N. W. Khun
School of Mechanical
and Aerospace Engineering,
Nanyang Technological University,
50 Nanyang Avenue,
Singapore 639798, Singapore

H. Zhang
School of Mechanical
and Aerospace Engineering,
Nanyang Technological University,
50 Nanyang Avenue,
Singapore 639798, Singapore

C. Y. Yue
School of Mechanical
and Aerospace Engineering,
Nanyang Technological University,
50 Nanyang Avenue,
Singapore 639798, Singapore

J. L. Yang[1]
School of Mechanical
and Aerospace Engineering,
Nanyang Technological University,
50 Nanyang Avenue,
Singapore 639798, Singapore
e-mail: mjlyang@ntu.edu.sg

Self-Lubricating and Wear Resistant Epoxy Composites Incorporated With Microencapsulated Wax

Self-lubricating and wear resistant epoxy composites were developed via incorporation of wax-containing microcapsules. The effects of microcapsule size and content and working parameters on the tribological properties of epoxy composites were systematically investigated. The incorporation of microcapsules dramatically decreased the friction and wear of the composites from those of the epoxy. The increased microcapsule content or the incorporation of larger microcapsules decreased the friction and wear of the epoxy composites due to the larger amount of released wax lubricant via the rupture of microcapsules during the wear test. The friction of the composites decreased with increased normal load as a result of the promoted wear of the composites and the increased release of the wax lubricant. [DOI: 10.1115/1.4026941]

Keywords: epoxy composite, microencapsulated wax, self-lubrication, friction, wear

1 Introduction

A tremendous interest is increasingly raised in scientific and industrial communities to apply polymer composites (PCs) as structural materials for aerospace, automotive, and chemical industries since PCs can provide various advantages such as high chemical resistance, easy manufacturing, and light weight alternative to traditional metallic materials [1,2]. A number of the above mentioned applications are related to tribological components, such as gears, cams, wheels, bearings, and seals, where self-lubricating of polymers is of special advantage [3,4]. Therefore, tailoring of their properties with special additives is important to make PCs promising in industrial applications.

Zhang et al. [5] reported that PCs could have a much better tribological performance under lubrication by liquid paraffin than under dry sliding. It is clear that a liquid lubricant can improve the tribological performance of PCs. However, a difficulty in retaining a liquid lubricant on rubbing surfaces for a long run may limit the application of materials. In addition, external lubrication on rubbing surfaces may limit the application of materials too, due to the degradation of the materials caused by absorption and osmosis of the lubricant into the materials [5–7]. It was reported [1,8] that incorporation of microencapsulated lubricant oil in composites formed self-lubricating composites for wear or friction reducing applications. Recently, Khun et al. [9] reported that the friction coefficients of silicone composites filled with wax-containing microcapsules were significantly lower than that of pure silicone as the friction coefficient of the silicone composites apparently decreased with increased microcapsule content. Besides, Khun et al. [10] explored the tribological properties of epoxy composites incorporated with microencapsulated mixture of wax lubricant and multiwalled carbon nanotubes (MWCNTs) and found that the incorporation of microcapsules dramatically decreased the friction and wear of the composites due to the combined lubricating effects of released wax lubricant and MWCNTs. Therefore, microencapsulation of a liquid lubricant and then incorporation of the formed microcapsules in polymer matrices would be a promising solution because the lubricant released from broken microcapsules caused by surface wear during repeated rubbing could lubricate rubbing surfaces. In addition, the size and content of wax-containing microcapsules and the working parameters used during the tribological test could influence the friction and wear of PCs, which need to be systematically investigated. The fundamental understanding of a correlation between the incorporation of microencapsulated wax lubricant in PCs and their self-lubricating performance is essential for their successful tribological applications.

In this study, wax-containing microcapsules with two different size ranges of 38–63 and 63–90 μm in diameter were prepared through in situ polymerization in an oil-in-water emulsion and incorporated in epoxy matrices at different contents of 2.5 and 10 wt. %. The tribological properties of the epoxy composites were systematically investigated with respect to microcapsule size and content and working parameters such as normal load and sliding speed.

2 Experimental Details

2.1 Materials. Epoxy resin (Epolam 5015) and related hardener (5015) were supplied by Axson. The wax lubricant, Episol B2531, for encapsulation was purchased from Epichem International Pte. Ltd. Urea, 37 wt. % formaldehyde aqueous solution, resorcinol, ammonium chloride (NH_4Cl), sodium hydroxide (NaOH), 1-octanol, and hexane were purchased from Sigma-Aldrich (Singapore). The surfactant, ethylene maleic anhydride copolymer (EMA), was purchased from MP Biomedicals. All the chemicals were used as received unless otherwise specified.

2.2 Synthesis of Microcapsules Containing Episol B2531. The wax lubricant, Episol B2531, was encapsulated through the in situ polymerization of poly(ureaformaldehyde)

[1]Corresponding author.
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(PUF) in an oil-in-water emulsion using a similar procedure as stated in Refs. [9–11]. 5.0 g urea, 0.5 g resorcinol, and 0.5 g NH_4Cl were dissolved under agitation in 200 ml deionized (DI) water in a 1000 ml beaker that was located in a temperature-controlled water bath placed on a programmable hotplate (hotplate digital aluminum 230). After the dissolution of the added chemicals, 50 ml of an aqueous solution containing 2.5 wt. % EMA was poured into the mixture and one or two drops of 1-octanol ($CH_3(CH_2)_7OH$) were added to eliminate surface bubbles. The pH of the mixture was adjusted to 3.5 using a 1 M NaOH aqueous solution. After that, a slow stream of wax (Episol B2531) of 60 ml used for core materials was added into the mixture and dispersed for about 10 min. Then, 12.67 g of an aqueous solution containing 37 wt. % formaldehyde (CH_2O, PUF) used for shell-forming materials was slowly added into the mixture. The beaker was tightly covered with aluminum foil and the temperature of the water bath was increased to 55 °C at a heating rate of 35 °C/h using a hotplate. The system was reacted for about 4 h before the separation, rinsing, and final drying of wax-containing microcapsules. The collection was sieved to narrow down the size distribution of microcapsules and the two size ranges of 38–63 μm and 63–90 μm in diameter were selected for further usage.

The core percentage of the achieved microcapsules was measured by a physical separation method [12]. A certain amount of wax-filled microcapsules was broken completely in a 20 ml vial using a Teflon rod. After it was added with about 15 ml solvent, hexane, the vial was shaken dramatically to wash the wax away from the shell. Later the mixture was separated using a Buchner funnel and was rinsed with pure hexane 3 to 4 times for the complete removal of the residual wax. The filter paper with PUF debris was dried in an oven (Binder, model V53) at 60 °C for 24 h to evaporate the hexane. The weight difference of the filter paper before and after the process was adopted as the weight of the PUF shell for the microcapsules. By this method, the wax percentage inside the microcapsules is about 80 wt. %.

2.3 Fabrication of Self-Lubricating Epoxy Composites. First, Epolam 5015 and hardener 5015 were mixed at the recommended ratio of 100:30 for about 5 min. This mixture was evacuated for about 15 min to completely remove air bubbles. Then, 2.5 and 10 wt. % wax-containing microcapsules were well dispersed into the mixtures for about 5 min. The mixtures were evacuated again for 15 min to remove trapped air bubbles before they were molded in Teflon molds. The specimens were cured at room temperature (RT ~ 22–24 °C) for 24 h and then at an elevated temperature of 60 °C for another 3 h in an oven (Binder, model V53).

2.4 Characterization. The root-mean-squared surface roughnesses (R_q) of the samples were measured using surface profilometry (Talyscan 150) with a diamond stylus of 4 μm in diameter in a scan size of 1 mm × 1 mm. Three measurements on each sample were carried out to get an average R_q value.

The surface morphology of the samples was studied using scanning electron microscopy (SEM, JEOL-JSM-5600LV). Prior to SEM observation, the samples were coated with a gold layer to avoid charging.

The hardnesses and Young's moduli of the samples were measured using a micro-indenter (CSM MHT) with a pyramidal shaped diamond tip of 20 μm in diameter. The indentation test was performed in a load control mode with a total load of 3 N. In each indentation test, the loading and unloading rates and dwelling time at the peak load were 6 N/min, 6 N/min, and 5 s, respectively. The hardness and Young's modulus of the samples were derived using Oliver and Pharr's method and average values were taken from 12 indentation measurements carried out at different locations on each sample [13].

The tribological properties of the samples were investigated using a ball-on-disk microtribometer (CSM) operated in a rotary mode at RT. Two to three tests were conducted on each sample to get average tribological results. In a test, a steel ball (Cr6) of 6 mm in diameter was rotated on a sample in a circular path of 4 mm in diameter for about 100,000 laps at different sliding speeds under different normal loads. The wear tracks on the samples were then measured using the surface profilometry.

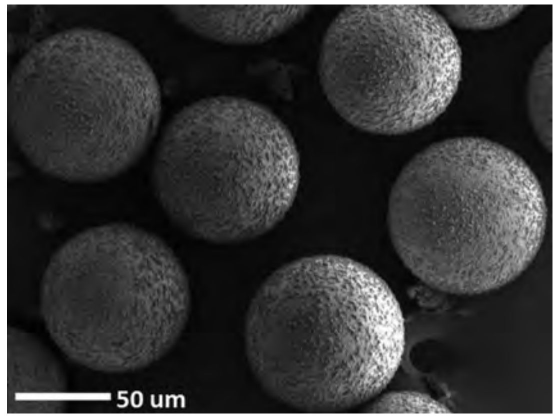

Fig. 1 SEM image showing wax-containing microcapsules with 63–90 μm in diameter

3 Results and Discussion

3.1 Morphology of Wax-Containing Microcapsules. Figure 1 shows the SEM micrograph of the wax-containing microcapsules with 63–90 μm in diameter, from which it is found that they possess rough outer shells. The microcapsules with different size ranges of 38–63 and 63–90 μm in diameter are incorporated in the epoxy matrices at different contents to investigate the effects of microcapsule size and content on the tribological properties of the epoxy composites.

3.2 Surface Morphology of Microcapsule Incorporated Epoxy Composites. Figure 2 presents the R_q values of the mechanically polished epoxy and epoxy composites using 1200 grit papers. The R_q value of the epoxy is about 0.32 μm. As the R_q values of the epoxy composites with 38–63 μm microcapsules are significantly larger than that of the epoxy because the rupture of microcapsules during the mechanical polishing forms single holes on the surfaces, their R_q value apparently increases from about 0.77 to 1.81 μm with increased microcapsule content from 2.5 to 10 wt. % due to the increased number of ruptured microcapsules. It is consistently found that the R_q value of the epoxy composites with 63–90 μm microcapsules increases from about 2.2 to 3.4 μm with increased microcapsule content from 2.5 to 10 wt. %. In addition, the R_q values of the epoxy composites with the larger 63–90 μm microcapsules are larger than those of the composites with the smaller 38–63 μm microcapsules and the epoxy because the rupture of the larger microcapsules during the mechanical polishing results in the larger single holes on the surfaces.

Figure 3(*a*) shows the surface morphology of the epoxy on which abrasive lines are apparently found. As the mechanical polishing apparently leaves the ruptured microcapsules as single holes on the surfaces of the epoxy composites (Figs. 3(*b*)–3(*e*)), the incorporation of the larger 63–90 μm microcapsules results in the larger single holes on the surfaces of the composites (Figs. 3(*d*) and 3(*e*)). The number of the ruptured microcapsules increases with increased microcapsule content for both microcapsule sizes. In addition, the epoxy composites with 63–90 μm microcapsules (Figs. 3(*d*) and 3(*e*)) possess the surfaces with the larger number of microcapsules than the ones with 38–63 μm microcapsules (Figs. 3(*b*) and 3(*c*)) because the much lower density of the wax lubricant than that of the epoxy induces the more

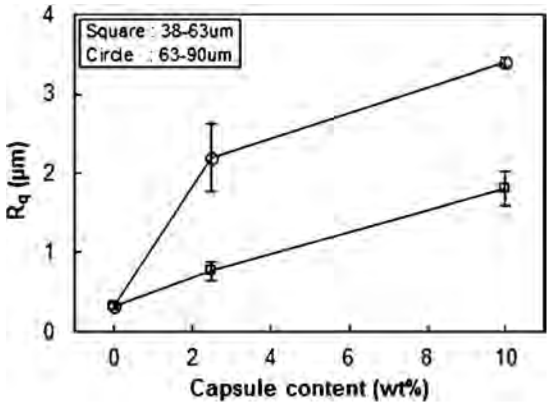

Fig. 2 Root-mean-squared-surface roughnesses (R_q) of epoxy and epoxy composites with different microcapsule size ranges and contents

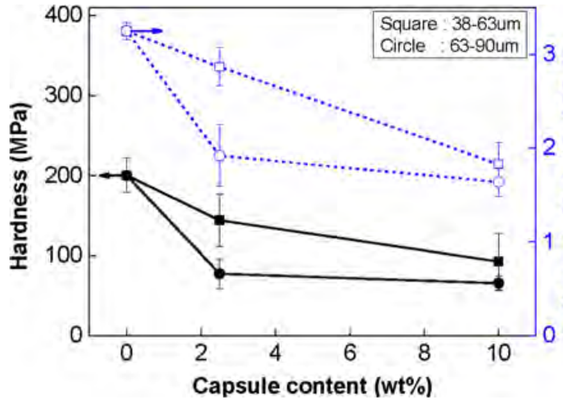

Fig. 4 Hardnesses and Young's moduli of epoxy and epoxy composites with different microcapsule size ranges and contents

diffusion of the larger microcapsules with the higher wax content to the surface during the curing at RT. It is therefore clear that the epoxy composites with the larger microcapsules have the larger number of the microcapsules on the surfaces.

3.3 Hardness and Young's Modulus of Microcapsule Incorporated Epoxy Composites. The hardnesses and Young's moduli of the epoxy and epoxy composites are presented in Fig. 4. The hardness and Young's modulus of the epoxy are about 200.5 MPa and 3.25 GPa, respectively. As the incorporation of 2.5 wt. % 38–63 μm microcapsules significantly decreases the hardness and Young's modulus of the epoxy composite to about 144.14 MPa and 2.87 GPa, respectively, the further increased 38–63 μm microcapsule content to 10 wt. % further decreases the hardness and Young's modulus to about 92.73 MPa and 1.83 GPa, respectively, as a direct result of the much lower hardness and elastic modulus of the microcapsules than those of the epoxy matrix [14,15]. Although the increased 63–90 μm microcapsule content from 2.5 to 10 wt. % consistently decreases the hardness and Young's modulus of the epoxy composites from about 77.4 MPa and 1.92 GPa to about 65.63 MPa to 1.64 GPa, respectively, the epoxy composites with the larger 63–90 μm microcapsules have the lower hardnesses and Young's moduli than the ones with the smaller 38–63 μm microcapsules due to the larger number of the larger 63–90 μm microcapsules on the surfaces. It can be seen that the size and content of the microcapsules have significant influences on the hardness and elastic modulus of the epoxy composites.

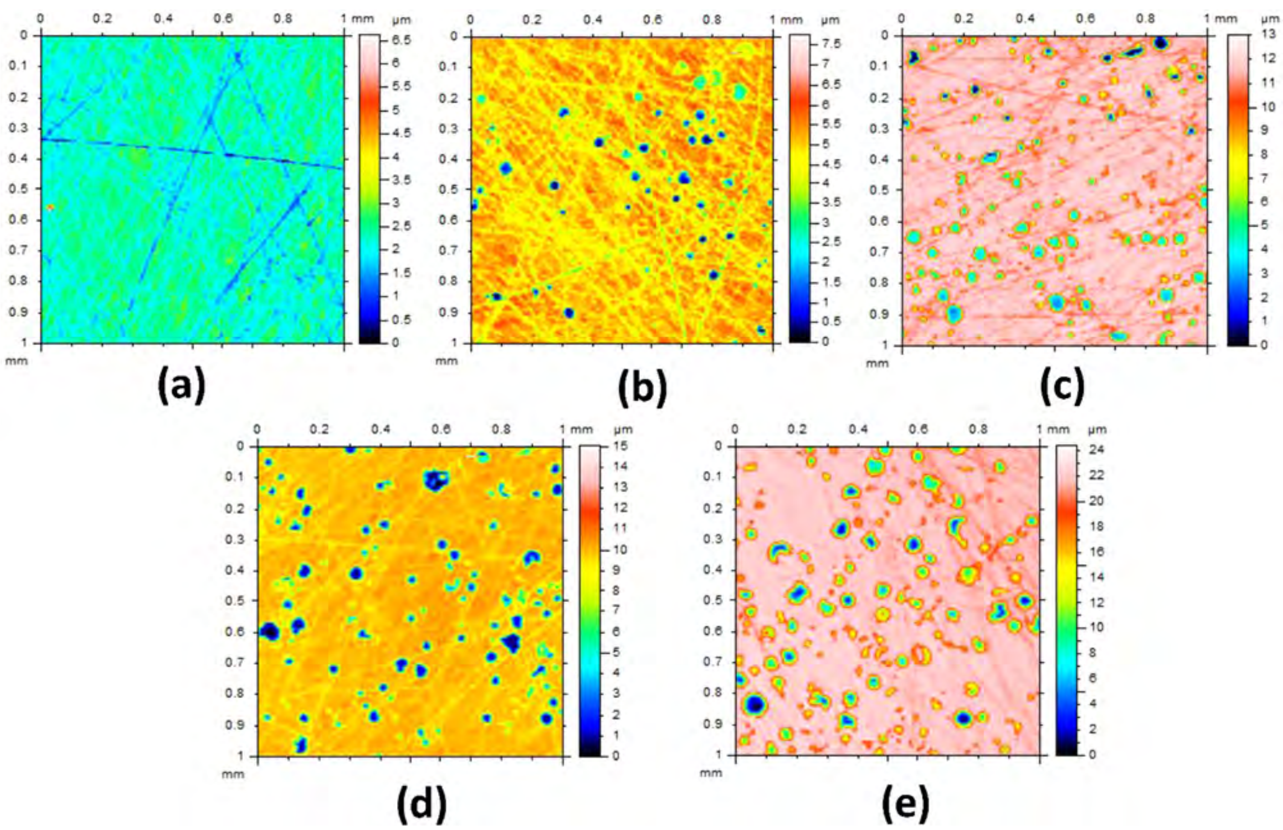

Fig. 3 Surface morphologies of (*a*) epoxy and epoxy composites with 38–63 μm microcapsule contents of (*b*) 2.5 and (*c*) 10 wt. % and 63–90 μm microcapsule contents of (*d*) 2.5 and (*e*) 10 wt. %

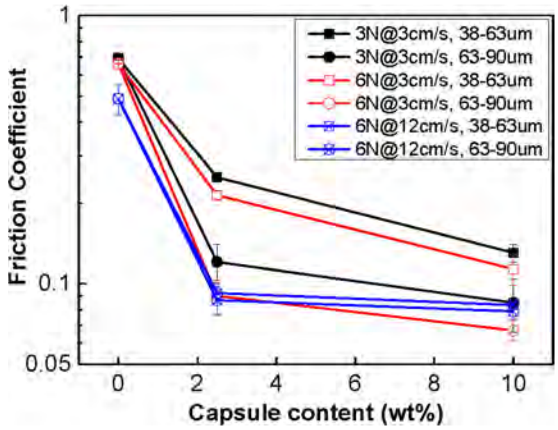

Fig. 5 Friction coefficients of epoxy and epoxy composites with different microcapsule size ranges and contents, slid against a Cr6 steel ball of 6 mm in diameter in a circular path of 4 mm in diameter for about 100,000 laps at different sliding speeds under different normal loads

3.4 Tribological Properties of Microcapsule Incorporated Epoxy Composites. The tribological properties of the epoxy and epoxy composites were investigated by sliding against a Cr6 steel ball for about 100,000 laps at different sliding speeds under different normal loads and their frictional results are presented in Fig. 5. The mean friction coefficient of the epoxy slid at a sliding speed of 3 cm/s under a normal load of 3 N is about 0.692. Increasing the normal load to 6 N at the same sliding speed slightly decreases the mean friction coefficient of the epoxy to about 0.662. The prolonged sliding of the steel ball under a high normal load generates high frictional heat which in turn causes an easy transfer of surface materials onto the steel ball surface to form a transfer layer so that the transfer layer reduces the friction by preventing direct contact between the steel ball and epoxy [16]. It is therefore supposed that the increased normal load decreases the friction of the epoxy by increasing the frictional heat and promoting the transfer of surface materials. In addition, the higher wear of the epoxy can give rise to its lower friction because the larger quantity of wear debris and the higher surface roughening of the rubbing surfaces lessen the contact between the steel ball and epoxy and lowers the effective interfacial shear strength between them [17,18]. As a result, the increased normal load leads to the decreased friction of the epoxy by promoting the wear of the epoxy. Under the same normal load of 6 N, the sliding of the steel ball on the epoxy at the higher sliding speed of 12 cm/s gives rise to the lower mean friction coefficient of about 0.489 probably due to the higher wear of the epoxy [17–19].

The mean friction coefficient of the epoxy composite with 2.5 wt. % 38–63 μm microcapsules slid at a sliding speed of 3 cm/s under a normal load of 3 N is about 0.249, that is significantly lower than that of the epoxy tested under the same conditions. It indicates that the incorporation of 2.5 wt. % wax-containing microcapsules effectively decreases the friction of the epoxy composite because the rupture of microcapsules via the surface wear releases wax lubricant to lubricate the rubbing surfaces [1,9,10]. In addition, the released wax lubricant lessens a direct solid–solid contact between the steel ball and composite [1,9,10,20–22]. Therefore, the increased 38–63 μm microcapsule content to 10 wt. % apparently further decreases the mean friction coefficient of the epoxy composite to about 0.13. It is consistently found that the increased 63–90 μm microcapsule content from 2.5 to 10 wt. % also decreases the mean friction coefficient of the epoxy composites from about 0.12 to 0.085 due to the promoted lubricating effect of the released wax lubricant. Moreover, the incorporation of the larger 63–90 μm microcapsules results in more reduction in the friction of the epoxy composites compared to that of the smaller 38–63 μm microcapsules because the larger number of the larger 63–90 μm microcapsules on the surface releases the larger amount of wax lubricant during the sliding (Fig. 3).

With increased microcapsule content from 2.5 to 10 wt. %, the epoxy composites slid against the steel ball at a sliding speed of 3 cm/s under a normal load of 6 N have significant reductions in the mean friction coefficients from about 0.213 to 0.113 for the 38–63 μm microcapsules and from about 0.09 to 0.067 for the 63–90 μm microcapsules. The results consistently show that the incorporation of the wax-containing microcapsules significantly decreases the friction of the epoxy composites slid under the normal load of 6 N as the increased microcapsule content further decreases the friction of the composites. In addition, the incorporation of the larger 63–90 μm microcapsules gives rise to the lower friction of the epoxy composites than that of the smaller 38–63 μm microcapsules. However, the mean friction coefficients of the epoxy composites slid under the higher normal load of 6 N are slightly lower than those of the ones tested under the lower normal load of 3 N because the higher wear of the epoxy composites associated with the higher normal load releases more wax lubricant for the effective self-lubricating of the composites during the sliding. The promoted wear of the epoxy composites causes their surface roughening so that the uneven surfaces of the composites keep and sustainably release the wax lubricant to lubricate the rubbing surfaces during the sliding [23–28]. At the same time, the promoted surface roughening and produced wear debris help to reduce the friction of the epoxy composites by lessening the contact between the rubbing surfaces.

In Fig. 5 the mean friction coefficients of the epoxy composites slid at a sliding speed of 12 cm/s under a normal load of 6 N are about 0.092 and 0.083 for the 38–63 μm microcapsule contents of 2.5 and 10 wt. %, respectively, and about 0.087 and 0.078 for the 63–90 μm microcapsule contents of 2.5 and 10 wt. %, respectively. It is clear that the increased microcapsule content consistently decreases the friction of the epoxy composites even at the relatively high sliding speed. It can be deduced that the size and content of the wax-containing microcapsules apparently influence the friction of the epoxy composites.

Figure 6(*a*) shows the friction coefficients of the epoxy and epoxy composites slid against the steel ball at a sliding speed of 3 cm/s under a normal load of 3 N, as a function of the number of laps. The friction coefficient of the epoxy reaches about 0.68 after 5000 laps and becomes stable at about 0.71 for the rest. The cyclic increase and drop in the friction coefficient are apparently found on the trend of friction coefficient versus laps of the epoxy as shown in Fig. 6(*a*), which is probably attributed to the repeated formation and detachment of tribolayers on the wear track during the sliding [7,26]. During the sliding, the surface wear of the epoxy produces wear debris and the repeated sliding of the steel ball compacts the wear debris to form a tribolayer on the wear track. Since the compacted tribolayer is somewhat harder, the formation of the tribolayer significantly decreases the friction of the epoxy by effectively lessening the contact area between the rubbing surfaces. At the same time, the repeated sliding of the steel ball wears out the formed tribolayer and eventually detaches it. It is therefore supposed that the formation and detachment of the tribolayers on the wear track of the epoxy during the sliding contribute to the wear of the epoxy. In Fig. 6(*a*) the incorporation of wax-containing microcapsules dramatically decreases the friction of the epoxy composites during the entire sliding via the effective lubricating effect of released wax lubricant as the increased microcapsule content further depresses the trends of friction coefficient versus laps of the composites. However, the epoxy composites with 38–63 μm microcapsules exhibit the slightly higher trends of friction coefficient versus laps for the relevant microcapsule contents than the ones with 63–90 μm microcapsules. Among the epoxy composites, the composite with 2.5 wt. % 38–63 μm microcapsules shows the highest trend of friction coefficient versus laps, while the incorporation of 10 wt. % 63–90 μm microcapsules gives rise to the lowest friction coefficient of the composite for the entire sliding.

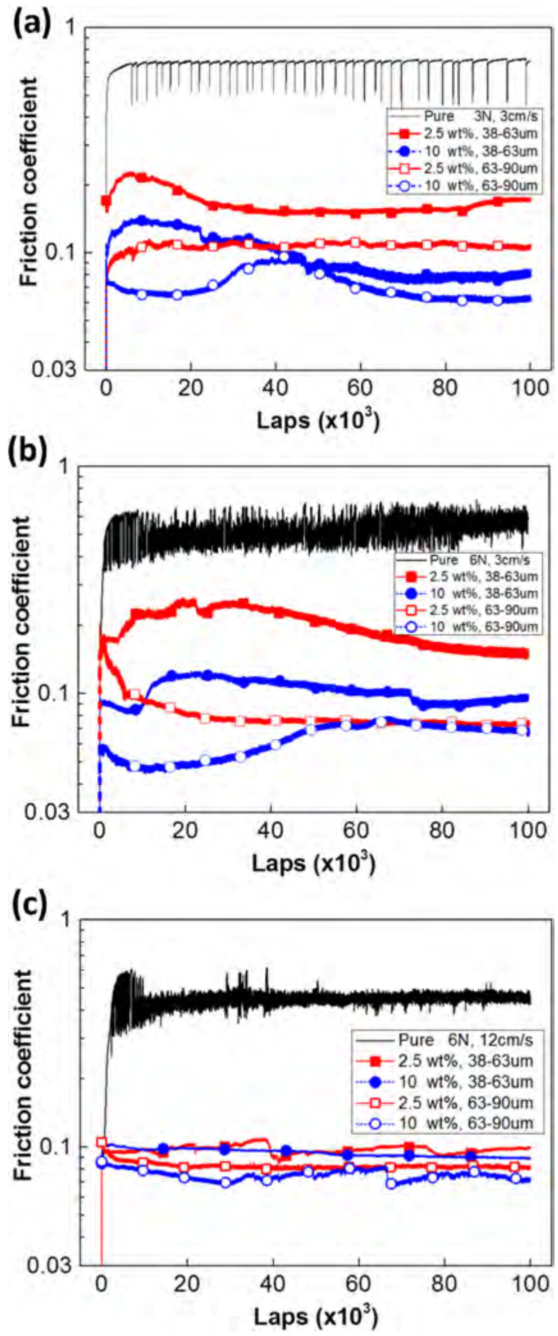

Fig. 6 Friction coefficients of epoxy and epoxy composites with different microcapsule size ranges and contents, slid against a Cr6 steel ball of 6 mm in diameter in a circular path of 4 mm in diameter for about 100,000 laps (*a*) at a sliding speed of 3 cm/s under a normal load of 3 N, (*b*) at a sliding speed of 3 cm/s under a normal load of 6 N, and (*c*) at a sliding speed of 12 cm/s under a normal load of 6 N, as a function of the number of laps

Figure 6(*b*) presents the friction coefficients of the epoxy and epoxy composites, slid at a sliding speed of 3 cm/s under a normal load of 6 N, as a function of the number of laps. Comparison of Figs. 6(*a*) and 6(*b*) clearly shows that the higher wear of the epoxy tested under the higher normal load of 6 N dramatically shortens the periods of the repeated formation and detachment of tribolayers on the wear track so that the cyclic increase and drop in the friction coefficient of the epoxy slid under the higher normal load of 6 N (Fig. 6(*b*)) are not as apparent as those of the one tested under the lower normal load of 3 N (Fig. 6(*a*)). In addition, the

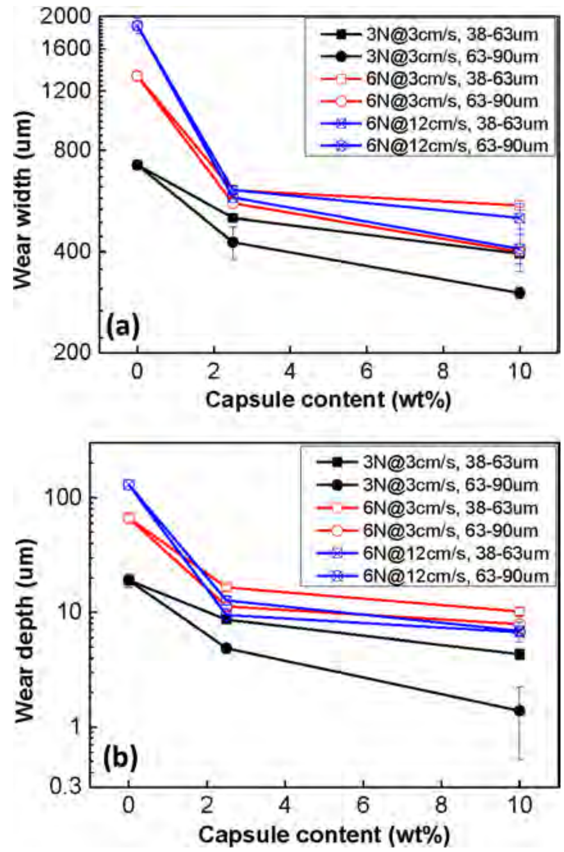

Fig. 7 (*a*) Wear widths and (*b*) depths of epoxy and epoxy composites with different microcapsule size ranges and contents slid under the same conditions as described in Fig. 5

friction coefficient of the epoxy increases with increased laps (Fig. 6(*b*)), which is indicative of the promoted wear of the epoxy with prolonged sliding. Although the incorporation of 2.5 wt. % 38–63 μm microcapsules significantly decreases the friction of the epoxy composite throughout the wear test, the epoxy composite with 2.5 wt. % 38–63 μm microcapsules exhibits the highest trend of friction coefficient versus laps among the epoxy composites slid under 6 N (Fig. 6(*b*)). The incorporation of the larger 63–90 μm microcapsules results in the lower trends of friction coefficient versus laps of the epoxy composites slid under the normal load of 6 N than that of the smaller 38–63 μm microcapsules. It is clear that the incorporation of the larger 63–90 μm microcapsules can give the better reduction in the friction of the epoxy composites slid under the relatively high normal load.

The friction coefficients of the epoxy and epoxy composites, slid at a sliding speed of 12 cm/s under a normal load of 6 N, are presented in Fig. 6(*c*) as a function of the number of laps. The epoxy slid at the higher sliding speed of 12 cm/s under the normal load of 6 N (Fig. 6(*c*)) does not apparently exhibit the cyclic periods on its trend of friction coefficient versus laps because the accelerated wear of the epoxy associated with the increased sliding speed almost suppresses the formation and detachment of the tribolayers on the wear track during the sliding. The epoxy composites exhibit the much lower friction coefficients during the entire sliding than the epoxy as the friction of the composites slid at 12 cm/s is relatively quite stable. The slightly lower trends of friction coefficient versus laps of the epoxy composites with the larger 63–90 μm microcapsules than those of the ones with the smaller 38–63 μm microcapsules clearly indicate that the size of the wax-containing microcapsules still has a significant influence on the friction of the composites slid at the relatively high sliding speed. The results clearly show that the incorporation of the wax-containing microcapsules is an effective way to reduce the friction of the epoxy composites during the sliding contact with the metal counterpart.

Figure 7 illustrates the wear widths and depths of the epoxy and epoxy composites. The wear width and depth of the epoxy slid at a sliding speed of 3 cm/s under a normal load of 3 N are about 724 and 19 μm, respectively. When the steel ball is slid on the epoxy at the same sliding speed of 3 cm/s under the higher normal load of 6 N, the wear width and depth of the epoxy increase to about 1332.5 and 66.5 μm, respectively. The wear width and depth of the epoxy slid at a sliding speed of 12 cm/s under a normal load of 6 N are about 1877.5 and 130 μm, respectively. It is clear that the wear of the epoxy becomes higher with higher normal load or sliding speed. The opposite trends between the decreased friction (Fig. 5) and increased wear (Fig. 7) of the epoxy confirm that the increased wear of the epoxy results in its decreased friction by promoting the surface roughening and the production of wear debris [17,18].

In Fig. 7 the wear width and depth of the epoxy composite with 2.5 wt. % 38–63 μm microcapsules slid at a sliding speed of 3 cm/s under a normal load of 3 N are about 503.8 and 8.6 μm, respectively, which are apparently smaller than those of the epoxy tested under the same conditions due to the lubricating effect of released wax lubricant. The increased 38–63 μm microcapsule content to 10 wt. % further decreases the wear width and depth of the epoxy composite to about 394.8 and 4.3 μm, respectively; as a result of the promoted lubricating effect of released wax lubricant. In addition, the wear width and depth of the epoxy composites with 63–90 μm microcapsules consistently decrease from about 427 and 4.9 μm to about 301.5 and 1.4 μm, respectively, with increased microcapsule content from 2.5 to 10 wt. %. The wear of the epoxy composites with the larger 63–90 μm microcapsules is slightly lower than that of the ones with the smaller 38–63 μm microcapsules (Fig. 7) because the larger number of the larger 63–90 μm microcapsules on the surface releases the larger amount of wax lubricant during the sliding.

The wear widths and depths of the epoxy composites slid at a sliding speed of 3 cm/s under a normal load of 6 N are about 608.3 and 16.5 μm for the 38–63 μm microcapsule content of 2.5 wt. %, about 548.5 and 10.2 μm for the 38–63 μm microcapsule content of 10 wt. %, about 557 and 11.3 μm for the 63–90 μm microcapsule content of 2.5 wt. %, and about 400.5 and 7.9 μm for the 63–90 μm microcapsule content of 10 wt. %, respectively, as found in Fig. 7. It can be seen that the increased microcapsule content decreases the wear of the epoxy composites slid under the higher normal load as the incorporation of the larger 63–90 μm microcapsules gives rise to the lower wear of the composites. However, the wear widths and depths of all the epoxy composites slid under the higher normal load of 6 N are slightly larger than those of the ones tested under the lower normal load of 3 N due to the promoted wear of the epoxy composites.

The wear widths and depths of the epoxy composites slid at a sliding speed of 12 cm/s under a normal load of 6 N are about 609.5 and 12.7 μm for the 38–63 μm microcapsule content of 2.5 wt. %, about 503.5 and 6.9 μm for the 38–63 μm microcapsule content of 10 wt. %, about 579.25 and 9.5 μm for the 63–90 μm microcapsule content of 2.5 wt. %, and about 408 and 6.7 μm for the 63–90 μm microcapsule content of 10 wt. %, respectively. It is clearly found that the higher microcapsule content or the incorporation of the larger 63–90 μm microcapsules can give rise to the lower wear of the epoxy composites slid at the relatively high sliding speed.

After the tribological tests, the wear morphologies of the epoxy and epoxy composites were observed using SEM. In Fig. 8(*a*) the worn surface of the epoxy slid at a sliding speed of 3 cm/s under a normal load of 3 N is relatively smoother than its untested surface, which is indicative of the formation of tribolayers on the wear track during the sliding. The removal of surface materials as platelets is found in the center of the wear track where the contact pressure is highest [27,28]. During the wear test, the repeated sliding of the steel ball under the high normal load induces cyclic stress

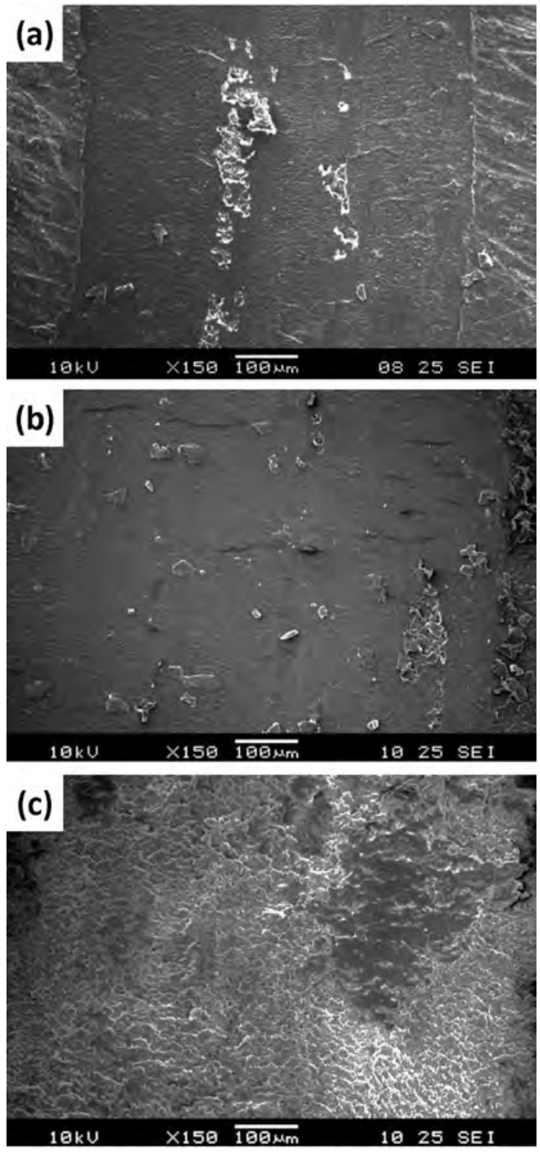

Fig. 8 SEM micrographs showing worn surfaces of epoxy slid against a Cr6 steel ball of 6 mm in diameter in a circular path of 4 mm in diameter for about 100,000 laps (*a*) at a sliding speed of 3 cm/s under a normal load of 3 N, (*b*) at a sliding speed of 3 cm/s under a normal load of 6 N, and (*c*) at a sliding speed of 12 cm/s under a normal load of 6 N

concentration in front of the steel ball, which in turn causes surface fatigue [27,28]. The surface fatigue initiates minute cracks in the subsurface and propagates the cracks parallel to a free surface for some extent and eventually results in the removal of materials as platelets that can be clearly seen in the center of the wear track (Fig. 8(*a*)). At the same time, the surface fatigue also initiates minute cracks perpendicular to the sliding direction and propagates the cracks into the subsurface [27,28]. Eventually, the formation of a network of microcracks forms microwave features on the wear track as found in Fig. 8(*a*).

Figure 8(*b*) shows the worn surface of the epoxy slid at the same sliding speed under the higher normal load of 6 N. The removal of surface materials as platelets is not apparently found in the center of the wear track because the higher surface wear of the epoxy tested under the higher normal load almost suppresses the initiation and propagation of minute cracks in the subsurface. However, the microwave features on the wear track indicate that the repeated sliding of the steel ball under the higher normal load still causes the surface fatigue.

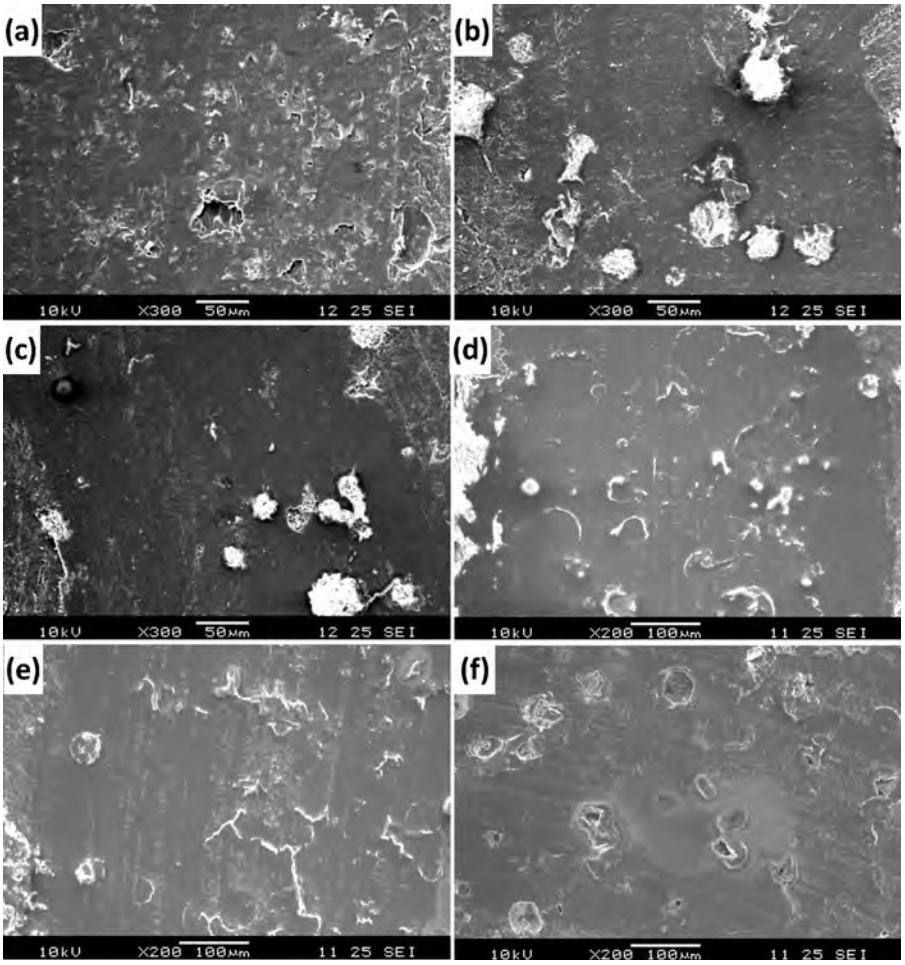

Fig. 9 SEM micrographs showing worn surfaces of epoxy composites with 38–63 μm microcapsule contents of (*a*), (*b*), and (*c*) 2.5 and (*d*), (*e*), and (*f*) 10 wt. % slid against a Cr6 steel ball of 6 mm in diameter in a circular path of 4 mm in diameter for about 100,000 laps (*a*) and (*d*) at a sliding speed of 3 cm/s under a normal load of 3 N, (*b*) and (*e*) at a sliding speed of 3 cm/s under a normal load of 6 N, and (*c*) and (*f*) at a sliding speed of 12 cm/s under a normal load of 6 N

The sliding of the steel ball at a sliding speed of 12 cm/s under a normal load of 6 N generates the highest wear on the epoxy surface so that the tribolayers are locally found on the wear track of the epoxy as shown in Fig. 8(*c*) because the severe surface wear of the epoxy hinders the formation of the tribolayers during the sliding. Moreover, the largest microwave features are found on the wear track of the epoxy as shown by the comparison of Figs. 8(*a*), 8(*b*), and 8(*c*) because the increased sliding speed accelerates the surface fatigue by increasing the frequency of cyclic loading during the sliding. Therefore, the most severe surface fatigue of the epoxy is responsible for its roughest wear morphology. The SEM observation clearly indicates that the prolonged sliding of the steel ball causes the surface fatigue of the epoxy as the increased sliding speed significantly promotes the surface fatigue of the epoxy.

Figures 9(*a*)–9(*c*) show the worn surfaces of the epoxy composite with 2.5 wt. % 38–63 μm microcapsules slid at different sliding speeds under different normal loads. In Fig. 9(*a*) the worn surface of the epoxy composite is relatively rough, resulting from the rupture of microcapsules and the localized removal of surface materials caused by surface fatigue. The incorporation of 2.5 wt. % 38–63 μm microcapsules significantly lowers the friction and wear of the epoxy composite slid at a sliding speed of 3 cm/s under a normal load of 3 N than those of the epoxy (Figs. 5 and 7), but does not result in the suppression of surface fatigue induced by the prolonged sliding of the steel ball (Fig. 9(*a*)). When the normal load is increased to 6 N, the promoted wear of the epoxy composite enhances the release of wax lubricant and eliminates the initiation and formation of microcracks during the prolonged sliding and consequently gives rise to the smoother wear morphology as found in Fig. 9(*b*). The worn surface of the epoxy composite slid at the higher sliding speed of 12 cm/s under the normal load of 6 N (Fig. 9(*c*)) is not apparently different from that of the one slid at the lower sliding speed of 3 cm/s under the same normal load (Fig. 9(*b*)) probably due to no significant differences in their wear with respect to sliding speed (Fig. 7).

Comparison of Figs. 9(*a*) and 9(*d*) shows that the sliding of the steel ball on the epoxy composite with 10 wt. % 38–63 μm microcapsules does not result in the significant surface fatigue of the composite because the increased release of wax lubricant during the wear test apparently suppresses the surface fatigue by lubricating the rubbing surfaces and lessening the direct contact between them [1,9,10,20–22]. As found in Fig. 9(*e*), increasing the normal load to 6 N forms cracks on the wear track of the epoxy composite, especially in the center of the wear track. The repeated sliding of the steel ball under the higher normal load initiates cracks around the corners of the ruptured microcapsules where the stress concentration is relatively high and the propagation of the cracks forms a network between the ruptured microcapsules on the wear track (Fig. 9(*e*)). However, the sliding of the steel ball on the

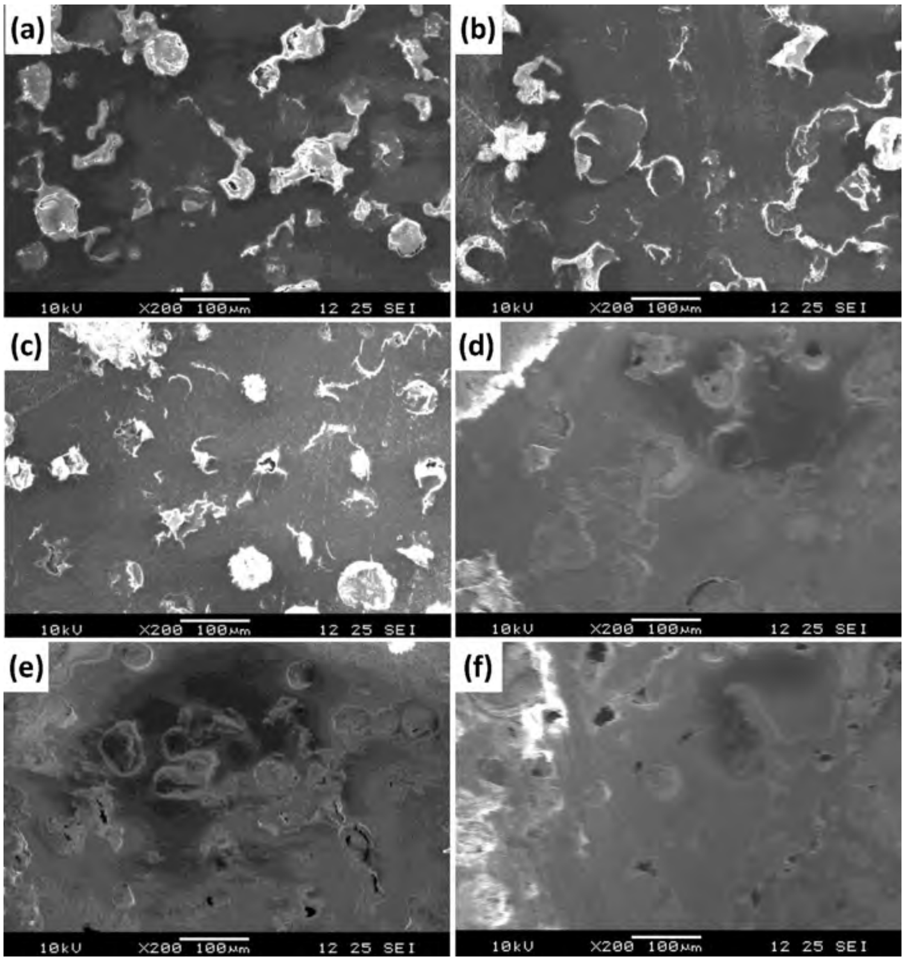

Fig. 10 SEM micrographs showing worn surfaces of epoxy composites with 63–90 μm microcapsule contents of (*a*), (*b*), and (*c*) 2.5 and (*d*), (*e*), and (*f*) 10 wt. % slid against a Cr6 steel ball of 6 mm in diameter in a circular path of 4 mm in diameter for about 100,000 laps (*a*) and (*d*) at a sliding speed of 3 cm/s under a normal load of 3 N, (*b*) and (*e*) at a sliding speed of 3 cm/s under a normal load of 6 N, and (*c*) and (*f*) at a sliding speed of 12 cm/s under a normal load of 6 N

epoxy composite at the higher sliding speed of 12 cm/s does not cause any cracking on the wear track (Fig. 9(*f*)).

The epoxy composite with 2.5 wt. % 63–90 μm microcapsules can release more wax lubricant during the sliding due to the larger number of the larger microcapsules on the surface compared to the one with 2.5 wt. % 38–63 μm microcapsules. Therefore, the increased release of wax lubricant is responsible for the suppression of surface fatigue of the epoxy composite with 2.5 wt. % 63–90 μm microcapsules slid at a sliding speed of 3 cm/s under a normal load of 3 N as shown by the comparison of Figs. 9(*a*) and 10(*a*). In Figs. 10(*b*) and 10(*c*) the wear morphologies of the epoxy composite with 2.5 wt. % 63–90 μm microcapsules do not apparently change with sliding speed as a result of the presence of released wax lubricant on the surface.

The epoxy composite with 10 wt. % 63–90 μm microcapsules slid under different conditions (Figs. 10(*d*)–10(*f*)) does not exhibit a significant surface fatigue on the wear track. However, the formation of a network of microcracks is still found on the wear track of the epoxy composite with 10 wt. % 63–90 μm microcapsules slid under the normal load of 6 N (Fig. 10(*e*)), confirming that the incorporation of 10 wt. % microcapsules results in the surface cracking during the prolonged sliding under the normal load of 6 N. The networked cracks are not found on the wear track of the composite slid at a sliding speed of 12 cm/s under a normal load of 6 N (Fig. 10(*f*)), which is in agreement with the observation in Fig. 9(*f*).

Comparison of Figs. 8, 9, and 10 clearly shows that the wear of the epoxy composites modified with wax-containing microcapsules is much lower than that of the epoxy due to the effective lubricating effect of released wax lubricant. The SEM observation confirms that the incorporation of wax-containing microcapsules greatly reduces the wear of the epoxy composites as the wear of the composites is apparently influenced by the size and content of the microcapsules.

4 Conclusions

In this study the tribological properties of the epoxy composites with different wax-containing microcapsule size ranges and contents were systematically investigated. The epoxy composites with the larger 63–90 microcapsules had the larger number of microcapsules on the surfaces than the ones with the smaller 38–63 microcapsules at the same microcapsule contents probably due to the more increase diffusion of the larger 63–90 microcapsules to the surfaces. The friction and wear of the epoxy composites slid against the steel ball were much lower than those of the epoxy because the rupture of microcapsules via the surface wear released wax lubricant to lubricate the rubbing surfaces. In addition, the friction and wear of the epoxy composites were lower for the higher microcapsule content or the incorporation of the larger 63–90 microcapsules. The friction of the epoxy composites slightly decreased with increased normal load due to the promoted wear of the composites and the increased release of the wax lubricant. It could be concluded that the incorporation of wax-containing microcapsules was an effective way to reduce the friction and wear of the epoxy composites.

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