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Wear and friction of epoxy based nanocomposites with silica nanoparticles and wax-containing microcapsules



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ABSTRACT

When subjected to wear by metal surfaces, epoxy normally exhibits rather high frictional coefficients (COF), causing high wear rate, exacerbated noise and frictional heat, thus limiting their service life. Liquid lubricants are known to reduce friction effectively, but they can be easily lost in operation. Therefore, encapsulating them is a proper solution to this issue. Herein, tribological properties of epoxy composites filled with wax-containing microcapsules (WMCs) and/or silica nanoparticles were investigated systematically. Results exhibited a tremendous decrease in the specific wear rates (W_s) and COF for the silica/WMC/epoxy ternary nanocomposites, specially three orders of magnitude reduction in W_s and a 10-fold reduction in COF were observed in specific test conditions. The wear mechanism was investigated based on worn surfaces and transfer films developed during wear tests. Furthermore, incorporating hybrid fillers negligibly deteriorates the mechanical properties of epoxy. Hence, the combination of rigid nanoparticles with WMCs is an appropriate choice when excellent tribological and mechanical properties are required.

1. Introduction

Polymer composites filled with solid or liquid lubricants have been frequently applied for wear and friction applications. The solid lubricants, such as graphite, graphene nanoplatelets and polytetra-fluoroethylene (PTFE), can reduce the wear rate of polymer composites by the formation of thin and uniform transfer films, which consequently, reduces the adhesion between the polymer and counterpart [1–9]. Generally, the liquid lubricants result in lowered wear rates and frictional coefficients (COF) more significantly [10–13], compared to the solid ones.

In practice, the depletion of liquid lubricants is a serious problem [11–13], which reduces the service time and thus limits their applications. In view of these, some works [14–21] provide a solution to the above problem by incorporating of liquid-lubricant-containing microcapsules into the polymers. The liquid lubricants are sealed in the micron-sized polymer or ceramic shells (i.e. microcapsule). During wear, the shells are broken by frictional force, thereby makes the lubricants release gradually from microcapsules onto the surfaces of composite

sample and counterface, and take effects.

However, a large number of microcapsules usually cause loss to the basic mechanical properties of polymers (e.g. hardness, modulus, and failure strength) [15–22]. A possible solution to this problem is to add some hard fillers to the microcapsule-containing polymers to recover these losses. So far, only a few works have been reported on the tribological properties of the ternary composites having both microcapsule and hard fillers. For example, Khun et al. [15,17] recently reported that short carbon fibers (SCFs) can increase the hardness of epoxy composites filled with wax-containing microcapsules (WMCs), and decrease the friction and wear of the samples simultaneously through lubricating effects of SCFs. In another work, they mentioned that the multiwall carbon nanotubes (MWCNTs) behaved in a similar way when improving hardness, modulus and wear resistance in the ternary composite samples [20].

In our previous work [23–27], we found that sol-gel silica nanoparticles, owing to their agglomerate-free dispersion and also a high surface area, can improve the basic mechanical properties (stiffness, strength, and fracture toughness) of epoxy materials as well as the wear

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resistance of acrylate-based polymers, as the nanoparticle content increases. It is, therefore, expected to tailor the wear resistance and other basic mechanical properties of the epoxy polymer by combining the WMCs with the silica nanoparticles together. Wear tests were carried out using a ball-on-disc mode at various testing conditions; the related wear mechanisms and the roles played by the different fillers were discerned by scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

2. Experimental

2.1. Materials

The nano-modified epoxy resin (Nanopox F400, Hanse Nanoresins, Evonik Industries, Germany) was used as the master batch, which consisted of 40 weight percent (wt.%) surface-modified nanosilica particles, $\sim\!25\,\mathrm{nm}$ in average size. The DGEBA neat epoxy resin (E51, equivalent weight = 185 g/eq) used was supplied by, Wuxi Resin Factory of Bluestar New Chemical Materials Co., Ltd., China. The curing agent (Albidur HE600) was a mixture of 4-methylhexahydrophtahlic anhydride (MHHPA, Puyang Huicheng Chemicals Co., Ltd., China) and N, N-benzyl dimethylamine (BDA, Sino-pharm Chemical Reagent Co., Ltd., China) at a weight ratio of 100:1. Their detailed information can be found in our previous work [28,29]. The WMCs were 200–300 μm in diameter; their information can be found in Refs. [15,17]. All materials were used without any further purification.

2.2. Sample preparation

For the preparation of silica/epoxy binary composites samples, the master batch "Nanopox F400" was diluted with a different mass of the neat epoxy resin E51; the mixtures were degassed in a vacuum oven at 60 °C for 30 min before a stoichiometric amount of the curing agent was added. The mixture was subsequently heated in an oil bath at 60 °C and mechanically stirred for 20 min prior to degassing in a vacuum oven for 15 min. Afterward, the degassed mixture was poured into a preheated steel mold and cured in a furnace. A four-step curing schedule was used: 30 min at 90 °C, 60 min at 120 °C, 30 min at 140 °C and finally 120 min at 160 °C, respectively. The cured samples were then allowed to cool down slowly to room temperature, then polished by a surface grinder on both sides to ensure sufficient surface flatness. The nomenclature of samples is as follows: Letter 'E' denotes the epoxy matrix; the first and second numbers stand for the weight fraction of silica nanoparticles, and/or WMCs, respectively. For example, 'E23_10' represents the ternary epoxy-based sample containing 23 wt% of nanosilica and 10 wt % WMCs.

TEM images of the silica/epoxy binary composites samples (Fig. 1) show the homogeneous dispersion levels of the nanoparticles in the epoxy matrix at low and high filler fractions, similar to the results in Refs. [24–27,30,31]. With the similar processes, WMC/epoxy and silica/WMC/epoxy ternary composite samples were also prepared.

2.3. Characterization

The Vickers hardness (H_v) measurements were performed using a Tukon 2500 automated Knoop/Vickers hardness tester. The hardness values were measured immediately after the indentation with a 0.02 kg load for 15 s. At least 3 specimens were used for each group of nanocomposites and 5 indentations were performed for each specimen.

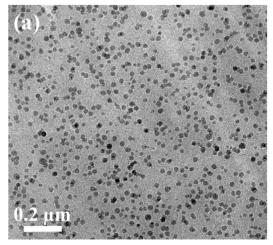
Three-point bending tests were performed at room temperature (25 °C) on a Zwick universal testing machine at a constant crosshead speed of 0.5 mm/min according to the standard DIN EN ISO 178. The displacement of each specimen during the test was accurately measured by an extensometer. Moduli were calculated by considering the load in the 0.05–0.25% strain range. All presented data corresponds to the average of at least five measurements [25].

The glass transition temperature (T_g) was measured with a DSC (TA Instruments Q2000, USA) in temperature modulation mode under a nitrogen atmosphere (50 mL min $^{-1}$). Approximately 10 mg of the samples were sealed in aluminum pans. Modulated DSC measurements were carried out in the temperature range of -20 to 200 °C at a heating rate of 2.5 K min $^{-1}$ with an amplitude of 0.5 K and the period of 60 s. Three samples of each composition were tested and their T_g s were obtained from the reversing heat capacity (C_p) curves [29].

Transmission electron microscopy (TEM, Tecnai G2 F20 U-TWIN, United States) and field emission scanning electron microscopy (SEM, Hitachi S-4800, Japan) were utilized to observe the homogeneity of nanosilica particles in the silica/epoxy nanocomposites and the transfer films formed on the steel ball counterparts.

2.4. Wear and friction test

Friction and wear behaviors of the samples were measured with universal tribometer (UMT-3, Bruker Corporation) under dry sliding condition through a ball-on-disc configuration, as illustrated in Fig. 2. The ball was made of 302 stainless steel with a diameter of 4.0 mm, a surface roughness, Ra, of 90 nm and hardness of HRC 39. Before the wear tests, the specimens were polished with grit papers (No. 2000). The surface roughness of the polished surfaces was in the range of 90–120 nm. The diameter of the sliding track was 20 mm. The wear tests were performed under ambient conditions at different sliding velocities of 0.08–0.32 m/s, normal forces of 2–8 N, and test duration of



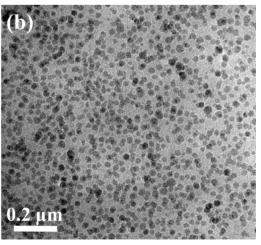


Fig. 1. TEM micrographs of ultra-thin sections for the silica/epoxy nanocomposite samples with (a) 5 wt% and (b) 23 wt% silica nanoparticles.

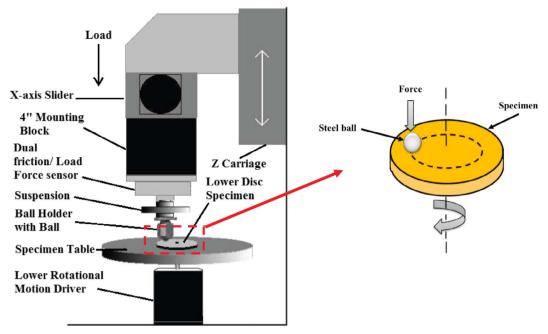


Fig. 2. Schematic illustration of UMT3 tribometer for the ball-on-disc sliding test.

3 h or 1300 m. The transient frictional coefficients were collected at a frequency of 1000 Hz and the average values of every 100 data points were used to assess changes in the transient frictional coefficient. Volume loss of the specimen, ΔV , was measured by a confocal laser microscope (Olympus 3D LEXT OLS4000) after test using a stitching method. The specific wear rate, $W_{\rm s}$, which describes the wear performance under the chosen conditions for a tribosystem, was calculated according to

$$W_s = \frac{\Delta V}{F_N d} \text{ [mm}^3/\text{N} \cdot \text{m]}$$
(1)

where F_N is the normal force applied on the steel ball during sliding; d is the total sliding distance. For each test condition, at least three repetitive tests were performed.

3. Results and discussion

3.1. Morphologies of microcapsules

Fig. 3a presents the morphologies of a single WMC observed under SEM. The WMC exhibits very rough surface and its diameter is measured to be around 300 μm . Fig. 3b shows the core-shell structure of the WMC and the shell thickness is measured to be around 10 μm . Element analysis (EDS) in Fig. 3c indicates that the WMC contains silicon, oxygen and carbon atoms, thus confirming that the shell is made of silicon dioxide, as claimed by the supplier [14,18]. Fig. 3d shows the fracture surface of a WMC/epoxy sample for three-point bending test. The breakage and pullout of WMCs can be clearly observed. Fig. 3e presents the melting point ($T_{\rm m}$) of the wax inside microcapsule is about 35 °C, which does not change obviously after compounding the WMCs with epoxy resin and/or silica nanoparticles.

3.2. Mechanical and thermal properties of the samples

The mechanical properties and glass transition temperature ($T_{\rm g}$) of the epoxy composite samples were systematically investigated and the results are summarized in Fig. 4. The steady improvements in flexural modulus, flexural strength, and Vickers hardness can be clearly observed with increasing filler fraction for the epoxy composite samples containing nanosilica particles only. The same trends have also been

discussed in Refs. [23,25,26]. Comparatively, these mechanical properties of epoxy samples containing only WMCs decline sharply with increasing the amount of WMCs, which is mainly due to two factors: (i) the WMCs are very brittle and cannot bear any load; (ii) the large size of WMCs (300 μ m) is apt to cause high level of stress concentration around WMCs and subsequent pre-mature fracture. A similar trend has been found in other epoxy composites with microencapsulated fluid [14,21,22]. However, when combining both silica and WMCs together, the mechanical properties of composite samples (especially for the flexural modulus and Vickers hardness) can be restored more or less, in some cases, nearly reach those of neat epoxy sample. For example, hardness and flexural strength of the epoxy sample with 23 wt% of silica and 10 wt% of WMCs increase by 72% and 100%, respectively, in comparison with those of the sample having only 10 wt% of WMCs.

The glass transition temperature ($T_{\rm g}$) of the samples measured by modulated DSC, is shown in Fig. 4e. The $T_{\rm g}$ of the silica/epoxy binary samples decreases gradually as a consequence of a continuous rise in silica fraction; this can be ascribed to the plasticizing effect of nanosilica [25,32]. Likewise, the samples containing solely WMCs show the decreased $T_{\rm g}$ with the rise in WMC fraction. The possible reason is that during mechanical mixing, some of the WMCs may be broken by the mechanical impact and accordingly, a small amount of wax can be released from WMCs, acting as a plasticizer, which obviously reduces the $T_{\rm g}$. In the case of silica/WMC/epoxy ternary samples, the decrease in $T_{\rm g}$ becomes more obvious due to the coupling effect as mentioned above.

3.3. Wear and friction properties of the samples

First, we tested the wear and friction properties of the samples at the normal force of $4\,\mathrm{N}$, sliding velocity of $0.12\,\mathrm{m/s}$ and sliding time of $3\,\mathrm{h}$. The representative curves of COF vs. sliding time for the samples are shown in Fig. 5a. At the beginning of sliding wear (running-in), the neat epoxy sample shows the dramatic increase in COF value, which is likely due to the gradually increased real contact area between friction pairs. After the running-in stage, the COF curve for neat epoxy oscillates around the value of 0.6, which is due to the repeated adhesion and separation of the transfer film and debris (generated by the neat epoxy sample during wear) on the steel ball [33,34]. For the epoxy sample with nanosilica only, it develops generally similar COF curve and value,

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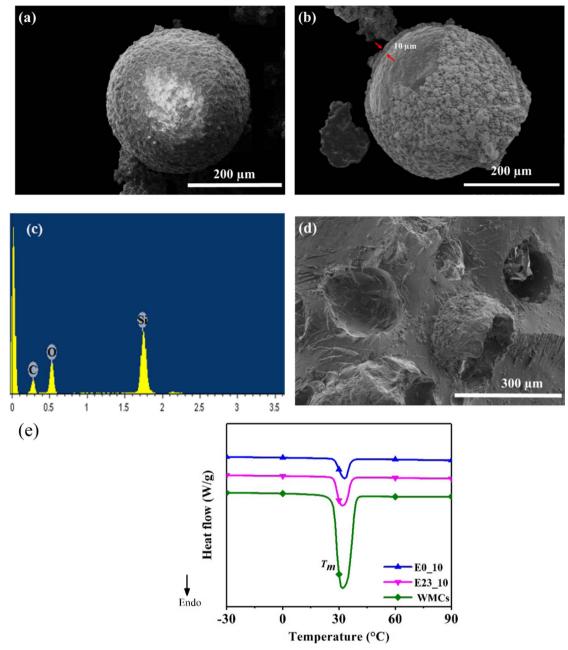


Fig. 3. Morphologies of microcapsules containing wax lubricant (WMCs): (a) SEM of a single WMC, (b) the core-shell structure of a WMC indicated by red arrows, (c) EDS of WMCs, (d) the fracture surface taken from WMC/epoxy sample for three-point bending test, and (e) the melting points of wax inside WMCs before and after compounding the WMCs with epoxy resin. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

compared to the neat epoxy, even though the COF value is more stable for most of the sliding time. For the epoxy samples containing WMCs, their COF curves become very stable and smooth without obvious oscillation, and the COF values dramatically decrease to $\sim\!0.1\text{--}0.2.$ Among them, the silica/WMC/epoxy ternary sample has the lowest COF value during the wear stage.

Fig. 5b and c give a comparison of the wear and friction properties between the four groups of the samples. Fig. 5b shows that the specific wear rate (W_s) values increase slightly in the ascending order with increasing of silica weight fraction, which is probably due to the fact that during wear, the hard silica nanoparticles may act as the abrasives that may result in some scratch and cutting effects on the relatively softer surface of composite samples; on the contrary, the W_s values show dramatic decrease after addition of WMCs only; it is because the wax can be released onto the contact area during wear, thus forming the thin

and continuous transfer film on the steel counterface, resulting in polymer-polymer contact instead of the original steel-polymer contact mode. For the silica/WMC/epoxy ternary samples, they also show relatively lower W_s values with increasing of the hybrid filler fraction; the sample 'E23_10' (with 23 wt% nanosilica & 10 wt% WMCs) exhibits the lowest W_s among all samples studied, c.a. three orders of magnitude smaller, as compared to that of the neat epoxy sample tested at the same testing conditions. Regarding the COF shown in Fig. 5c, all filled samples show decreased COF values more or less, compared to the neat epoxy. For the epoxy samples containing silica nanoparticles only, the reduction in COF may be ascribed to the rolling effects of the spherical nanoparticles, even though their W_s increases slightly (Fig. 5b); for the samples containing WMCs, their COF values decrease obviously, which is due to the formation of transfer films; once again the sample 'E23_10' has the smallest COF value of 0.074 among all samples studied,

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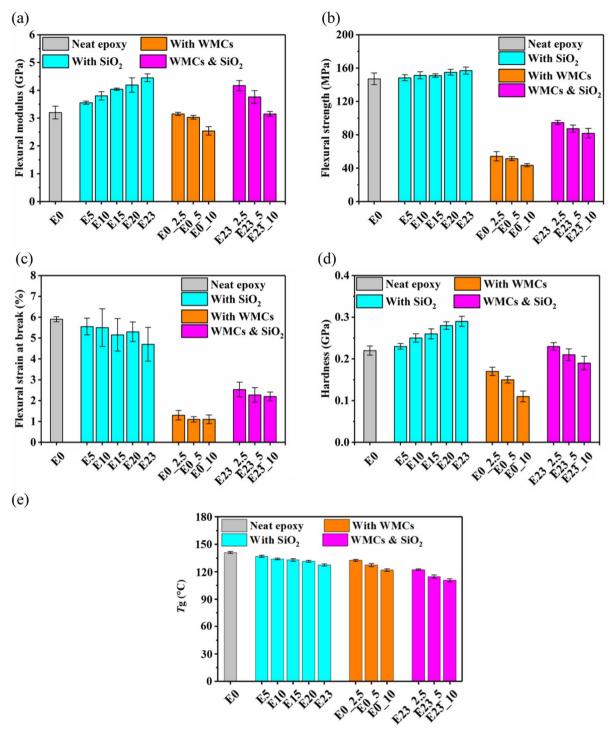


Fig. 4. Mechanical properties and glass transition temperatures (T_g) of the epoxy composite samples: (a) flexural modulus, (b) flexural strength, (c) flexural strain at break, (d) Vickers hardness and (e) T_g .

comparatively, the neat epoxy shows the COF value of 0.72.

Based on the results in Fig. 5, we selected four representative samples and studied their wear and friction properties further at different wear conditions. The results are shown in Figs. 6 and 7. It is clear from Fig. 6 (W_s vs. sliding velocity or normal force) that for a given sample, increasing the sliding velocity generally causes increased W_s , whereas the influence of the normal force on W_s seems complicated; this is because that the W_s is not only dependent on the normal force but also volume loss, as indicated in Eq. (1). With an increase in sliding velocity and/or normal force, ample frictional heat energy accumulates around contact area, making the polymer composite samples softer at

elevated temperatures and easier deformation during wear [34]. This enhanced deformation can reduce wear and friction between frictional pairs, as a result, the COF values fall with the sliding velocity and/or normal force (Fig. 7) for a given sample.

When comparing the tribological properties between different samples from Figs. 6 and 7, the general tendency seems: once the WMCs are incorporated to the epoxy, both W_s and COF values decline significantly, which show much better wear resistance over the neat epoxy and silica/epoxy samples. Moreover, at a majority of the test conditions, the silica/WMC/epoxy ternary samples behave better than the WMC/epoxy binary samples, even though some ambiguous results are

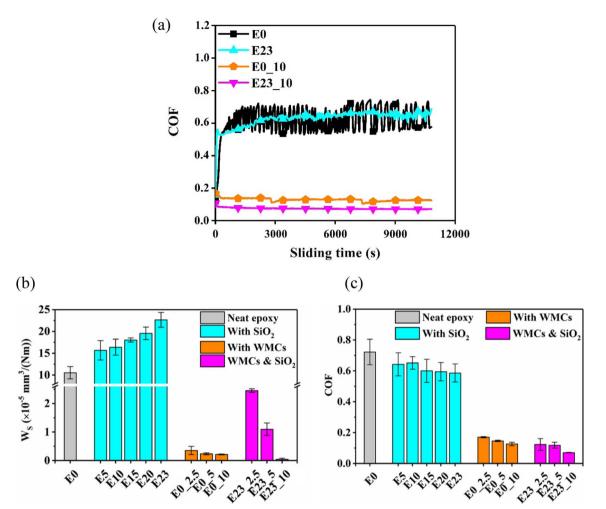


Fig. 5. Representative plots of COF vs. sliding time of epoxy composite samples (wear test conditions: normal force = 4 N, sliding velocity = 0.12 m/s, sliding time = 3 h).

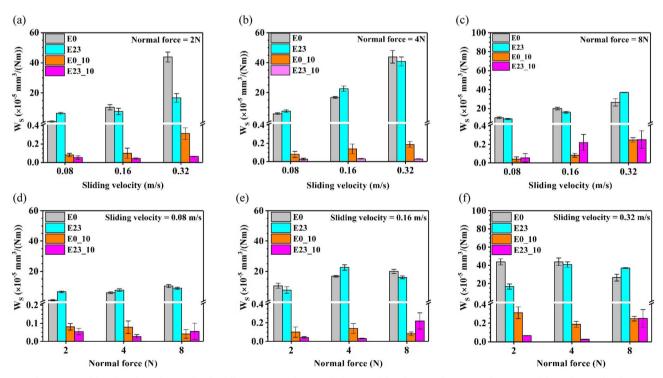


Fig. 6. Specific wear rate of the selected samples tested under different wear conditions with the same sliding distance of 1300 m: (a, b, c) W_s vs. sliding velocity, (d, e, f) W_s vs. normal force.

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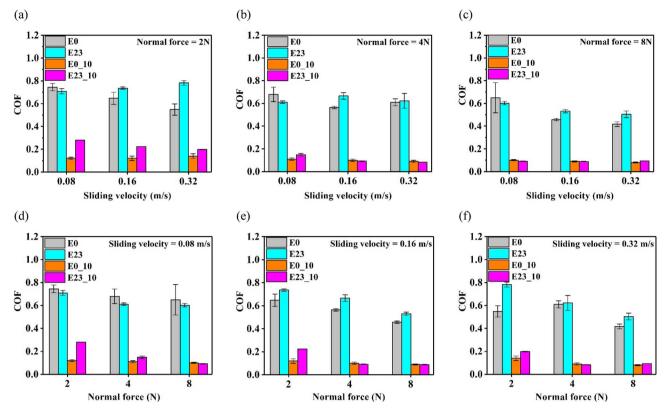


Fig. 7. Frictional coefficient of the selected samples tested under different wear conditions with the same sliding distance of 1300 m: (a, b, c) frictional coefficient vs. sliding velocity, (d, e, f) frictional coefficient vs. normal force.

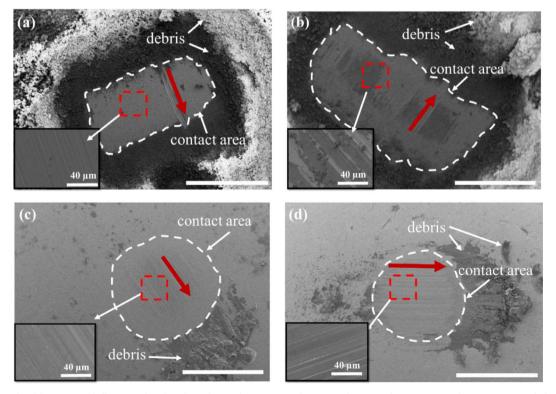


Fig. 8. SEM micrographs of the worn steel balls against the selected samples: (a) the neat epoxy, (b) epoxy with 23 wt% silica, (c) epoxy with 10 wt% WMCs and (d) epoxy with 10 wt% WMCs & 23 wt% silica. The thick red arrows indicate the sliding directions during wear tests. The scale bar is $500 \, \mu m$ (wear test conditions: normal force $= 4 \, N$, sliding velocity $= 0.12 \, m/s$, time $= 3 \, h$). The insets are the highly-magnified views of the red-dotted square areas. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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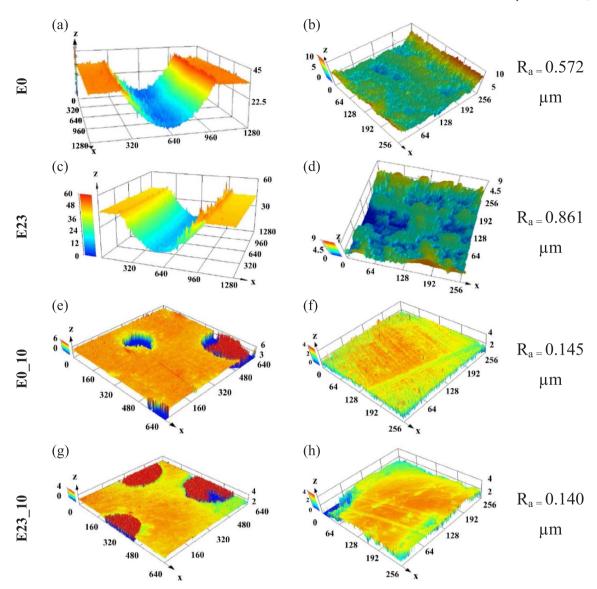


Fig. 9. 3D laser microscope images of the worn surfaces for the selected samples: the left column (a, c, e, g) showing the low magnification images; the right column (b, d, f, h) showing the high magnification images, together with arithmetic mean values of surface roughness, R_a . (wear test conditions: normal force = 4 N, sliding velocity = 0.12 m/s, time = 3 h). All the units specified above are in um.

observed in some cases.

3.4. Analysis of the worn surfaces of the samples

Fig. 8 depicts SEM micrographs of the worn steel balls for the selected samples. Previous works showed [35,36] that the development of a transfer film is typically due to the adhesion and interlocking of polymer fragments into metal asperities during sliding. Some inorganic particles can enhance bonding between the transfer film and metallic counterpart, leading to improved wear resistance; but some fillers can generate more discontinuities in the same material, showing opposite effects. Fig. 8a shows that no obvious transfer film is observed for the neat epoxy sample, while with the addition of silica nanoparticles inside the epoxy matrix, a non-continuous thick transfer film is observed (Fig. 8b); the highly-magnified view of the red-dotted square area is shown in the inset of Fig. 8b, clearly presenting this discontinuity. This kind of transfer film can be easily detached during the test, which may cause slightly increased W_s and slightly decreased COF values in many cases (refer to Fig. 5). As seen in Fig. 8c and d, the addition of WMCs develop thin and continuous transfer films on the steel ball surfaces, which results in good wear and friction resistances. On the other hand,

due to the insufficient protection by the transfer films, the neat epoxy and silica/epoxy samples are found to generate a large amount of debris accumulating around the contact areas (Fig. 8a and b); on the contrary, there are only small amounts of debris formed during wear for the WMC-containing samples, this may be ascribed to the fact that the high-quality transfer films can alleviate the cutting and scratching effects from the asperities of the steel balls.

Fig. 9 presents the worn surfaces of epoxy composite samples after wear tests, which can supply further information for wear and friction mechanisms. Fig. 9(a, c, e, and g) shows the worn surfaces of the four representative samples at low magnification observed by a confocal laser microscope. The worn surface of the neat epoxy sample is rough with flaky zones, suggesting the adhesive and fatigue wear behaviors (Fig. 9a); the epoxy sample containing nanosilica only, shows the similar features as the neat epoxy (Fig. 9c); comparatively, the two epoxy samples containing WMCs show relatively smooth worn surfaces, even though some holes which were formed due to the debonding of microcapsules are clearly recognized (Fig. 9e and g). Furthermore, Fig. 9(b, d, f, and h) shows the high magnification images of the neat epoxy and its composite samples after the wear test; the arithmetic mean values of surface roughness (R_a) of the worn surfaces are also

given, which may reflect the different worn levels of the samples during wear tests. Higher $R_{\rm a}$ may suggest higher levels of abrasive effects during wear. As seen, all the four samples show similar $R_{\rm a}$ values (c.a. 0.090–0.120 µm) before wear. After wear, the neat epoxy and 23 wt% silica/epoxy binary samples show significantly increased $R_{\rm a}$ values (0.572 and 0.861 µm, respectively); in the case of WMC-containing samples, the increase in $R_{\rm a}$ values are very small (0.145 and 0.140 µm, respectively). Therefore, it is reasonable to consider that the first two samples underwent severe abrasion wear than the last two ones.

4. Conclusions

In this work, epoxy and its composites containing silica nanoparticles and/or WMCs were prepared and their basic mechanical and tribological properties were studied. The results indicated that the combination of a proper ratio of silica nanoparticles and WMCs improved the wear resistance (in terms of specific wear rate, W_s) and simultaneously decreased the COF of epoxy materials. For instance, three orders of magnitude reduction in W_s (1.05 × 10⁻⁴–4.39 × 10⁻⁷ mm³/ Nm) and a 10-fold reduction in COF (0.72-0.07) were achieved for the silica/WMC/epoxy ternary composite samples as compared to those of the neat epoxy, tested at a normal force of 4N and a sliding velocity of 0.12 m/s. The WMC-containing samples (binary and ternary samples) were found to develop thin and continuous transfer films on the steel ball surfaces, which are pertinent to the wear and friction reductions. Moreover, the hybrid fillers also led to a good balance among the basic mechanical properties (such as stiffness, failure strength, failure elongation as well as hardness). In general, the addition of these hybrid fillers can be taken into consideration when the polymers having both the excellent mechanical and tribological properties are designed.

Acknowledgements

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