epoxy resin and MFREC were conducted through a universal tensile machine (DHY-10080, Shanghai, China) according to ASTM D-638–91. Samples were machined into dumbbell shapes before testing. The deformations of samples were measured by an extensometer at a tensile rate of 2 mm/min. Five samples were used to obtain the average value and standard deviation for each test.

Based on the ASTM D7264–07, the flexural tests of epoxy resin and MFREC were conducted through the same universal tensile machine. The rectangular size of the sample was determined to be 60 × 13 × 4 mm. The displacement rate of tensile machine grip was set to 2 mm/min. Five samples were repeated to obtain the average value and standard deviation for each test.

2.3. Thermal property testing

The thermal properties of epoxy resin and MFREC were characterized by the dynamic mechanical analysis (DMA-Q800, TA Instrument Co. Ltd., New Castle, DE, USA). The samples were conducted with the size of 40 × 8 × 2 mm. The clamping mode was the single cantilever beam with a loading frequency of 1 Hz and a heating rate of 5 °C/min, where temperature range was from R.T. to 150 °C. The average glass transition temperature (T_g) was obtained from two samples. In addition, the storage modulus and loss modulus were obtained to analyze the effect of fillers on the thermal performances of epoxy resin.

2.4. Friction and wear property testing

The reciprocating friction and wear machine (HRTA02, Jinan, China) is presented in Fig. 2. By measuring the volume change of MFREC before and after testing, the wear rate was obtained, where the formula is as follows:

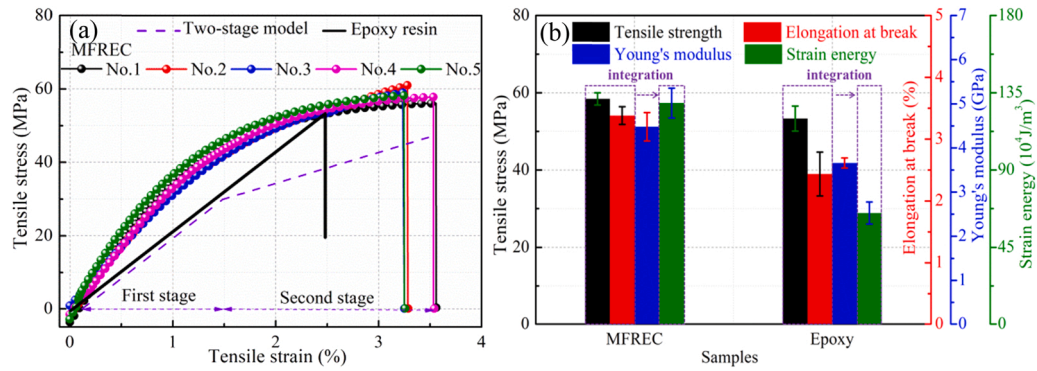


Fig. 3. Tensile performances of epoxy resin before and after modification: (a) stress-strain curves; (b) tensile parameters.

Table 2

Tensile and flexural performances of epoxy resin and MFREC.

Mechanical properties	Tensile performances			Flexural performances	
	Strength (MPa)	Modulus (GPa)	Elongation at break (%)	Strength (MPa)	Modulus (GPa)
Epoxy resin	53.33 (± 3.24)	3.67 (± 0.11)	2.43 (± 0.35)	93.34 (± 3.28)	3.18 (± 0.31)
MFREC	58.43 (± 1.59)	4.48 (± 0.32)	3.38 (± 0.14)	110.61 (± 4.02)	3.95 (± 0.24)

$$Ws = \frac{\Delta V}{lF} (mm^3/N \cdot m)$$

where ΔV is the volume change of MFREC before and after testing (mm^3); l is the friction distance (m); F is the normal loading (N).

The MFREC were installed in the wear machine tray, the harsh

service environments were set through controlling the loads, sliding speeds, temperatures and water lubrication. The applied loads were set to 500, 1000, 1500 and 2000 g, and the sliding speeds were applied to 60, 80, 100 and 120 mm/s. In addition, the service temperatures were divided into two types, dry and wet friction states, which were R.T., 60, 80, 100, 120 °C and R.T., 60, 80, 95 °C, respectively. The MFREC has dimensions of $46 \times 20 \times 3$ mm, and a steel-ball of 6.35 mm diameter was used as the counterpart, where the wear cycle and total sliding length were 10,000 and 100 m, respectively.

2.5. Morphology analysis

Scanning electron microscopy (SEM, VEGA3 TESCAN) was used to analyze the surface morphology of MFREC after tensile and friction tests at a test voltage of 20 kV. Samples were mounted on aluminum plates. Before testing, a thin gold layer was applied to the scanning surface as a

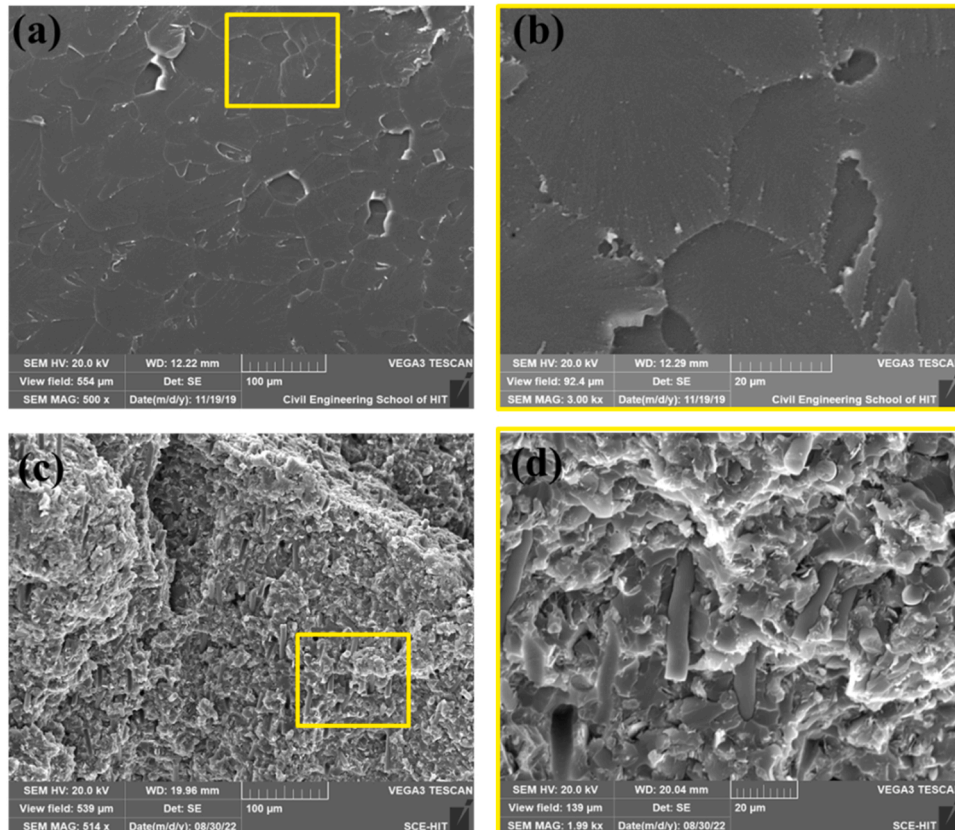


Fig. 4. Surface morphology analysis of the tensile fracture at the low and high magnification: (a) and (b) epoxy resin; (c) and (d) MFREC.

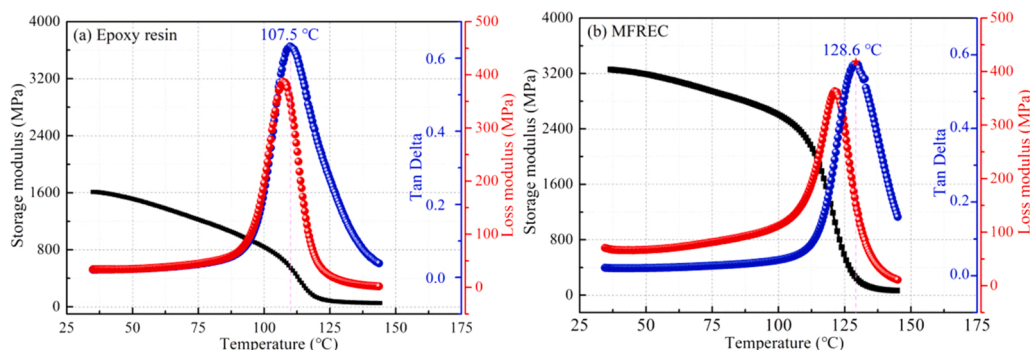


Fig. 5. Dynamic mechanical analysis of (a) epoxy resin and (b) MFREC.

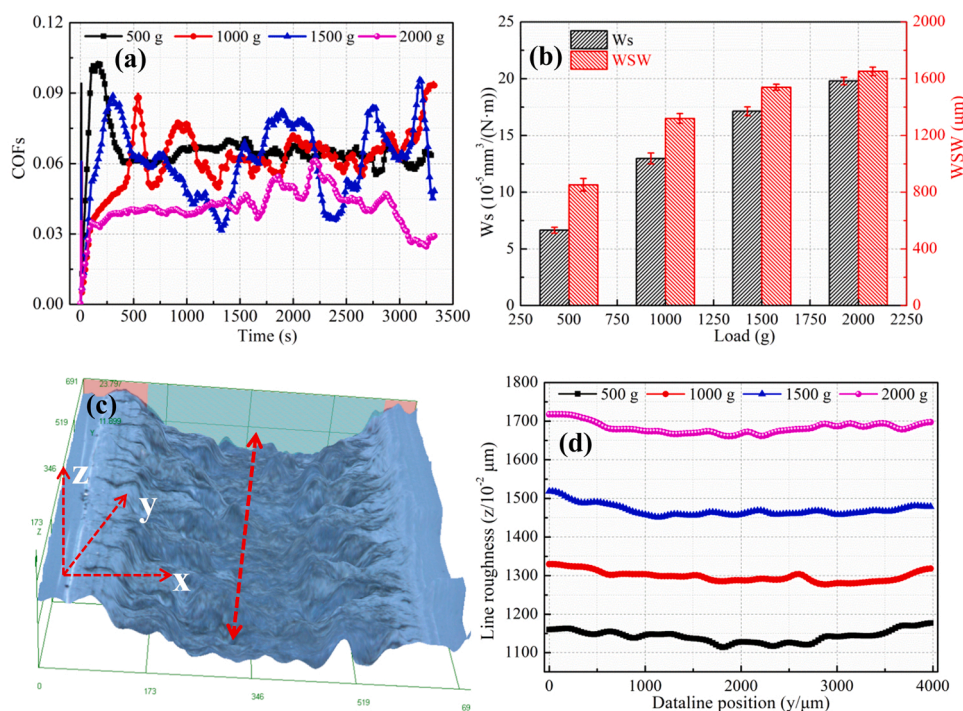


Fig. 6. Effect of the loads on friction behavior and wear rate of MFREC: (a) COFs; (b) Ws and WSW; (c) three-dimensional skeleton diagram of wear track; (d) LR along the sliding direction at the wear track.

conductive material using an E5200 automatic sputter coater (Cambridge, UK) and the test current and spraying time were 10 mA and 180 s, respectively.

Super-depth three-dimensional microscopy system (SDM, OLYMPUS DSX500) was used to photograph the MFREC and grinding ball surfaces after friction tests. The middle part of the wear track (~ 4.5 mm in length) was measured and analyzed. The width and length of the wear marks were obtained from the average of 10 test results for calculating specific wear rate (Ws), wear scar width (WSW), and line roughness (LR).

3. Results and discussion

3.1. Mechanical properties

Fig. 3 shows the tensile performances of epoxy resin before and after modification. As shown in Fig. 3(a) (stress-strain curves), MFREC presented the obvious two-stage model, namely the initial linear elastic stage (first stage) and the subsequent plastic deformation stage (second stage). In contrast, epoxy resin only had the linear elastic stage (black

solid line), and the failure mode was characterized by significant brittle fracture. This was because the thermosetting resin was a three-dimensional spatial network structure, which had the strong stiffness due to the molecular chain cross-linking of high-density, so the excessive deformation would lead to the sudden cracking of resin matrix. Based on the slope of linear elastic stress-strain curves, the tensile modulus of epoxy resin was determined to be 3.67 GPa. Its tensile strength and elongation at break were 53.33 MPa and 2.43%, respectively. The area enclosed was calculated by integrating the X-axis (strain) as the base and the stress-strain curve as the boundary, which was used to represent the tensile toughness (strain energy) of MFREC. Note that the tensile toughness values are simply magnitudes and has no physical meaning. Therefore, compared with the control sample, the tensile toughness of MFREC increased by 98.46% in Fig. 3(b), which was due to the increase of tensile strength (9.56%), tensile modulus (22.07%), elongation at break (39.09%) as well as their coupling effects. The improvement of tensile strength and modulus of MFREC could be attributed to the crack inhibition ability of nano-TiO₂ [23] and the high strength-modulus of SCFs [24], and the enhancement of elongation at break was due to the lubrication between the graphite layers [15] and mechanical

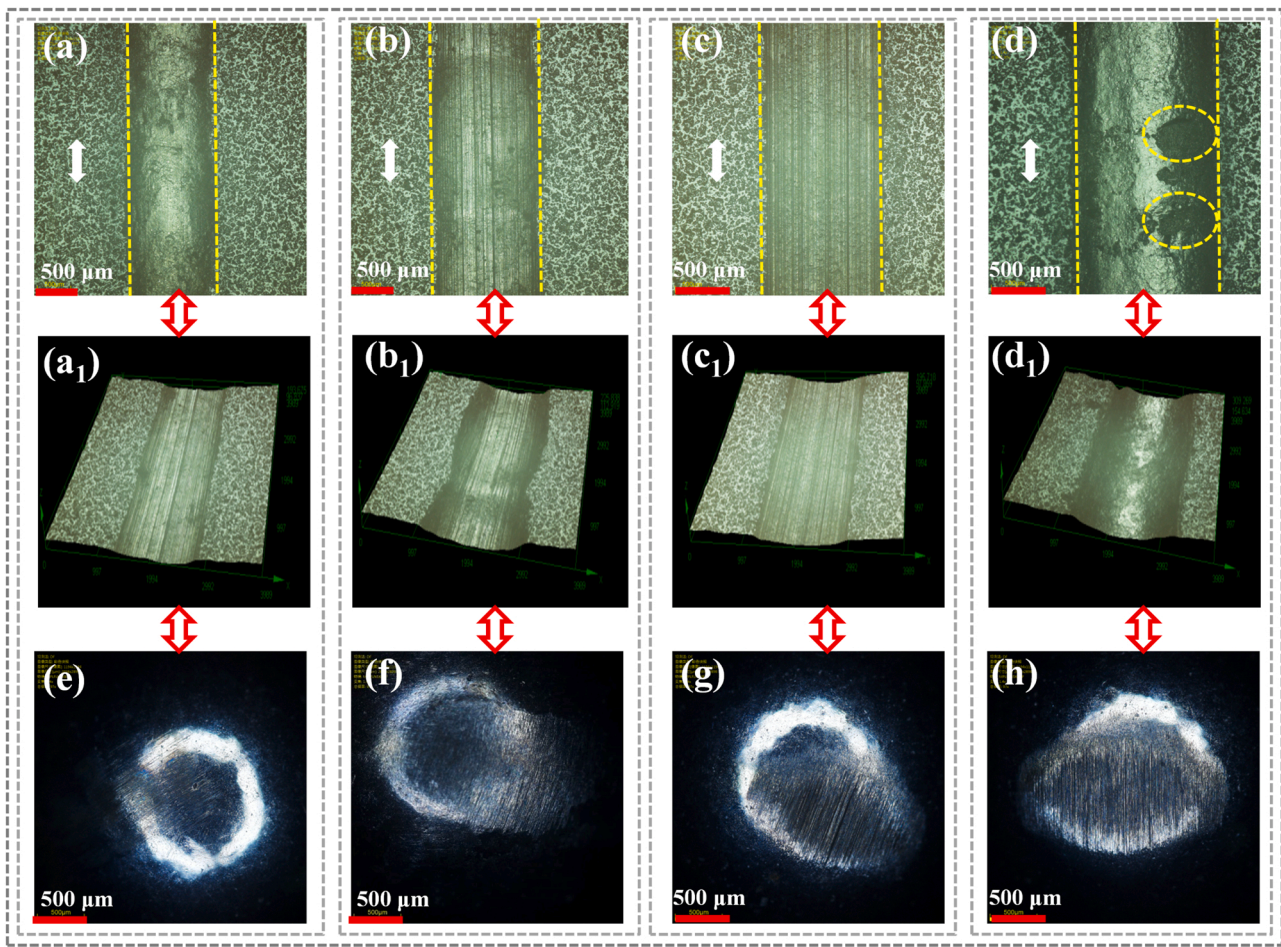


Fig. 7. Surface wear tracks and micro-profiles of MFREC and micro-morphologies of steel-balls under the different loads: (a) and (e) 500 g; (b) and (f) 1000 g; (c) and (g) 1500 g; (d) and (h) 2000 g.

interlocking of ZnS particles on SCFs/epoxy interface [10].

Furthermore, it was noted that the tensile stress-strain curves of five MFREC samples were similar (Fig. 3(a)), which indicated the better stability of experimental data and could be certified by a smaller standard deviation as shown in Table 2. Additionally, the flexural performances of MFREC had an obvious increase (18.50% for flexural strength, 24.21% for flexural modulus) owing to the addition of multiple functional fillers (Table 2), and the increase reason was similar to the tensile performances. In summary, the functional particles could improve the toughness of MFREC by enhancing the internal load transfer ability of epoxy matrix and control the crack initiation and propagation direction. Among the sensitive factors for enhancing toughness were, in order, elongation at break (tensile), modulus (tensile and flexural) and strength (tensile and flexural).

3.2. Improvement mechanism

Fig. 4 shows the effect of multiple functional fillers on tensile fracture morphologies of epoxy resin at the low and high magnification. As shown in Fig. 4(a) on epoxy resin, the fracture morphology presented the typical fish scale shape and some resin sheets showed the surface tearing, which indicated the obvious brittle failure characteristics. Furthermore, with an increase in magnification in Fig. 4(b), it can be more clearly observed that the tensile fracture of epoxy resin showed an obvious brittle fracture characteristic, which was an indication of small energy dissipation and could be used to explain that the stress-strain curve had only linear phase (Fig. 3(a)). Compared with epoxy resin, the tensile fracture morphology of MFREC had the significant textures

and grooves (Fig. 4(c)), which indicated that the propagation of the internal cracks was continuously blocked, forcing the cracks to expand from different planes to penetrate. Additionally, Fig. 4(d) shows the SCFs were uniformly dispersed in epoxy matrix and tightly wrapped by resin without the obvious debonding and particle agglomeration phenomena, which indicated the functional fillers could play a critical role in load transfer and load-bearing skeleton. It was noted the improvement of mechanical properties of MFREC was attributed to the coupling effects of the reinforced particles (TiO_2 /SCFs) and particles/epoxy bonding interfaces. Therefore, the tensile stress-strain curve of MFREC presented a two-stage failure model compared with epoxy resin (Fig. 3 (a)), which was responsible for the improvement of mechanical properties.

3.3. Dynamic mechanical analysis

Dynamic mechanical analysis can express the thermodynamic properties of epoxy resin before and after modification, such as storage modulus, T_g , etc [25]. After analyzing the mechanical properties, the effects of multiple functional fillers on thermodynamic properties of epoxy resin were obtained in Fig. 5. Before adding the fillers in Fig. 5(a), the maximum storage modulus and T_g of epoxy resin were 1600 MPa and 107.5 °C, respectively. Furthermore, as shown in Fig. 5(b), the storage modulus (30–100 °C) of MFREC had an obvious increase compared with epoxy resin, which was attributed to the interaction between functional particles and epoxy matrix. For example, the graphite fillers took precedence over epoxy matrix in energy absorption and reduced its sensitivity to temperature [16]; SCFs or TiO_2 particles

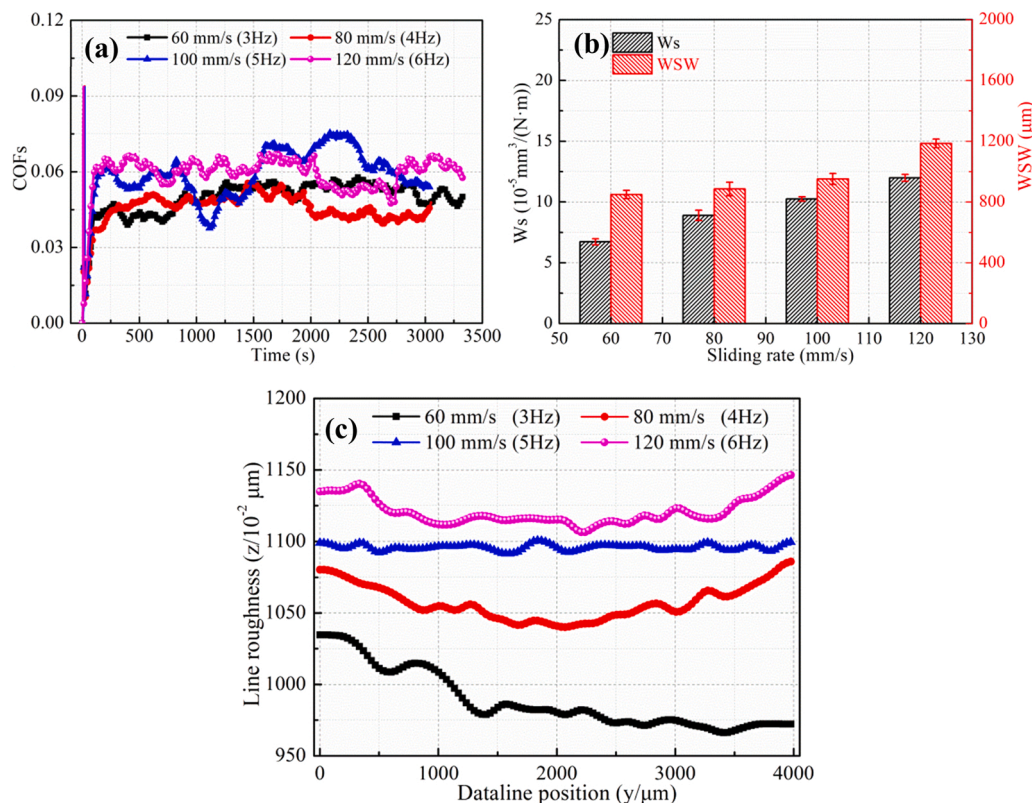


Fig. 8. Effect of sliding speeds on friction behavior and wear rate of MFREC: (a) COFs; (b) Ws and WSW; (c) LR along the sliding direction.

increased the stiffness of MFREC [26]; the hydrogen bonding between ZnS particles and epoxy matrix increased the crosslink density of MFREC [10]. It is well known that the peak temperature of tan delta curve is T_g . It can be observed that the T_g of MFREC increased by 19.6% compared with epoxy resin, and this was because the addition of nano-/micro-fillers can fill the micro-pores or micro-cracks within the epoxy matrix and increased the overall compactness. This could be verified by the higher storage modulus of MFREC in Fig. 5(c), due to the great dispersion of nano-/micro-particles in the epoxy matrix and the tight adhesion to the fiber/resin interface in Fig. 4(d). In summary, the storage modulus and T_g of the modified epoxy resin with multiple functional fillers increased significantly, which indicated that the MFREC had great crosslinking degree. The internal defects can be filled by multiple well-dispersed additives in the resin matrix, led to increasing the thermodynamic properties of MFREC.

3.4. Friction behaviors and wear mechanisms

3.4.1. Effect of the applied loads

In general, the frictional force is proportional to the normal applied load between the friction surfaces and is independent of the contact area. However, for the polymers and their composites, when the pressure is higher, the frictional force is not proportional to the normal load, and is related to the contact area. Therefore, in order to study the friction behavior, wear law and microscopic roughness of MFREC under different loads, Fig. 6 shows the quantitative relationship between the tribological variables. With an increase in load (Fig. 6(a)), the COFs of MFREC firstly decreased and then tended to be stable, and its fluctuation range became larger. This was mainly because the effect of loads on COFs was realized by the changes of the real contact area. For the lower load (500 g), the contact between MFREC surface and steel-ball was in the elastic or viscoelastic state, where the actual contact area was proportional to the load, so COFs was highest due to direct point contact. For the higher load (2000 g), the contact state between MFREC and

steel-ball changed from elastic or viscoelastic to plastic or viscoplastic. In this case, the increase of the area completely depended on the plastic deformation, and COFs did not change with the load changes, so it remained roughly constant. Additionally, the higher load can cause the increased wear and surface temperature, resulting in a large amount of debris at the wear track. For example, compared with that applied 500 g load (Fig. 6(b)), Ws and WSW of MFREC increased by 197.7% and 93.9%, respectively, which indicated that the wear was severe, and MFREC was great sensitive to the influence of high load factors. Possible reason was that mechanical reinforcement phases (SCFs and TiO_2) were pulled out or debonding from epoxy matrix due to the action of reciprocating high shear stress. In addition, Fig. 6(c) presents the three-dimensional skeleton diagram of MFREC surface, which can directly show the friction mark and wear profile. Meanwhile, with the continuous generation→extrusion→discharge of wear debris on the friction surface, the wear track depth in the Z-axis direction gradually increased, and its value and fluctuation range represented the friction loss of MFREC. It was noted that even under highest load (2000 g), the wear track depth of MFREC was only about $16.8 \mu\text{m}$ and its fluctuation was small, which indicated that the friction loss was uniform and there was no large area shedding phenomenon (Fig. 6(d)). This was because the graphite or ZnS particles could fill the defects and loopholes in the furrows, and then relieved the intense friction of the contact interface.

Fig. 7 shows surface wear tracks and micro-profiles of MFREC and micro-morphologies of steel-balls under the different loads to explain friction behavior and wear degree. As shown for the lower loads (Figure 7(a-a₁) and (b-b₁)), the wear track surfaces had the uniform and dense lubricated film, which can prevent the SCFs from being exposed to the grooves and also resist higher stress shearing of grinding balls. This was due to the coupling effect of the graphite and ZnS particles which together resisted high shear forces, i.e. the graphite sheets had a natural lubrication function due to the easy sliding between the layers, and the lattice-transformable ZnS can form a friction film in the furrow. Meanwhile, there were no obvious wear tracks on the corresponding steel-ball

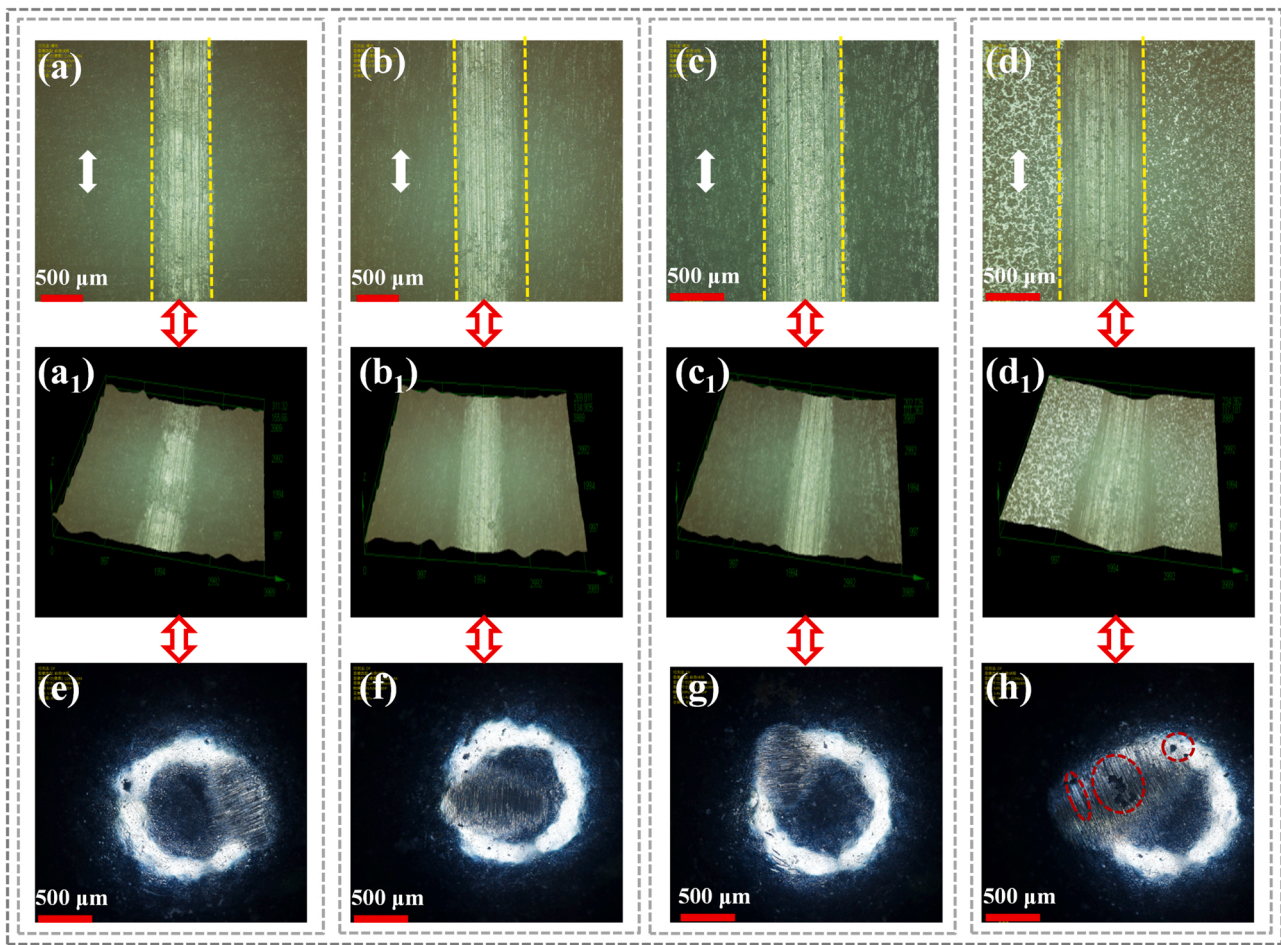


Fig. 9. Surface wear tracks and micro-profiles of MFREC and micro-morphologies of steel-balls under the different sliding speeds: (a) and (e) 60 mm/s; (b) and (f) 80 mm/s; (c) and (g) 100 mm/s; (d) and (h) 120 mm/s.

surfaces (Fig. 7(e) and (f)). As a comparison, with the increase of loads (1500 and 2000 g) in Figure 7(c-c₁) and (d-d₁), the lubricated film gradually produced micro-cracks perpendicular to the sliding direction, especially under the load of 2000 g, which was scraped off in blocks. This was because the increased load changed the contact state from elastic or viscoelastic to plastic or viscoplastic, resulting in excessive plastic deformation on the wear track surface. Furthermore, the surfaces of steel-balls in Fig. 7(g) and (h) had many parallel wear tracks, which indicated MFREC/ball interface had severe friction and wear. Possible reason was that SCFs or TiO₂ particles exposed by the shedding of the friction film increased the roughness and hardness in the furrow, thus increasing the mechanical obstruction and frictional resistance against the steel-ball.

3.4.2. Effect of the sliding speeds

The effect of sliding speed on friction behavior and wear rate was no negligible, especially in high sliding speed, easily leading to excessive plastic deformation or even material transfer at the wear track. As the increase of sliding speed in Fig. 8(a), the COFs of MFREC had the limited increase from 0.04 to 0.07, which indicated the friction force was relatively stable. However, compared to the lower speed (60 mm/s) in Fig. 8 (b), the Ws and WSW of MFREC in higher speed (120 mm/s) increased by 78.2% 39.6%, respectively. This was because the higher speed difference of MFREC/ball interface could cause uncoordinated deformation of surface and subsurface, increasing the probability of material surface loss. Meanwhile, the LR profiles were relatively clear under the 80 and 120 mm/s speeds in Fig. 8(c), which could be attributed to local damage of lubricating film due to the intense friction of SCFs and steel-

balls. Additionally, it was noted that the LR value of MFREC at 100 mm/s speed was extremely stable, which was because graphite or ZnS could encapsulate the thinning→cracking→breaking series of SCFs, avoiding the exposure of their tips and reducing the intense friction at the MFREC/ball interface.

The wear morphologies of MFREC and steel-balls under the different sliding speeds were shown in Fig. 9(a-d) and (e-h). With the increase of sliding speed, the furrow surfaces all had wear tracks parallel to the sliding direction, especially at the 100 and 120 mm/s speed with visible wear debris on the surface Fig. 9(c) and (d). This was because the high shear rate increased the frictional heat at the interface, resulting in the epoxy matrix to be softened due to untimely heat dissipation, which was manifested as adhesive wear and slight abrasive wear. Furthermore, as shown from the micro-profiles at different sliding speeds in Fig. 9(a₁-d₁), the width of the wear tracks were getting wider, which indicated that the wear depth of MFREC also increased. Additionally, the surfaces of steel-balls all showed varying degrees of wear, especially at a speed of 120 mm/s, and the steel-balls had been worn out and showed many spots. This was because the excessive plastic deformation of epoxy matrix at higher speed caused the SCFs or nano-TiO₂ to be continuously extruded and impeded the reciprocating movement of steel-balls. As a result, the reinforced-particles were stripped from the MFREC and the steel-balls were worn out.

3.4.3. Effect of the service temperatures

The temperature was a significant indicator affecting the tribological performances of polymers and their composites. Especially in higher temperature, the frictional heat generated by the frictional pair was

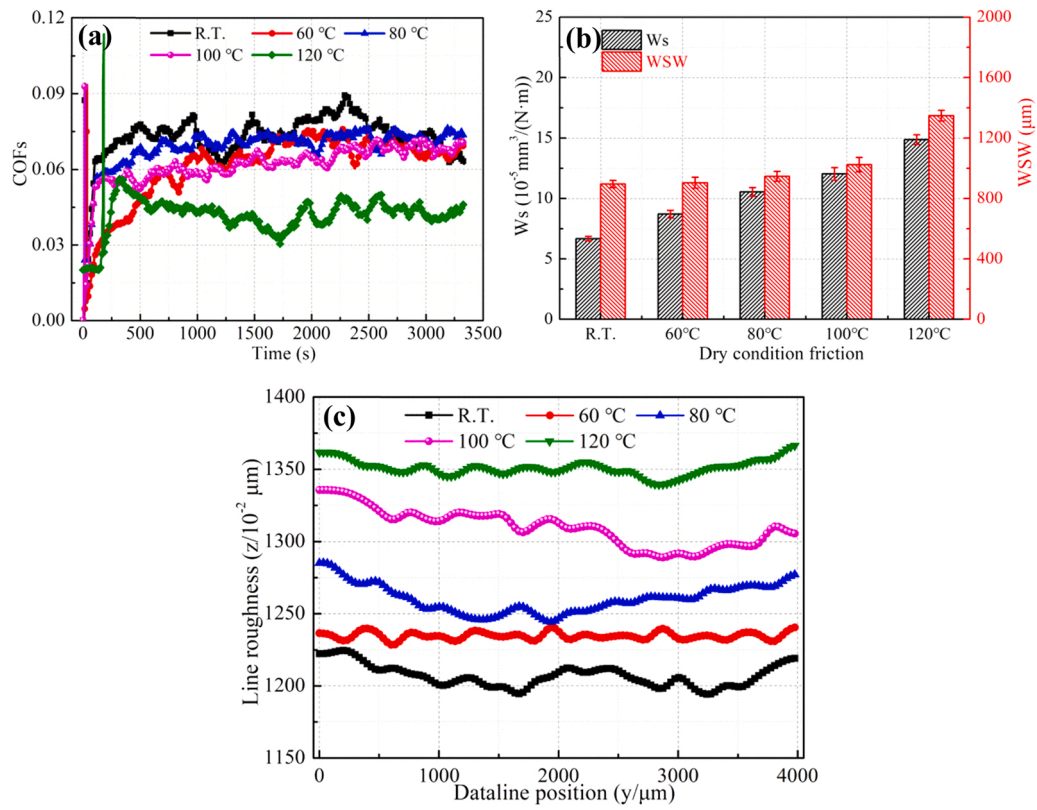


Fig. 10. Effect of service temperatures on friction behavior and wear rate of MFREC: (a) COFs; (b) Ws and WSW; (c) LR along the sliding direction.

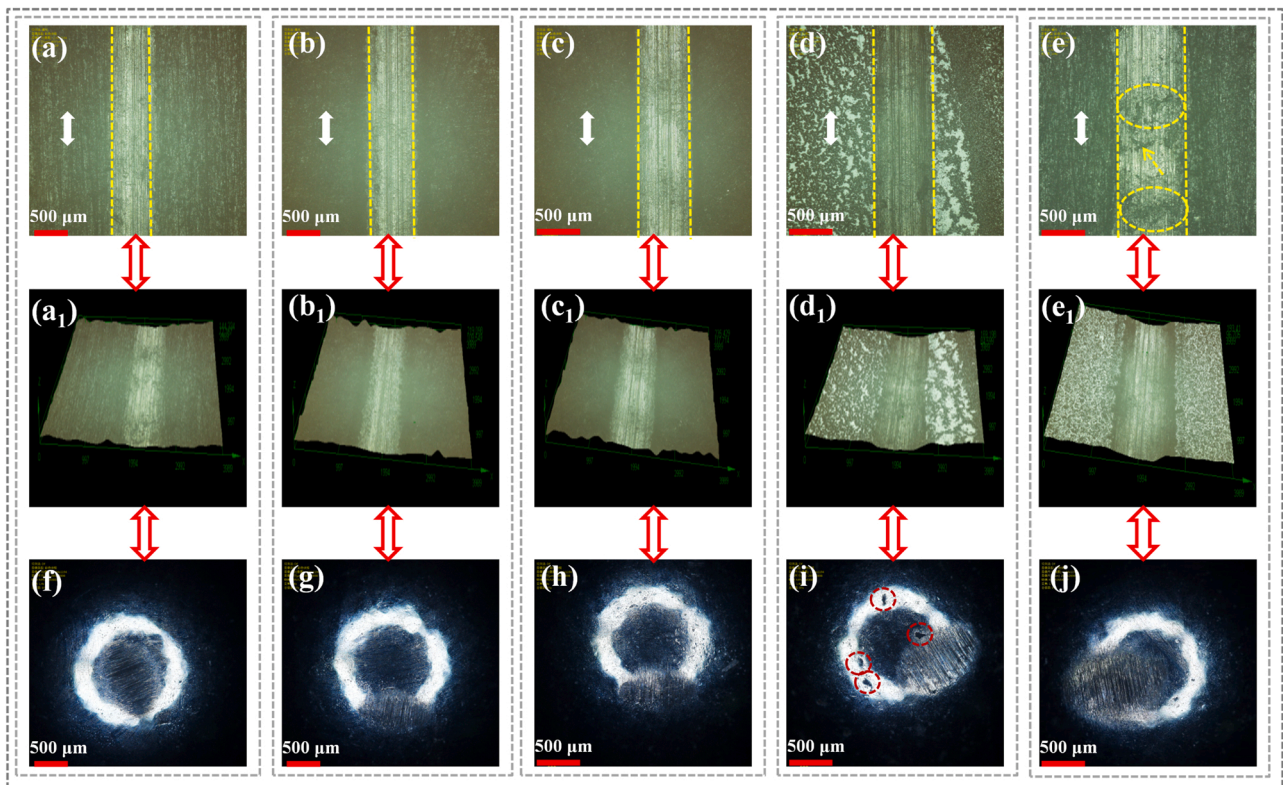


Fig. 11. Surface wear tracks and micro-profiles of MFREC and micro-morphologies of steel-balls under the different service temperatures: (a) and (f) R.T.; (b) and (g) 60 °C; (c) and (h) 80 °C; (d) and (i) 100 °C; (e) and (j) 120 °C.

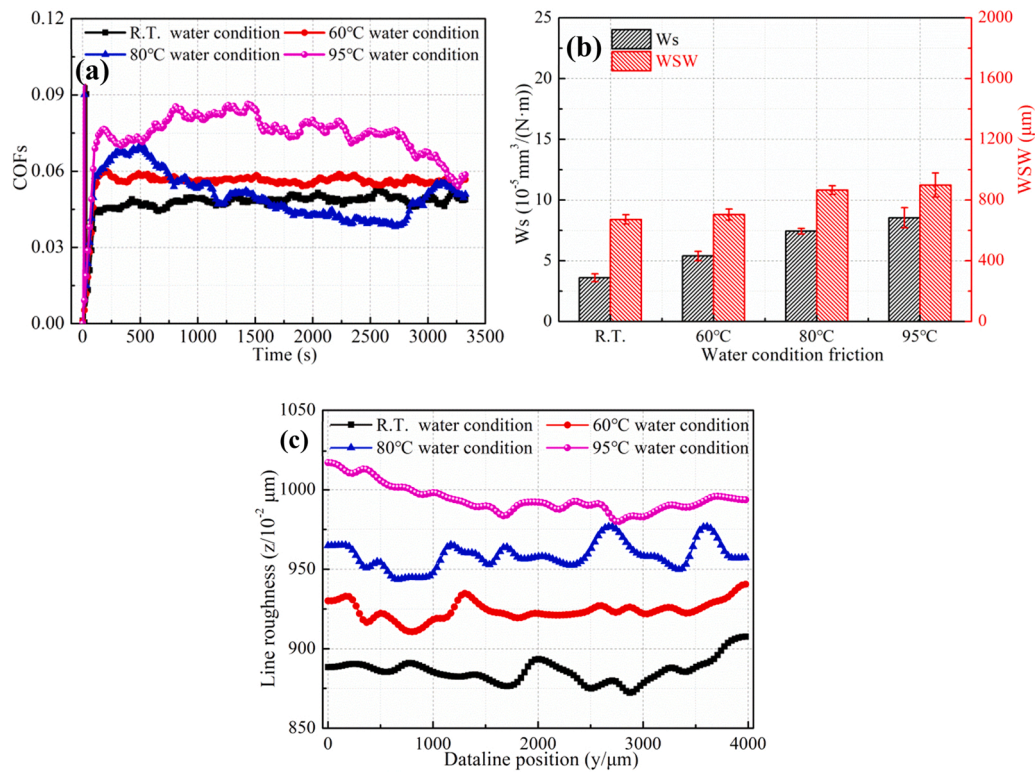


Fig. 12. Effect of water lubrication at the different temperatures on friction behavior and wear rate of MFREC: (a) COFs; (b) Ws and WSW; (c) LR along the sliding direction.

hindered to propagate to the subsurface of epoxy matrix, resulting in higher local temperature at the contact point and significant plastic deformation. As shown in Fig. 10, the effect of different service temperatures on friction behavior and wear law of MFREC was presented. With an increase in service temperature (Fig. 10(a)), the COFs gradually decreased, especially at 120 °C, by 43.8% compared to that at R.T., which was because the excessive plastic deformation could protect the fibers tips from exposure and prevent epoxy matrix damage. Therefore, the Ws and WSW of MFREC at 120 °C was highest (Fig. 10(b)), increasing by 122.6% and 50.6% compared to the R.T., respectively. This was because the increase in temperature caused the physical state of the polymer surface to change from a glassy state to a high elastic state or a viscous flow state, forming a low-viscosity viscous-fluid layer and resulting in MFREC with a higher wear volume. Additionally, with the increase of temperature, the thermal deformation of epoxy matrix increased obviously, indicating that the resistance to thermal deformation and hardness of MFREC decreased, which would reduce its wear resistance. Furthermore, the LR values of MFREC increased with the increased temperatures in Fig. 10(c), which was because the service temperature was closed to that of T_g (128.6 °C in Fig. 5(b)), and the plastic deformation of epoxy matrix was more obvious due to the increase of its molecular chain activity.

Fig. 11 shows the worn surface appearances of MFREC and steel-balls under the different service temperatures. The worn surfaces of MFREC at the relatively lower temperatures (R.T., 60 and 80 °C) did not show serious fiber cracking, pulverization (Fig. 11(a-c)), and visible contour changes (Fig. 11(a₁-c₁)), which was because the epoxy matrix still remained glassy state and did not lose surface hardness due to the high thermal conductivity of graphite flakes. In contrast, as the temperature continued to rise to 100 and 120 °C (Figure 11(d-d₁) and (e-e₁)), the surfaces of MFREC showed more wear tracks perpendicular to the sliding direction, especially in the latter, appearing the local plastic deformation and grooves. Furthermore, the steel-ball surfaces at 100 and 120 °C (Fig. 11(i) and (j)) appeared to the clear wear signs

compared to those at the lower temperature conditions (Fig. 11(f-h)). However, it was noted that the pits appeared on the steel-ball surface only at 100 °C, which may be because the fibers or nanoparticles coated by the epoxy matrix were exposed to the wear track surface and directly contacted with the grinding ball due to the transition of resin from glassy state to high elastic state. Additionally, as the temperature increased again, the pits could not be easily detected by the ball surface (Fig. 11(j)), which was due to the formation of a continuous friction film under the higher temperature and normal force.

3.4.4. Effect of the water lubrication

Based on the above dry friction state, the wet friction at different temperatures was selected as comparison to verify the tribological service performances of MFREC. In contrast to the dry friction, the friction behavior and wear state of MFREC were affected by the coupling effects of the self-lubricating film and water film. Meanwhile, the integrity and continuity of water film also were depended on the experimental temperature, so four kinds of temperatures (R.T., 60, 80 and 95 °C) were selected for the friction and wear tests. It was noted that in order to prevent the rapid boiling and evaporation of water molecules at 100 °C, and the water environment at 95 °C was used for friction lubrication. With an increase in temperature, the fluctuation amplitude of COFs increased in Fig. 12(a), especially in 95 °C water condition, which was attributed to the discontinuity of water molecule film due to the transition from liquid to gaseous water at the elevated temperature. Additionally, compared with the dry state friction (different loads, sliding speeds and service temperatures), the MFREC presented the increased wear resistance under the wet friction of different temperatures. For example, compared to the 2000 g load (Fig. 6(b)), 120 mm/s (Fig. 8(b)) and 120 °C (Fig. 10(b)), the Ws and WSW of MFREC (Fig. 12(b)) at 95 °C water lubrication condition decreased by 132.9% and 83.9%, 41.1% and 31.9%, 74.1% and 50.0%, respectively. Furthermore, the anti-wear performance of MFREC was more stable to water lubrication than temperatures. Despite the large increase in temperature, the change in

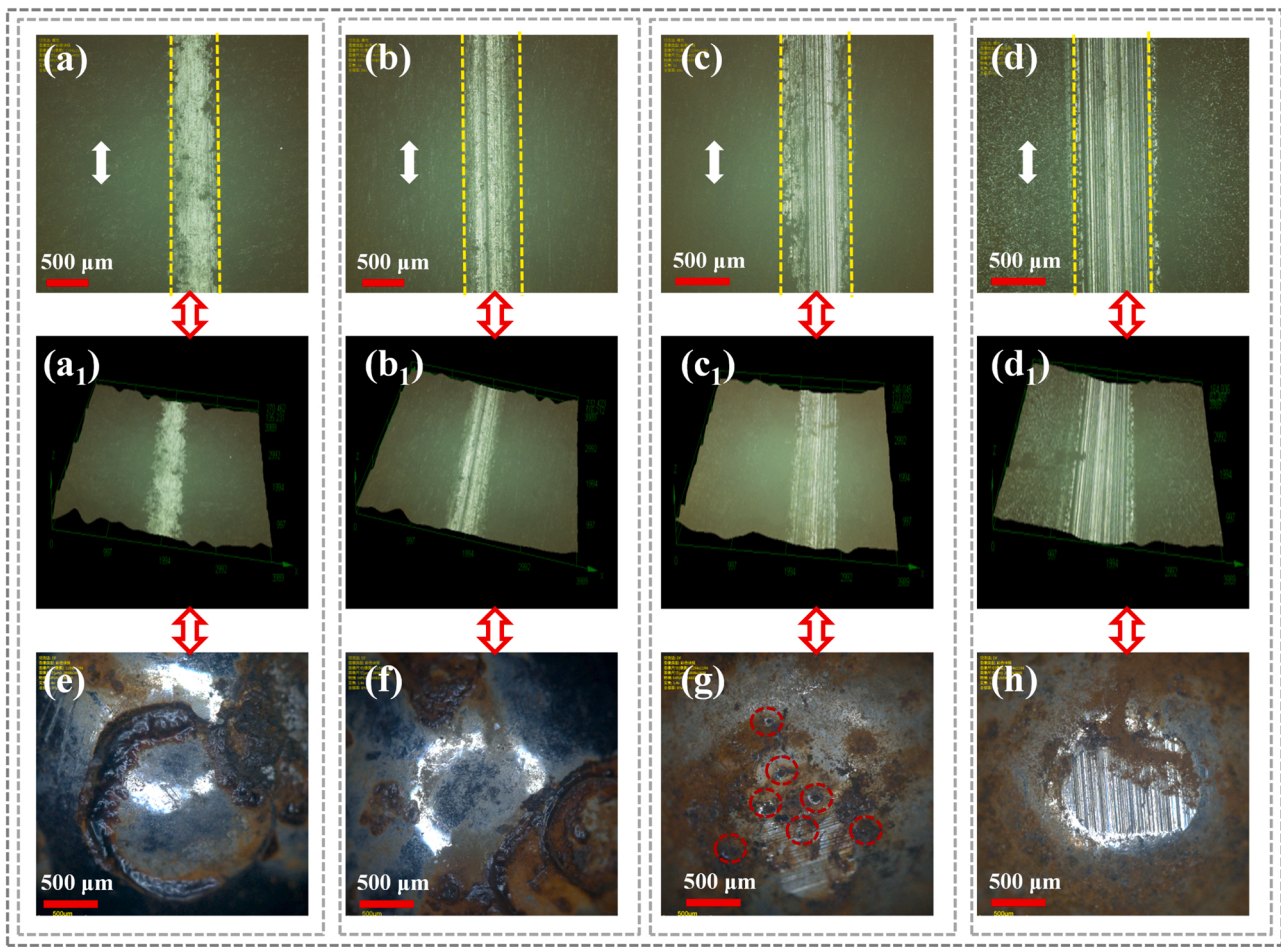


Fig. 13. Surface wear tracks and micro-profiles of MFREC and micro-morphologies of steel-balls under the water lubrication of different temperatures: (a) and (e) R. T.; (b) and (f) 60 °C; (c) and (g) 80 °C; (d) and (h) 95 °C.

wear rate of MFREC was relatively small. For instance, the maximum percentage increase in LR values from R.T. to 95 °C water lubrication only was 12.1% in Fig. 12(c), and this was because the carrying capacity of water films can partly resist the compression load and relieve the direct contact between steel-ball and MFREC. For example, Zhang et al. [27] pointed out that the friction pair exposed to seawater environment worked in wet friction state, and the seawater can play a lubricating role and increase the distance between material and counterpart, led to a transition of wear mechanism from severe fatigue wear to slight abrasive wear.

Fig. 13 presents surface morphologies of wear tracks and steel-balls after friction tests in water lubrication conditions at the different temperatures. Compared with the lower temperatures (R.T. and 60 °C) as shown in Fig. 13(a) and (b), the MFREC surfaces (Fig. 13(c) and (d)) had the obvious wear tracks along the sliding direction, and this was because the self-lubricating film of MFREC was damaged owing to the rapid vaporization of water molecules at the elevated temperatures, leading to the distance reduction between friction pairs. Furthermore, as can be seen from the micro-profiles of wear tracks (Fig. 13(a₁)-(d₁)), the increased temperatures intensified the depth and width of furrows, which indicated the water lubrication at the elevated temperature would aggravate the wear volume of MFREC. This can be attributed to two reasons. One was that an elevated temperature increased the molecular chains creep of epoxy matrix and reduced the surface hardness of MFREC. The other was an elevated temperature could destroy the continuity of lubricating water film, leading to the increased probability of the direct contact between the steel-ball and MFREC due to the rapid evaporation and boiling of water molecules. Additionally, the steel

counter-faces in Fig. 13(e) and (f) had no the obvious wear lines, except for electrochemical corrosion traces. However, the ball surface in Fig. 13 (g) had numerous corrosion pits (marked with red circles), which indicated that the severe corrosion wear had occurred due to the destruction of the dense oxide film on the steel surface by reciprocal friction. As the temperature increased to 95 °C, the above corrosion pits disappeared and was replaced by a bright wear plane, which indicated that the intense friction and wear occurred between the friction pairs due to the decreased surface hardness of MFREC and discontinuity of lubricating water film. This phenomenon could explain the results of the increased COFs, Ws, WSW and LR values (Fig. 12).

3.4.5. Analysis of the sensitive factors

In order to compare the sensitivity of friction behavior and wear rate of MFREC to external environmental conditions, Fig. 14 summarizes the micro-topographies of MFREC with the most serious wear under the load (2000 g), sliding speed (120 mm/s), temperature (120 °C) and water lubrication factor (95 °C). As shown, the lubricated films of different morphologies are presented at the low magnification (Fig. 14(a)-(d)), which can reflect the frictional evolution behavior of MFREC/steel-ball interface by the integrity and compactness of films at the wear track area.

For the applied load effect (Fig. 14(a)), the lubricated film was discontinuous and had a certain hierarchical structure, which indicated a violent mechanical occlusion due to the higher shear force of applied-load steel-ball. Furthermore, for the corresponding enlarged images (Fig. 14(a₁) and (a₂)), the plastic deformation of epoxy matrix and pull-out of fibers were identified in the wear area, which was attributed to the

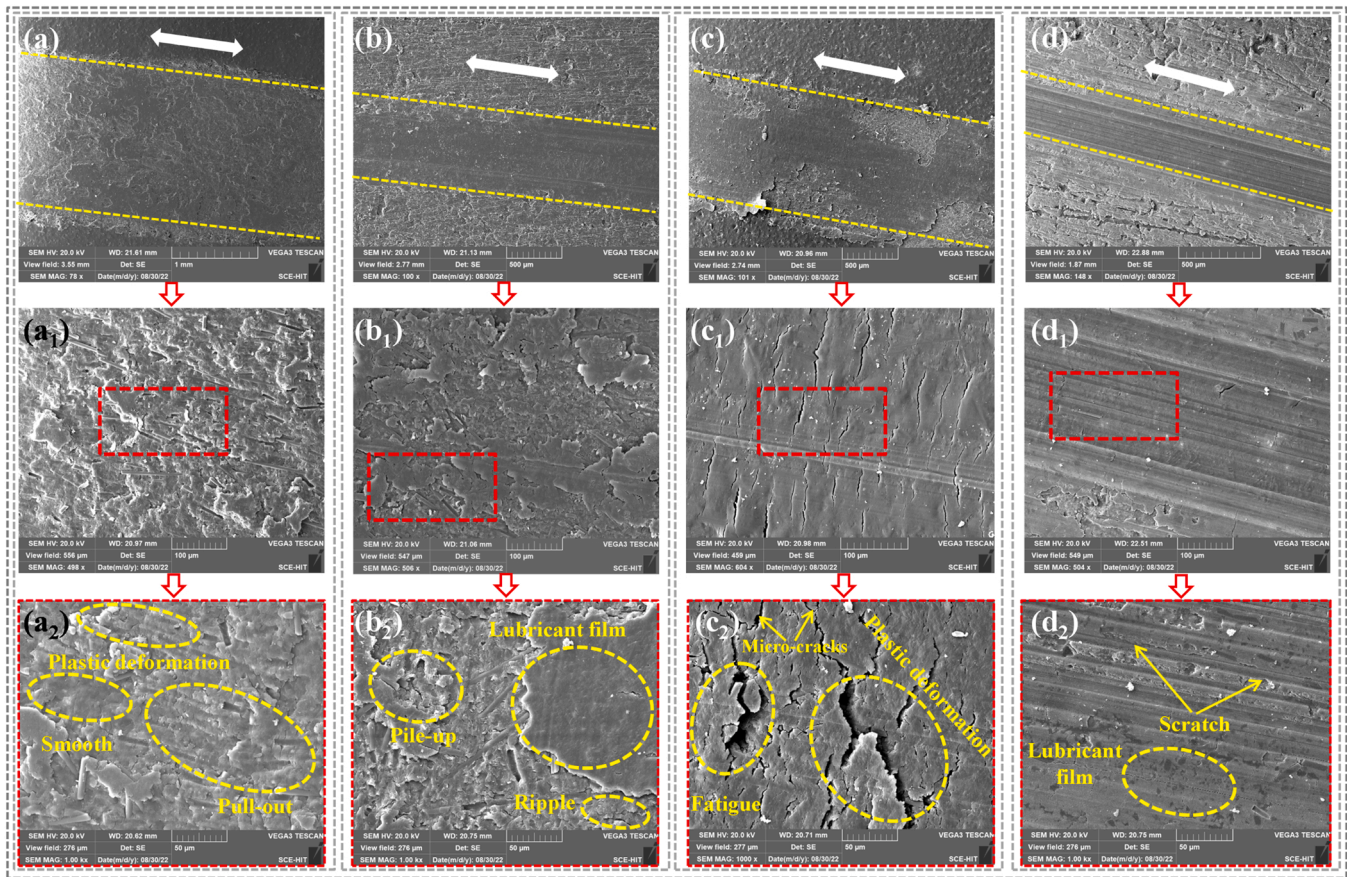


Fig. 14. Comparison of worn morphologies of MFREC under each of the most severe external environmental conditions: (a) 2000 g load; (b) 120 mm/s sliding speed; (c) 120 °C of dry friction; (d) 95 °C water lubrication condition. ((a₁)-(d₁) and (a₂)-(d₂)) were the enlarged images corresponding to (a)-(d)).

large frictional force generated by a tangential displacement on the surface. The convex surface of softer surface was easy to deform and some were fractured under the repeated friction action, and finally a relatively smooth area was generated or part of the grooves were reshaped or removed, resulting in a highest *Ws*, *WSW* and *LR* (Fig. 6), according with delamination wear mechanism [28]. For the sliding speed effect (Fig. 14(b)), the wear surface was relatively smooth and a complete lubricated film was presented along the sliding direction, which can be used to alleviate further scratching of MFREC surface by high-speed shear stress. Meanwhile, for the enlarged pictures (Fig. 14 (b₁) and (b₂)), a large isolation film was appeared and the epoxy tightly wrapped the fibers in the wear track, where fibers/epoxy interface did not debonding. In addition, the ripple of epoxy matrix and pile-up of wear debris were presented due to the high-speed and reciprocating sliding of the steel-ball, resulting in excessive plastic deformation of surface layer and lagging elastic deformation of the underlay layer on MFREC, led to the stable *Ws*, *WSW* and *LR* variations (Fig. 8), which was consistent with adhesive wear mechanism [28]. For the experimental temperature effect (Fig. 14(c)), although a lubricant barrier film was produced at the wear track, this film had a lower degree of adhesion to the underlay layer and was easily subject to warping. Additionally, for the enlarged views (Fig. 14(c₁) and (c₂)), there were many micro-cracks with different sizes and uniform distribution perpendicular to the sliding direction on the MFREC surface. This was because the higher service environment decreased the MFREC ability to resist thermal deformation, which in turn led to a lower surface hardness. Meanwhile, due to the increase of molecule activity of epoxy matrix at higher temperature (close to *T_g*), the inconsistent deformation of each area at the wear track caused the continuous initiation and development of cracks, and finally evolved into tearing and surface fatigue phenomenon, which meted to

the fatigue wear mechanism [28]. In brief, the surface fatigue resulted in wear damage and fracture, which were due to the accumulation of irreversible changes, leading to repeated stressing and un-stressing of contacts, thus having the second highest *Ws*, *WSW* and *LR* (Fig. 10). For the water lubrication effect at 95 °C (Fig. 14(d)), the wear track surface was flat and compact due to the lubricating effect and heat dissipation function of water film at the MFREC-surface/steel-ball interface, which indicated the MFREC had the remarkable wear resistance against reciprocating frictional force and higher shear stress. In addition, Fig. 14 (d₁) and (d₂) show the presence of wear tracks parallel to the sliding direction, and those were great shallow and hardly caused excessive plastic deformation of epoxy matrix, which could explain the reasons why *Ws*, *WSW* and *LR* were the smallest (Fig. 12) compared to other factors. Meanwhile, the formation of wear debris (Fig. 14(d₂)) during the friction process was caused by plowing, cutting and cracking mechanisms induced by the hard asperities of steel ball, which was known as the abrasive wear [28]. In conclusion, among the several influencing factors studied for friction behavior and wear rate, MFREC was the most sensitive to the load effect due to the higher shear force of applied-load steel-ball by a tangential displacement, temperature second, sliding speed third and water lubrication last because of the lubricating effect and heat dissipation function of water film.

3.4.6. Analysis of the wear mechanisms

Based on the analysis of the above-mentioned sensitive factors on friction behavior and the comparison of different wear morphologies, Fig. 15 shows the wear types and mechanisms of MFREC under several experimental conditions in order to illustrate their wear resistance and service life. For the delamination wear (load effect) as shown Fig. 15(a), the local plastic deformation in two surfaces that were only in contact at

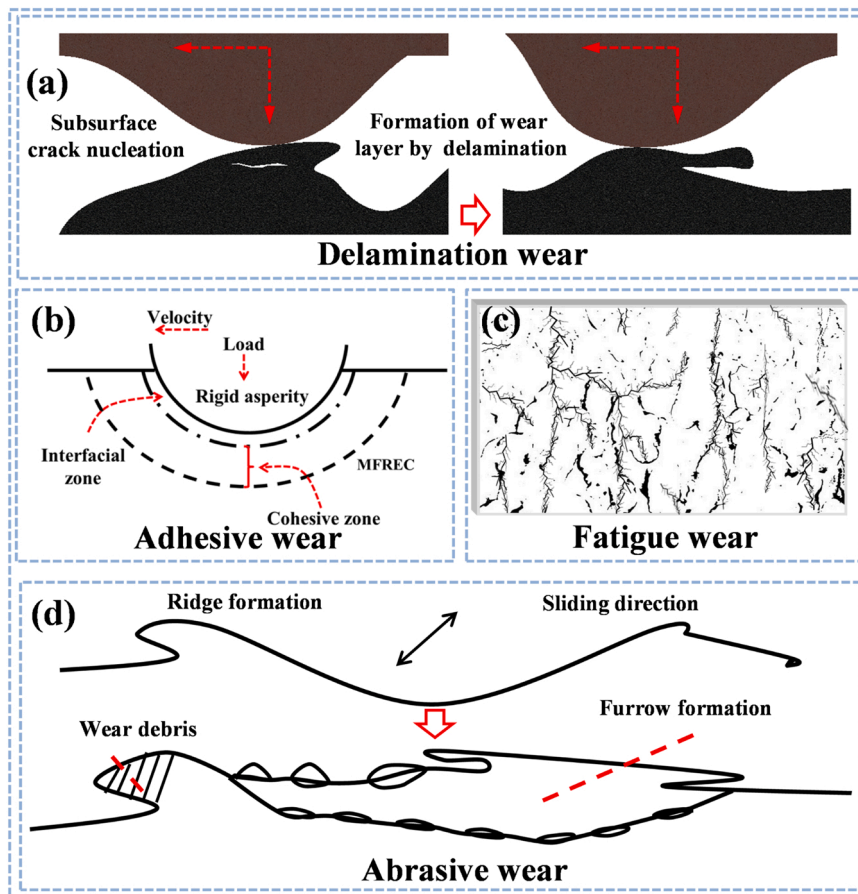


Fig. 15. Types of reciprocating friction and wear of MFREC: (a) delamination wear; (b) adhesive wear; (c) fatigue wear; (d) abrasive wear.

the interface occurred in these uneven places due to the small contact area. Therefore, the combination of normal and tangential traction forces resulted in a zone of compressive stress below and ahead of the asperities and a zone of tensile stress behind the asperities. Meanwhile, MFREC close to the surface would undergo repeated loading and unloading cycles whenever a rough surface passed by, eventually leading to a transformation of the contact surface from asperity-to-asperity to asperity-to-plane, where the wear debris fell off in the form of layers. For the adhesive wear (sliding speed effect) as shown Fig. 15(b), the epoxy matrix on the MFREC surface underwent a series of changes, such as fracture→connection→fracture, and then its fragments were continuously squeezed again. This was because the relative movement between interfaces led to the replacement of the above “fracture” and “connection” phenomena, thus creating the frictional adhesion morphology. In addition, the above contact area of the “connection” was a result of physical or chemical interactions in the MFREC/steel-ball interfaces, such as van der Waals force and hydrogen bonding. At the same time, local frictional heat may cause the polymer to melt or soften, when the bonding strength at the adhesive interface was higher than the shear strength of epoxy matrix, and the abrasive debris started to fall off from the MFREC surface, thus causing the adhesive wear. For the fatigue wear (temperature effect) as shown Fig. 15(c), its characteristic feature was the accumulation of irreversible changes due to the repeated stressing and un-stressing of contacts, which caused the generation and development of micro-cracks. As a result, the surface of epoxy matrix was constantly torn due to the cyclic high shear stress, thus the numerous defects were formed perpendicular to the sliding direction, led to the fatigue wear. For the abrasive wear (water lubrication effect) as shown Fig. 15(d), the wear debris squeezed by the steel-ball would form ridges along the sides of grooves, after the repeated loading and unloading, and the ridge edges were flattened by the micro-convex body

and caused the small plastic deformation of subsurface. As a result, the surface and subsurface cracks could form nucleation points and connect with adjacent cracks, and eventually extend to the surface to form wear furrows or wear ridges, which was manifested as abrasive wear.

In summary, the delamination wear was a result of compressive stress and tensile stress in the area around the contact points, leading to a transformation of contact surface from asperity-to-asperity to asperity-to-plane, where the wear debris fell off in the layer form from MFREC surface. The adhesive wear referred to the relative movement of contact surface, due to the continuous “fracture” and “connection” between the contact points, and the bonding strength of interface formed by the adhesive point was higher than the shear strength of adhesive body, so that the material fell off from the surface in the form of free abrasive debris. The fatigue wear was the surface fatigue of MFREC under reciprocating high shear stress, and the initiation and expansion of surface crack were caused by repeated stress and non-stress contact, and finally fatigue fracture occurred. The abrasive wear was the process or phenomenon of material migration caused by the interaction between the bulge and contact surface. Along with matrix deformation→tillage→fracture→cutting, the surface wear tracks of MFREC finally formed a ridge or furrow shape.

4. Conclusions

In the present paper, the MFREC was prepared and analyzed combined with the mechanical reinforcement and frictional lubrication phases. Then, the mechanical (tensile/flexural), thermal and tribological tests under the various service conditions were systematically investigated to characterize the mechanical/thermal reinforcement and friction/wear resistance of MFREC. Furthermore, the anti-friction sensitive factors and wear mechanisms of MFREC under the external

environmental conditions were analyzed and compared in details, which would provide a theoretical basis for the actual applications, and the following conclusions were finished based on the testing results and discussion.

- 1) After adding the functional fillers, the tensile toughness of MFREC increased by 98.46%, which was attributed to the increase of tensile strength (9.56%), tensile modulus (22.07%) and elongation at break (39.09%) due to the crack inhibition and high strength-modulus of nano-TiO₂/SCFs as well as the interlayer lubrication and mechanical interlocking effects of graphite/ZnS.
- 2) Compared with epoxy resin, Tg of MFREC increased by 19.6%, which was because the multi-fillers can fill the micropores or microcracks in epoxy matrix, leading to a improvement of its curing degree. Furthermore, its storage modulus (30–100 °C) had an obvious increase, which indicated the fillers and epoxy matrix had excellent thermodynamic compatibility behavior.
- 3) Among the several influencing factors studied for friction behavior and wear rate, MFREC was the most sensitive to the load effect due to the higher shear force of applied-load steel-ball by a tangential displacement, temperature second, sliding speed third and water lubrication last because of the lubricating effect and heat dissipation function of water film.
- 4) The delamination wear was a contact surface transformation from asperity-to-asperity to asperity-to-plane owing to compressive stress and tensile stress. The adhesive wear was due to the continuous “fracture” and “connection” between the contact points. The fatigue wear was the initiation and expansion of surface crack due to repeated stress and non-stress contact. The abrasive wear was caused by the interaction between the bulge and the contact surface.

Compliance with ethical standards

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Declaration of Competing 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.

Data availability

The authors do not have permission to share data.

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