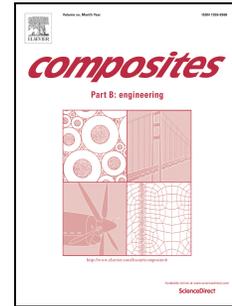


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micro-sized ZrB₂ particles**

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Tribological performance of in-situ epoxy composites filled with micro-sized ZrB₂ particles

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Abstract

The tribological performance of in-situ epoxy composites reinforced with micro-sized particles of zirconium diboride (ZrB₂) were investigated in dry sliding conditions. The frictional heat flow in the contact areas was analyzed using finite element modeling. It was found that the dominant wear mechanisms of composites were changed from adhesive wear to abrasive wear due to the introduction of ZrB₂ particles under the imposed sliding conditions. The coefficient of friction (CoF) and coefficient of wear K of composites decreased significantly with the increase of ZrB₂ particle content. The increase in thermal conductivity due to the addition of ZrB₂ particles was proposed to contribute to the enhanced wear resistance under given sliding conditions.

Keywords: A. Polymer-matrix composites (PMCs); B. Wear; C. Analytical modeling; D. Thermal analysis

1 Introduction

The epoxy resin has been widely applied as the friction parts of oil-free applications in the fields of high-speed rail, automobile industry and aerospace as gears

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and shaft parts due to its good elasticity, abrasive resistance and light weight[1,2]. However, the adhesive wear and high local temperature during the friction process will reduce the service life of the friction parts. According to some reports, the introduction of some ceramic fillers, such as SiC, ZrO₂ and h-BN, with high wear resistant performance and thermal conductivities is an effective method to improve the tribological and thermal performance of polymeric composites under high loading[3-5]. These studies were mainly focused on the discussion of effects of filler properties, such as size, shape and content, etc. on the related performance of composites, and the researches about the effects of the thermal conductivity of fillers on the tribological properties of filling composites have still not been performed sufficiently, in particular, the research of tribological performance of the epoxy composites filled by micro-sized ZrB₂ particles has not been reported by the finite element analysis of frictional heat. ZrB₂ is one of the most important boride ceramic materials. Compared with other ceramic fillers, ZrB₂ is regarded as the potential filler with high tribological performance due to its lamellar crystal structure similar to the graphite and high hardness which are contributed to the increase of tribological properties[6]. In some studies, ZrB₂ has been applied in ceramic matrix composites (CMCs) and metal matrix composites (MMCs) as the reinforcements[7-9]. It shows great tribological performance in these composites. Thus, ZrB₂ seems a potential reinforcement in the improvement of tribological performance in polymeric matrix composites. Also, the addition of ZrB₂ to epoxy matrix is expected to present higher tribological performance than that of other

inorganic fillers.

In this work, epoxy composites filled with micro-sized ZrB_2 particles were prepared by means of in-situ synthesis method for better dispersion of the particles in the matrix. The dispersion of ZrB_2 particles in epoxy matrix has been discussed in our previous work[10]. The friction and wear characteristics of epoxy composites were investigated by the wear tester to determine the optimal fraction of ZrB_2 particles. The finite element models (FEMs) were run for the analysis of frictional heat during the friction process.

2 Experimental

The epoxy resin was synthesized with BPA (bisphenol A, >99.0 wt% ,white crystal, Sinopharm Chemical Reagent Co. Ltd) and epichlorohydrin (>99.0 wt%, colorless transparent oily liquid, Sinopharm Chemical Reagent). BPA was solved in NaOH (solution, 0.3mol/L) at 70°C, and ZrB_2 particles (>99.5 wt% (The detailed chemical composition is shown in Table 1), mean particle size 1-3 μ m, microhardness 22.1 GPa, elasticity modulus 343.0 GPa, supplied by Aladdin Reagent, preprocessed by the ultrasound with ethanol for 30 min) were introduced into the solution under mechanical agitation at 200 r/min. The temperature of the solution was reduced to 50°C after uniform dispersion of ZrB_2 particles, and then the epichlorohydrin was added by droplets slowly. The reaction product was poured into the mold after the temperature of the solution being raised to 90°C for 2 hours. The mold was heated over 150 °C and vacuumized in the vacuum oven until all solvent and trapped bubbles removed. The

mold was cooled to 70 °C and then triethylenetetramine served as curing agent (Sinopharm Chemical Reagent Co., Ltd. China) was added and mixed evenly in each mold. Then the mold was heated again to 120 °C for 5 hours in order to cure the resin matrix. ZrB₂ particles were added in different percentages: 1 vol%, 3 vol%, 5 vol% and 7 vol% with regard to epoxy volume. The volume fraction of ZrB₂ particles in epoxy matrix can be determined from the following equation.

$$\text{Volume fraction (vol.\%)} = \frac{\text{mass fraction (wt.\%)} \times \rho_{ep}}{(1 - \text{wt.\%}) \times \rho_{ZrB_2}} \times 100\% \quad (1)$$

Where wt.% is the mass fraction of the particles with regard to filled composites mass, and ρ is the density and the subscripts “ep” and “ZrB₂” refer to epoxy matrix and filler ZrB₂, respectively.

The tribological properties were evaluated on the Amsler block-on-ring friction and wear tester under dry sliding conditions at room temperature, and the relative humidity was 60%. The contact schematic diagram of sample with counterpart is shown in Figure 1. As shown, the specimen for wear tests was machined with the geometry of 30mm×7mm×6mm. The steel ring made of Steel 45, of which the element composition is displayed in Table 2., was used as the counterpart in wear tests in this study. The sample was sliding against the counterpart for 2 hours with a velocity of 0.42 and 0.84 m/s under the normal load of 250 and 150N, where the maximum contact pressures during the whole experimental process were 11.9 and 7.1MPa, respectively. The steel ring and specimens were polished with abrasive papers (5 μ m to 7 μ m) and the mean surface roughness R_a was controlled at 0.2-0.3 μ m and then cleaned with acetone before

each test. In order to reduce the error, each sample with the certain volume fraction of ZrB₂ fillers was tested three times. The worn surfaces of samples were observed with a scanning electron microscope (SEM, Philips, XL30FEG, Holland). Coefficient of wear K was calculated by the following equation:

$$\text{coefficient of wear } K = \frac{\Delta m}{L\rho F_N} \text{ m}^3/(\text{Nm})^{-1} \quad (2)$$

Where Δm is the mass loss (kg), ρ is the density of specimen (kg/m^3), F_N is the normal load (N), and L is the sliding distance (m). The coefficient of friction was calculated by dividing the frictional force with the normal load.

3. Results and discussion

Figure 2 shows the variation of CoF and coefficient of wear K for the ZrB₂/epoxy composites as a function of filler content at a sliding speed of 0.42 m/s and under 250 N load. As shown in Fig. 2, both the CoF and coefficient of wear K of epoxy composites are reduced significantly with the addition of ZrB₂ particles. When the volume fraction of ZrB₂ particles is 5 vol%, the filled epoxy composite presents the lowest CoF and coefficient of wear K. Subsequently, both the CoF and coefficient of wear K of composites slightly increase when the ZrB₂ particle contents exceed 5 vol%, which could be resulted from the agglomeration of ZrB₂ particles in epoxy matrix. The friction and wear characteristics of epoxy composites under 150 N and at the same speed of 0.42 m/s are shown in Figure 3. As seen in the Fig.3, the CoF and coefficient of wear K of composites are reduced further compared with the results in Fig. 2. With the addition of ZrB₂ particles, the surface hardness and modulus of the epoxy composites were

improved. The harder and stiffer surface of epoxy composites filled with ZrB_2 particles could resist wear better. Besides, the asperities of epoxy composites were deformed less under lower load which could decrease the adhesion between the friction parts. Thus, the lower CoF and coefficient of wear K of the epoxy composites were obtained as shown in Fig.3. The CoF and coefficient of wear K of the epoxy composites under 250 N and at a sliding speed of 0.84 m/s are shown in Figure 4. As shown, the changes in trend of the friction and wear characteristics of epoxy composites at higher sliding speed are similar to that at lower sliding speed (see Fig.2.), and both the CoF and coefficient of wear K are slightly increased in comparison with the results in Fig. 2. Under all load-speed conditions, 5 vol% ZrB_2 particle content is the optimal concentration to improve the friction and wear characteristics of epoxy composites.

Figure 5 presents comparative curve graphs of the variations of CoF against sliding time for the epoxy composites filled with 0 to 5 vol% ZrB_2 particles under 250 N and at a sliding speed of 0.42 m/s. As shown, the CoF of epoxy composites filled with ZrB_2 particles are lower than that of epoxy resin, and with the increase of ZrB_2 particle content, the CoF of epoxy composites decreases, which can be attributed to high tribological performance of ZrB_2 particles. Also, it can be observed that the CoFs of epoxy composites increase rapidly in the initial stage, and then the growth rate of CoFs becomes slow and reaches the peak values. It was mainly due to the increased real contact areas between samples and counterpart during initial friction process[11]. The large contact pressure would cause the severe adhesion and increase the CoF of epoxy

composites. With the samples being worn, the contact area increased and thus the contact pressure decreased, which resulted in the slowdown of the growth rate of CoF of epoxy composites, and then the CoFs of epoxy composites reached the peak values. In comparison, it took more time for the CoFs of epoxy composites with higher filler content to reach the peak values. The peak time of the CoF of epoxy composite with 5 vol% ZrB₂ particles is about 40 mins, while the peak time of the CoF of epoxy composite with 1 vol% ZrB₂ particles is 32 mins. It can be resulted from higher filler content being contributed to the increase of wear resistance of filling composites, which delayed the increase of contact areas. After peak, CoF decreases with the increase of sliding time. It can be suggested that the incorporation of ZrB₂ particles can accomplish a self-lubricating purpose[12]. In addition, the micro-sized ZrB₂ particles could polish the surface of the counterpart to a fine scale[6]. As a result, the CoFs of epoxy composites were reduced.

Figure 6 shows SEM images of worn surfaces of the epoxy composites with 0 vol% and 5 vol% ZrB₂ fillers under 250 N and at a sliding speed of 0.42 m/s. It can be seen in Fig. 6a that the epoxy resin is peeled from the surface and some ploughed grooves are formed on the worn surface, and the worn surface is rough, which indicates that the adhesive wear is the dominant wear mechanism of epoxy resin. Specially, under the sliding condition, the epoxy resin can be soften and deformed and further peeled from the block resin due to the temperature rise caused by friction to form the pits as well as ploughed grooves on the surface ultimately. It is in accordance with that of Ref [13]. In

the case of ZrB_2 /epoxy composites, a different worn surface morphology is observed. As shown in Fig.6b, the worn surface of the composites with 5 vol% ZrB_2 fillers becomes much smoother compared to the pure epoxy, and the evenly dispersed ZrB_2 particles in the resin matrix can be found. This can be further confirmed by high magnified SEM micrograph (Fig.6c, 1000 \times) of the worn surface in Fig.6b (200 \times). Meanwhile, some shallow ploughing and convex particles and tiny debris on the worn surface can be also observed. This indicates that the dominant wear mechanism has been changed from the adhesive wear of pure epoxy to the abrasive wear of composites [2]. In other words, after the introduction of ZrB_2 fillers, the plastic deformation of epoxy resin can be well reduced because the ZrB_2 particles with high thermal conductivity can promote the heat dissipation inside the composites and decrease the frictional temperature rise effectively. Besides, under the sliding condition, the convex hard ZrB_2 particles on the contact surface can undertake a certain part of load force and decrease the peeling and damage of epoxy resin to a certain extent. Also, the ZrB_2 fillers which are pulled out from matrix can act further as the abrasive particles, which makes abrasive wear the dominant wear mechanism finally.

The corresponding SEM images of the counterpart surfaces are presented in Figure 7. In Fig. 7a, it is clearly seen that the counterpart surface of pure epoxy is very rough and adhered with some darker epoxy resin (marked with arrows) which is mainly resulted from surface flaking off epoxy sample and being transferred to the surface of the steel ring during friction process, which further confirms the mechanism of adhesive

wear presented in Fig. 6a. While the surface of the counterpart sliding against epoxy composites filled with ZrB_2 particles is relatively smooth and has some clear furrows. Further, it can be found that there are tiny abrasive particles (marked with arrows) and less adhered resin on the counterpart surface. This indicates the dominance of the abrasive wear again and verifies the mechanism that we have described in Fig. 6b as well. Because of good adhesiveness of epoxy, the adhesive wear mechanism determined that the coefficient of wear K of epoxy resin could be enlarged by the shearing stress between the epoxy resin and the counterpart, which caused massive peeling loss. With the introduction of ZrB_2 particles, the mechanical performance of filling composites was enhanced and the adhesiveness of the epoxy composites was reduced, and thus the peeling loss from adhesion was also decreased.

The tribological performance of composites is also affected by its efficiency of heat dissipation[14]. The composites could fail once the temperature exceeds the failure temperature due to the accumulation of frictional heat. The experimental friction temperature between sample and counterpart was detected by an infrared thermometer once per half minute from 112 mins to 120 mins. The infrared ray irradiated the side of the friction pair during each friction process. The infrared detection area is shown in Fig. 1. The averaged friction temperature detected was about $290\text{ }^\circ\text{C}$ and $120\text{ }^\circ\text{C}$ corresponding to epoxy composites filled with 0 vol% and 5 vol% ZrB_2 particles. This difference of friction temperature could be attributed to the addition of ZrB_2 particles with high thermal conductivity. The thermal conductivities of epoxy composites could

be improved significantly with the addition of ZrB_2 fillers in our previous work[10]. In order to illuminate the thermal conductive status which affected the tribological performance of epoxy composites during the friction process, the frictional heat vector diagram obtained by the FEMs (method from the literature[15]) of epoxy composites filled with ZrB_2 particles is shown in Figure 8. It is shown that the values and densities of frictional heat flow vector of ZrB_2 particles are higher than those of epoxy matrix. It suggests that ZrB_2 particles can help to dissipate frictional heat efficiently in epoxy composites. Meanwhile, the epoxy composites filled with ZrB_2 particles presented lower CoF, which contributed to the decrease of frictional heat of ZrB_2 /epoxy composites generated during the friction and wear tests. These properties can prevent the degradation of tribological performance of epoxy composites caused by high friction temperature[6].

During the friction process, the asperities on the surface of epoxy resin were deformed and stucked to the surface of counterpart due to the load and the adhesiveness of epoxy resin. As the counterpart rotated, the asperities of the epoxy resin would be pulled out, which caused the adhesive wear of the epoxy resin. With the introduction of ZrB_2 particles, the exposed ZrB_2 particles could play the role of asperities on the surface of epoxy composites. Because of high elastic modulus and hardness of ZrB_2 ceramic, the deformation of the asperity on the epoxy composites was reduced. This contributed to the improvement of wear resistance and the reduction of adhesive wear of epoxy composites. According to previous studies[16], the micro-sized ceramic particles could

improve the modulus and strength of polymeric composites due to the anchor effect of ceramic particles. This implies that the addition of ZrB_2 particles into epoxy composites can result in the strengthening of mechanical performance of epoxy composites, and thus the tribological properties of filling composites are significantly improved. Some ZrB_2 particles fell off the epoxy composites during the friction process and still took part in the friction process. It resulted in the abrasive wear of epoxy composites. Therefore, it could be concluded that the main wear mechanism of epoxy composites was changed from the adhesive wear to the abrasive wear with the introduction of ZrB_2 particles. In addition, the lamellar crystal structure of ZrB_2 particle has low shear strength[17], which can lubricate effectively the friction pair. It was also contributed to further decrease of the CoF and coefficient of wear K of epoxy composites filled with ZrB_2 particles.

4. Conclusions

The tribological performance of in-situ epoxy composites filled with micro-sized ZrB_2 particles was investigated by the experimental and FEMs methods. The incorporation of ZrB_2 particles into epoxy matrix with a low content can significantly enhance the tribological performance of epoxy composites. The ZrB_2 particles content of 5 vol% is the optimum filler content in decreasing the friction coefficient and coefficient of wear K. The friction coefficients and coefficients of wear K of epoxy and its composites increase in varying degrees with the increase in the normal load and the sliding speed under the experimental load-speed conditions. The dominant wear

mechanism of epoxy composites is changed from adhesive wear to abrasive wear with the increase of ZrB_2 particle content in epoxy matrix. The high wear resistance and even dispersion of ZrB_2 particles in epoxy matrix are beneficial to prevent the further wear damages of epoxy resin matrix. FEM of frictional heat flow in the composites indicates that the thermal diffusion of epoxy composites is improved by the addition of ZrB_2 particles with higher thermal conductivity in epoxy matrix, which is finally contributed to enhancing tribological properties of epoxy composites.

Acknowledgements

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Figure Captions

Figure 1. The contact schematic diagram of wear tester and specimen.

Figure 2. The coefficient of friction and coefficient of wear K of epoxy composites filled with 0 vol%, 1 vol%, 3 vol%, 5 vol% and 7 vol% ZrB₂ particles at 250 N and 0.42 m/s.

Figure 3. The coefficient of friction and coefficient of wear K of epoxy composites filled with 0 vol%, 1 vol%, 3 vol%, 5 vol% and 7 vol% ZrB₂ particles at 150 N and 0.42 m/s.

Figure 4. The coefficient of friction and coefficient of wear K of epoxy composites filled with 0 vol%, 1 vol%, 3 vol%, 5 vol% and 7 vol% ZrB₂ particles at 250 N and 0.84 m/s.

Figure 5. The coefficient of friction of epoxy composites filled with (a) 0 vol%, (b) 1 vol%, (c) 3 vol% and (d) 5 vol% ZrB₂ particles as a function of sliding time at 250 N and 0.42 m/s.

Figure 6. SEM images of the surface morphology of worn (a) epoxy resin, (b) epoxy composites filled with 5 vol% ZrB₂ particles at 250 N and 0.42 m/s, and (c) high magnified SEM micrograph of Fig.(b) after 120 min friction process.

Figure 7. SEM images of worn surfaces of counterpart sliding against (a) epoxy resin and (b) epoxy composites filled with 5 vol% ZrB₂ particles at 250 N and 0.42 m/s after 120 min friction process.

Figure 8. Frictional heat flow vector diagrams of epoxy composites filled with 5 vol%

ZrB₂ particles.

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Table 1. Chemical composition of ZrB₂ particles (wt.%)

ZrB ₂	ZrO ₂	B ₂ O ₃
> 99.5	< 0.45	< 0.05

Table 2. Element composition of Steel 45 (wt.%)

C	Si	Mn	P	S
0.42-0.45	0.17-0.37	0.58-0.80	≤0.040	≤0.040

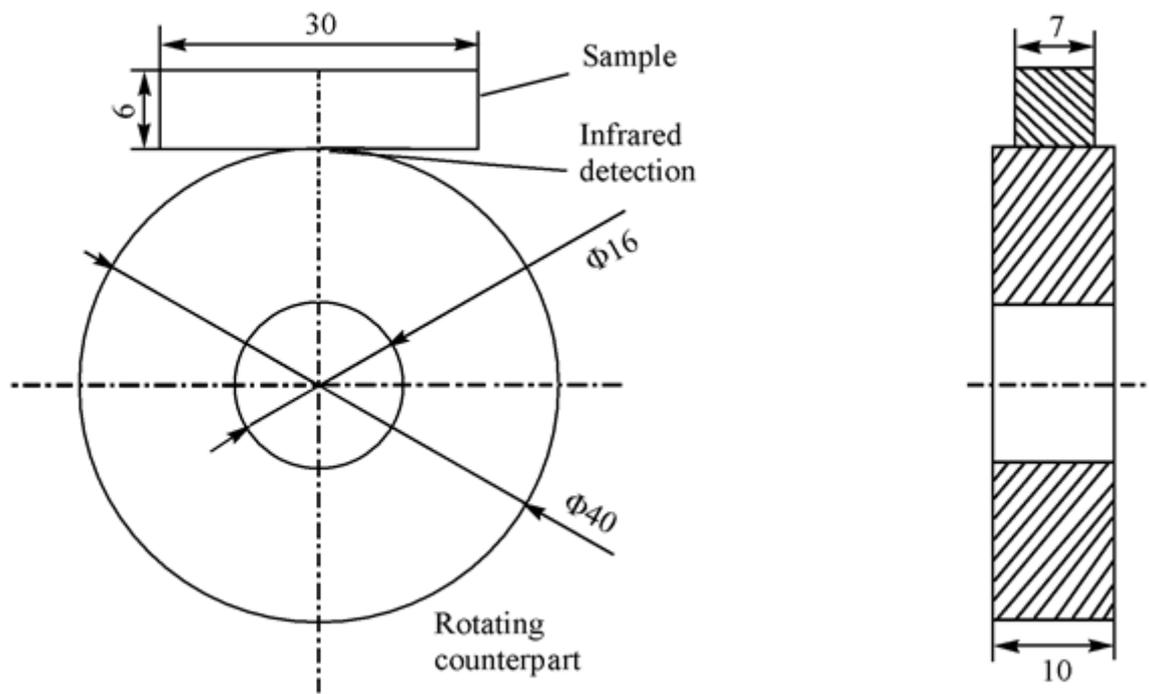


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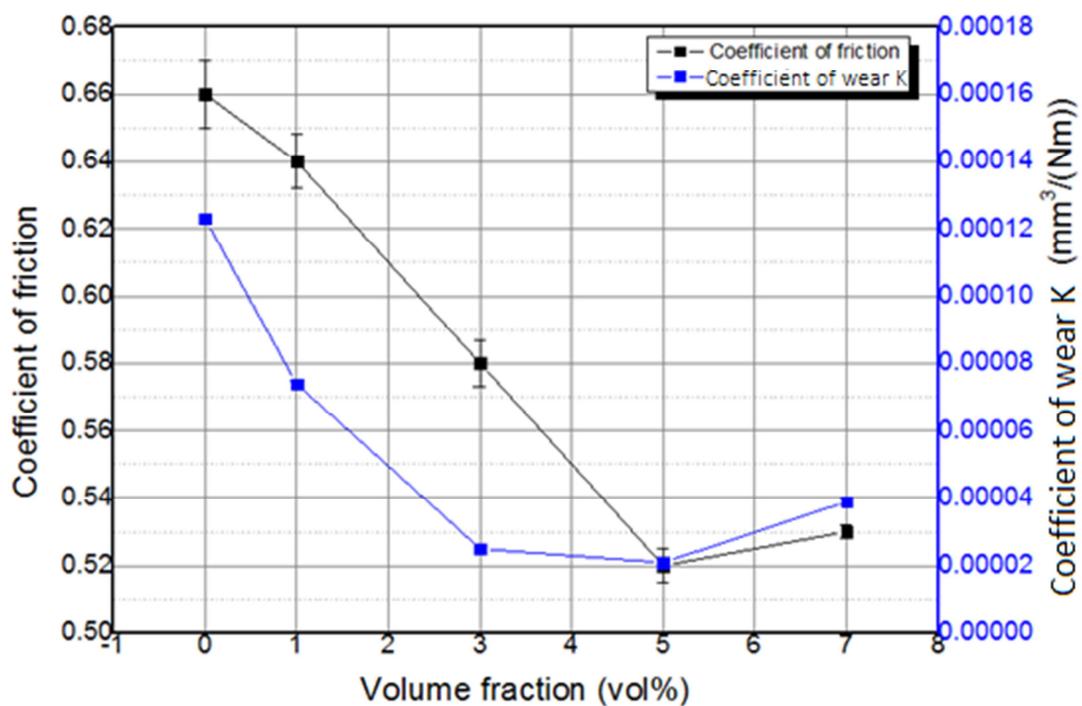


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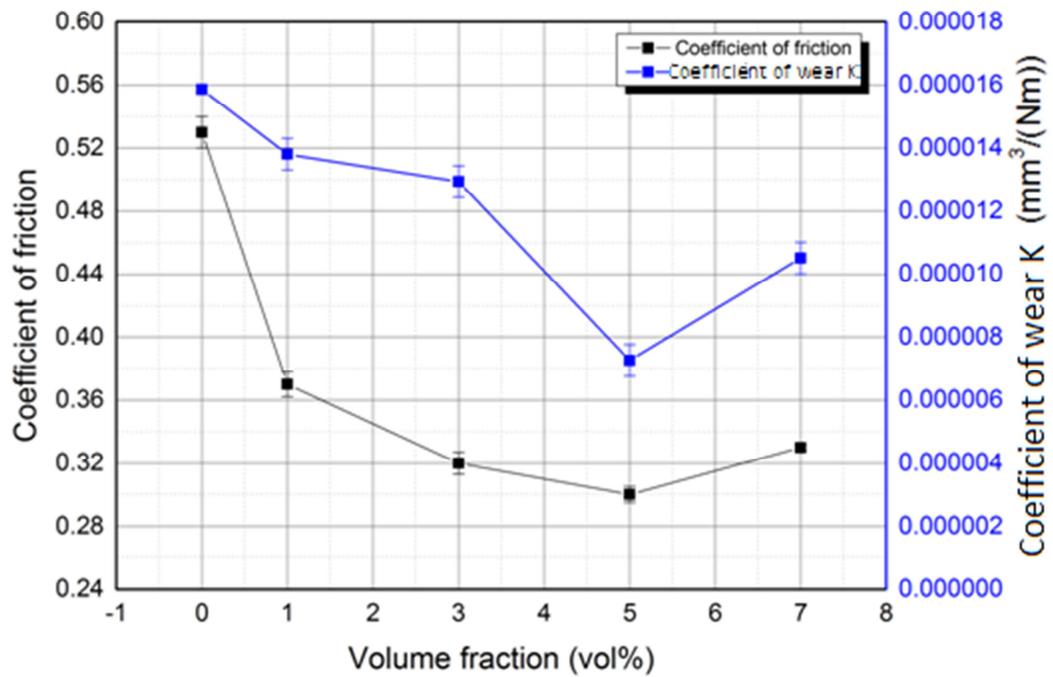


Figure 3. The coefficient of friction and coefficient of wear K of epoxy composites filled with 0 vol%, 1 vol%, 3 vol%, 5 vol% and 7 vol% ZrB₂ particles at 150 N and 0.42 m/s.

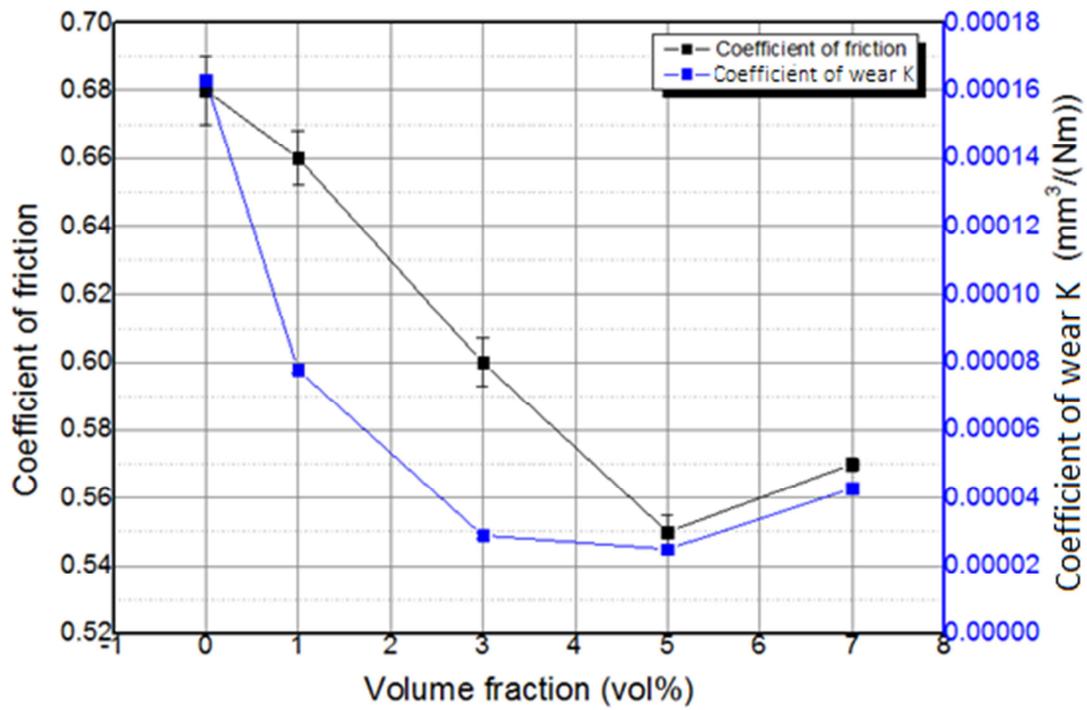


Figure 4. The coefficient of friction and coefficient of wear K of epoxy composites filled with 0 vol%, 1 vol%, 3 vol%, 5 vol% and 7 vol% ZrB₂ particles at 250 N and 0.84 m/s.

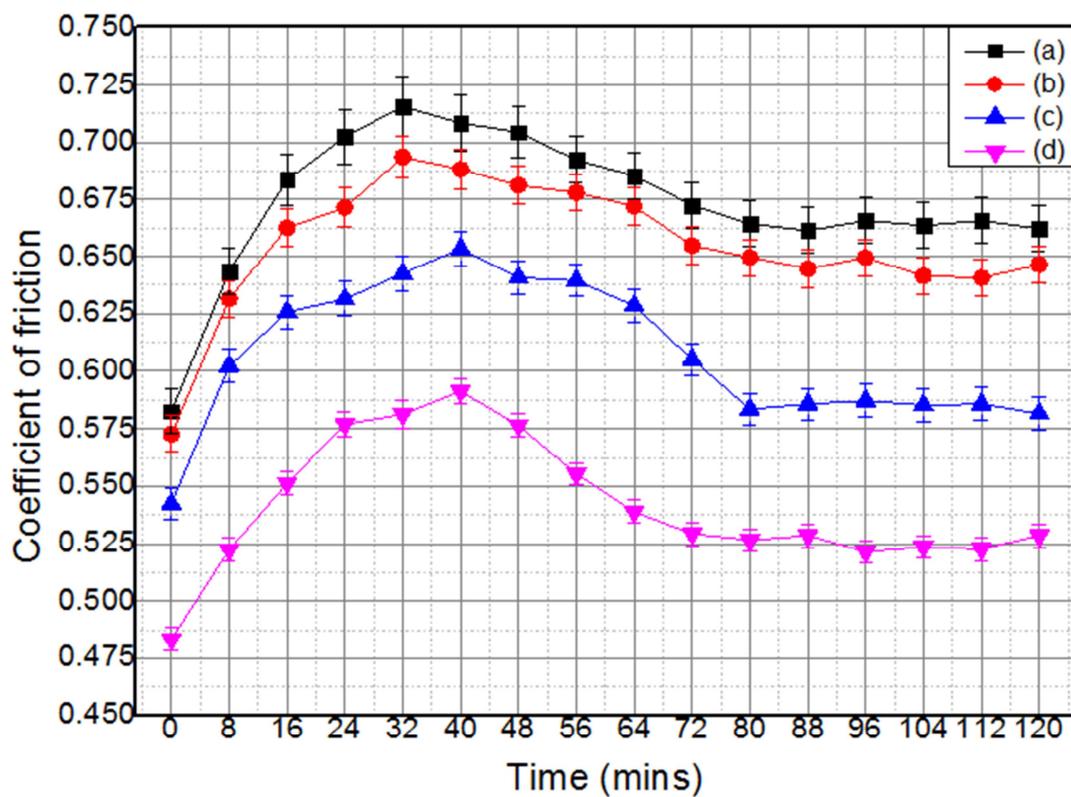


Figure 5. The coefficient of friction of epoxy composites filled with (a) 0 vol%, (b) 1 vol%, (c) 3 vol% and (d) 5 vol% ZrB₂ particles as a function of sliding time at 250 N and 0.42 m/s.

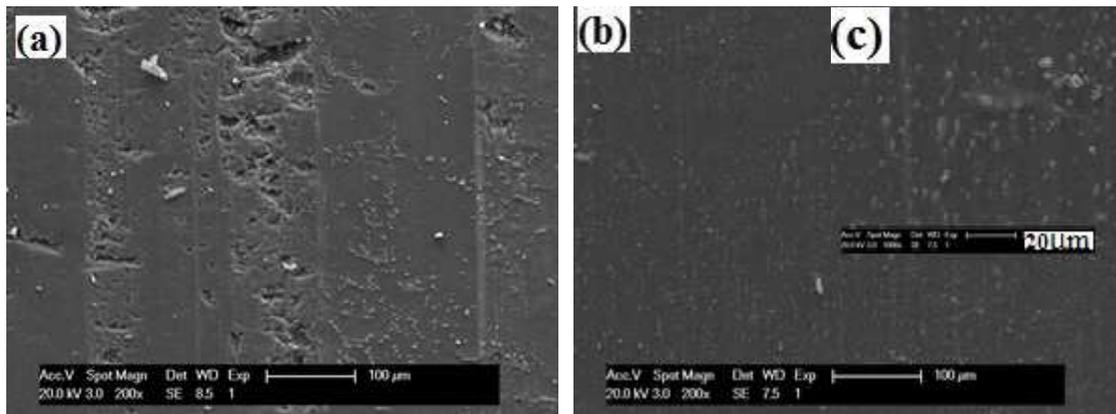


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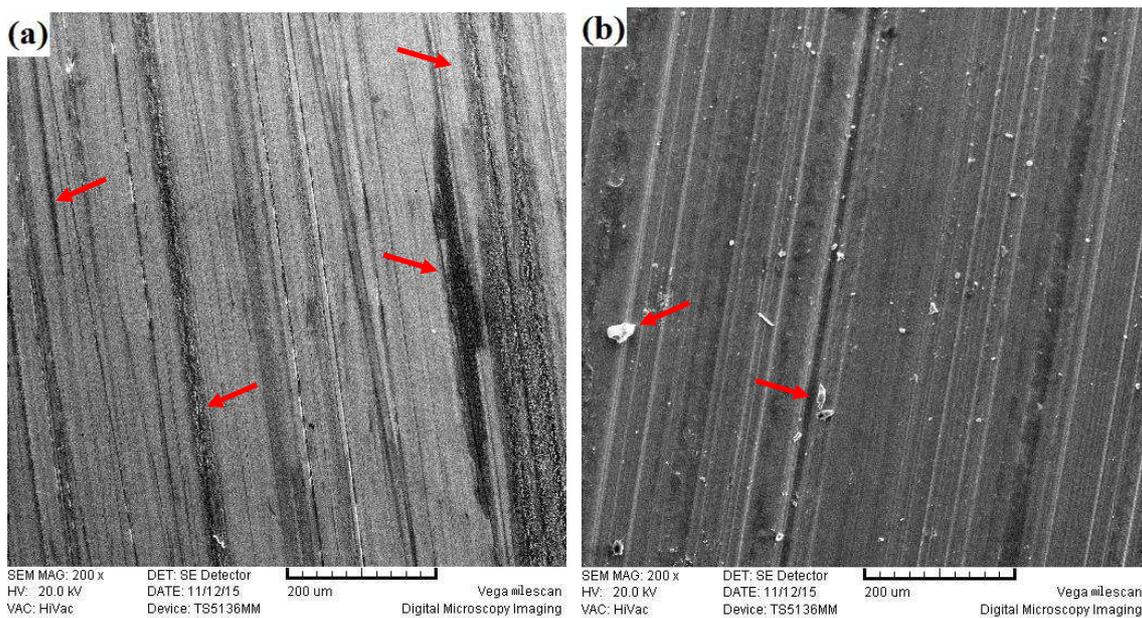


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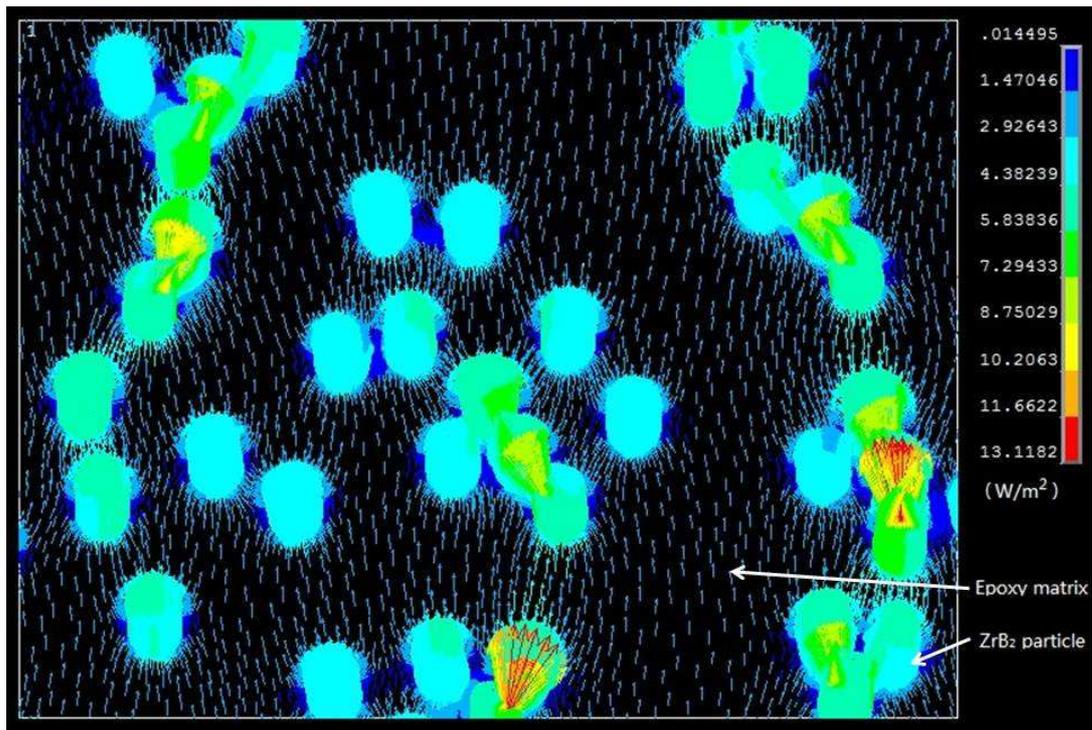


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