

## The Tribological Properties of Benzoxazine-Bismaleimides Composites with Functionalized Nano-SiO<sub>2</sub>

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**ABSTRACT:** To improve the tribological properties of benzoxazine (BOZ) resin, bismaleimides (BMI) resin is chosen as organic phase, hyperbranched polysilane functionalized SiO<sub>2</sub> nanoparticles (HBPSi-SiO<sub>2</sub>) are chosen as inorganic modifiers to prepare HBPSi-SiO<sub>2</sub>/BOZ-BMI composites using high shear and ultrasonic processes. The effect of content of HBPSi-SiO<sub>2</sub> on the mechanical properties and tribological properties of the composites are investigated. The results show that suitable addition of HBPSi-SiO<sub>2</sub> can largely enhance the impact strength, reduce the friction coefficient, and wear rate of BOZ-BMI resin. Scanning electron microscopy is employed to research the wearing mechanism of materials. The severe wear of the BOZ pure resin is owing to fatigue wear, and the moderate wear of BOZ-BMI resin is attributed to adhesive wear. While, the mild wear of the composites with HBPSi-SiO<sub>2</sub> is due to abrasive wear. © 2013 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 129: 3150–3155, 2013

**KEYWORDS:** composites; friction; wear and lubrication; resins

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### INTRODUCTION

Benzoxazine (BOZ) resin as a kind of new high-performance phenolic resin, which has been widely used in electronic and aerospace industries,<sup>1</sup> is claimed to possess better flame and chemical resistances, electrical insulation, thermal stability, and lower cost in comparison with conventional phenolic resins.<sup>2–6</sup> In addition, BOZ exhibits high dimensional stability, low volume contraction during the curing, and its ring-opening polymerization during the curing process do not produce by-products, thus its composite is more dense and homogeneous.<sup>7–9</sup> Despite these excellent properties, its brittleness and higher crosslink densities largely limited its applications.<sup>10</sup> To overcome these shortcomings, varieties of modifying methods should be researched. Two main routes have been developed to prepare new BOZ matrices, that is, synthesizing new matrices or modifying present matrices.<sup>11</sup> Obviously, the latter process is more frugal and advantageous. For the “modifying” process, thermoplastic resins such as polyurethane and polyetherimide resins are chosen to improve the integrated performance,<sup>12,13</sup> thermosetting resins such as phenolic and epoxy resins are employed to enhance the mechanical properties.<sup>14,15</sup> Nevertheless, there is no reference has been reported on the tribological properties of BOZ resin. Our research discovers that BOZ resin exhibits low anti-wear property, which largely limits its application.

Bismaleimide (BMI) is one kind of representative heat resistant thermosetting resin, which has wide applications in high performance adhesives and coatings, as well as the matrices of advanced composites.<sup>16,17</sup> BMI has the excellent processability, balance of thermal, electrical, and mechanical properties.<sup>18,19</sup> Especially, the thermal stability of BMI is increased by cocuring with BOZ, which attribute to cross hydrogen bonding and network interpenetrating.<sup>15,20</sup> Moreover, BMI contained allyl ethers has the higher rheological property and better processing characteristics.<sup>21</sup> Approximately, its cured resin even has almost same mechanical and tribological properties as that of the common BMI resin modified with *o,o'*-diallylbisphenol-A. With these advantages represented above, BMI contained allyl ethers can be chosen as the organic phase for modifying BOZ.

Nano-SiO<sub>2</sub> is the good inorganic phase to enhance the toughness and tribology properties of composites.<sup>22</sup> The addition of nano-SiO<sub>2</sub> exhibits the unique properties, such as antiwear, reducing friction, and high load capacity.<sup>23</sup> However, nano-SiO<sub>2</sub> additives in the resins have a strong tendency to agglomerate due to its high surface energy and poor consistency between the material surface and resins.<sup>24</sup> To address this, surface modifying of nano-SiO<sub>2</sub> is necessary.

Hyperbranched polysiloxane has the advantages of both the hyperbranched polymers and polysiloxane. It has highly

branched, nonentangled architecture, active terminal groups, and even has lower viscosity than polysiloxane.<sup>25,26</sup> In addition, its thermal and moisture resistance are also much better in comparison with conventional hyperbranched polymers.<sup>27,28</sup> Thus, hyperbranched polysiloxane grafted on the surface of nano-SiO<sub>2</sub> can greatly improve the dispersibility of the nano-SiO<sub>2</sub> in resins.<sup>29</sup>

In this article, the hyperbranched polysilane grafted nano-SiO<sub>2</sub> nanoparticles (HBPSi-SiO<sub>2</sub>) are chosen as the inorganic fillers, BMI contained allyl ethers are chosen as the organic phase to fabricate composites. The influence of the content of HBPSi-SiO<sub>2</sub> on tribological properties of the composites is investigated. This investigation is expected to propose a new approach to develop BOZ resin in the fictional application fields.

## EXPERIMENT

### Raw Materials

The BOZ resin was provided by polymer science and engineering college of Sichuan university. The BMI prepolymer contained allyl ethers was provided by Rongchang Ning research group at Northwestern Polytechnical University. Nano-SiO<sub>2</sub> particles were purchased from Hebei east nano material. The  $\gamma$ -Glycidyloxypropyltrimethoxysilane (KH-560, purchased from Jingzhou fine chemical) was chosen to functionalize the nano-SiO<sub>2</sub>. The other reagent and solvent used without further purification were supplied by Tianjin fuchen chemical reagents factory: chlorhydric acid, ethanol, and acetic acid.

### Preparation of HBPSi-SiO<sub>2</sub>

One gram Nano-SiO<sub>2</sub> and 93.5 mL KH-560 were added into a 3-neck flask, and then ultrasonic dispersed for 30 min. Right amount of hydrochloric acid solution (1 mol/L) was dropwise-added into the 3-neck flask above. The mixture was then stirred at 70°C for 6 h to functionalize SiO<sub>2</sub> by the reaction of hydrolytic condensation. The schematic of HBPSi-SiO<sub>2</sub> were shown in Figure 1.

### Preparation of Materials

The HBPSi-SiO<sub>2</sub>/BOZ-BMI composites were prepared by casting method. BOZ and 15.0 wt % BMI were mixed under 150°C in

a glass beaker and kept at this constant temperature until totally melting. Then the HBPSi-SiO<sub>2</sub> were added into the prepolymer and stirred 30–40 min to evaporate acetone. The mixture was then poured into a preheated mold with release agent, and degasses in a vacuum drying oven at 150°C for 40–50 min. Finally, the mixture was cured following the schedule of 150°C/2 h + 170°C/2 h + 180°C/4 h. A post curing process was 200°C/2 h. As control experiments, BOZ pure resin, and BOZ-BMI resin were prepared in the same manner as above.

### Property Investigation of Materials

**Impact Strength.** The impact strength was performed according to GB/T2570-1995 (Chinese Standard) on test machine (XCJ-40). In each system, more than five specimens were tested.

**Friction and Wear Tests.** The friction and wear tests were performed according to GB3960-83 (Chinese Standard) on test machine (MM-200). The accuracy of the machine could achieve  $\pm 5\%$  in measuring the frictional force and wear rate. Before each test, the counterpart steel ring and the materials were abraded with No. 900 water-abrasive paper. Then the steel ring and samples were cleaned by acetone. All the friction and wear experiments were conducted at room temperature. Friction coefficient was measured under a load of 196 N and test duration of 120 min. Wear rate was measured using electronic balance to an accuracy of  $\pm 0.0001$  g.

**Scanning Electron Microscope (SEM) Observation.** Surface morphology of fractured surface and the wear surface of the samples were characterized with CHIS-570 scanning electron microscope.

## RESULTS AND DISCUSSION

### Impact Strength of Materials

Table I summaries the mechanical, physical, and chemical properties of BOZ pure resin. It can be concluded that BOZ pure resin exhibits high thermal stability, low volume contractive rate, but possesses the low impact strength, which indicate that it is a brittle material. Thus, BMI and HBPSi-SiO<sub>2</sub> are chosen for the modifying of BOZ pure resin in our research.

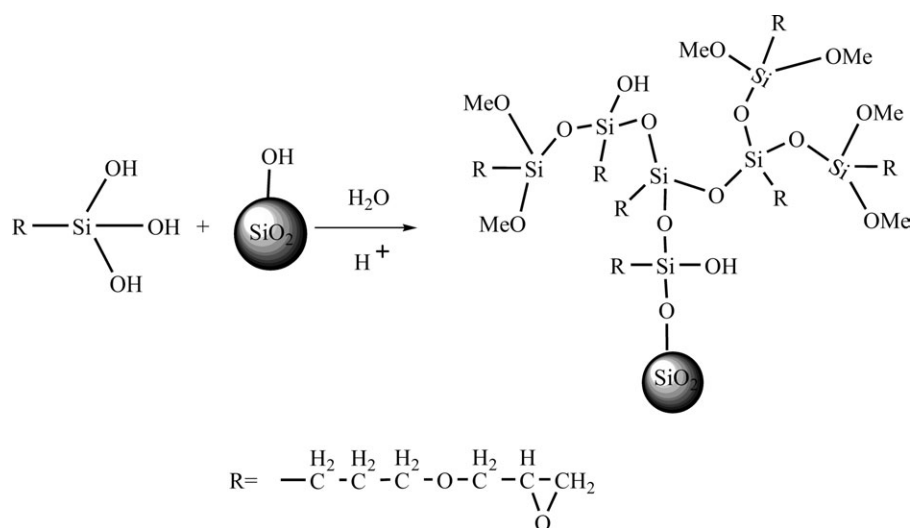


Figure 1. The schematic of hyperbranched polysilane functionalized SiO<sub>2</sub>.

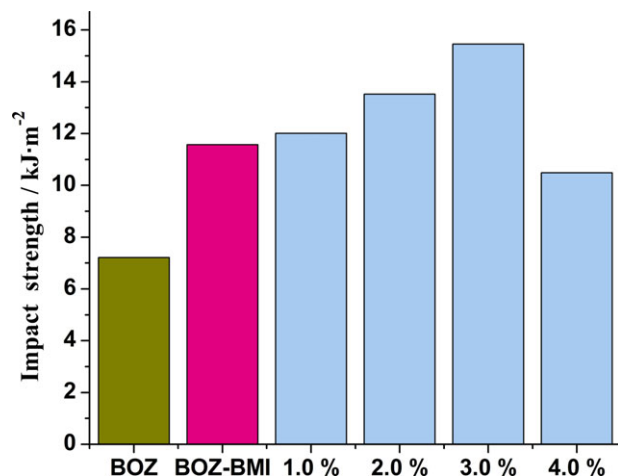
**Table I.** Mechanical, Physical, and Chemical Properties of BOZ Pure Resin

Mechanical properties		Physical and chemical properties	
Impact strength	7.21 kJ/m <sup>2</sup>	Volume contractive rate	0.4%
Bending strength	133 MPa	Gelation time	>10 min (210°C)
Breaking elongation	1.3%	Softening point	65–70°C
Tensile strength	64 MPa	T <sub>g</sub>	180°C

The impact strength of the BOZ pure resin, BOZ-BMI resin and the composites with different content of HBPSi-SiO<sub>2</sub> is shown in Figure 2. It can be seen that BOZ pure resin has the lowest impact strength, which is 7.21 kJ/m<sup>2</sup>, while the impact strength of BOZ-BMI is 11.57 kJ/m<sup>2</sup>, which increases 60.5% than that of BOZ pure resin. Furthermore, suitable amount of HBPSi-SiO<sub>2</sub> can obviously improve the impact strength of BOZ-BMI resin. The impact strength increases continuously with the addition of HBPSi-SiO<sub>2</sub>, and reaches the maximum value by 15.46 kJ/m<sup>2</sup> with 3.0 wt % HBPSi-SiO<sub>2</sub>, the impact strength of the composites increases as much as 33.6% in comparison with BOZ-BMI resin, and increases 111.4% than BOZ pure resin.

#### Fracture Surface of Materials

To research the fracture characteristics of materials, the fracture surfaces of BOZ pure resin, BOZ-BMI resin and the composite filled with 3.0 wt % HBPSi-SiO<sub>2</sub> taken from impact tests are explored (Figure 3). It is shown that BOZ-BMI resin [Figure 3(b)] has the more dimples than the BOZ resin [Figure 3(a)]. This possible reason is that the curing of BOZ and BMI has largely enhanced the toughness of the resin, so that the resin can absorb more energy in impact test, these observations are in good agreement with the conclusion above that the BOZ-BMI resin has better impact strength than BOZ pure resin. However, compared with BOZ pure resin and BOZ-BMI resin, the fracture surface of the 3.0 wt % HBPSi-SiO<sub>2</sub> composite is



**Figure 2.** The impact strength of BOZ, BOZ-BMI, and the composites with different content HBPSi-SiO<sub>2</sub>. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

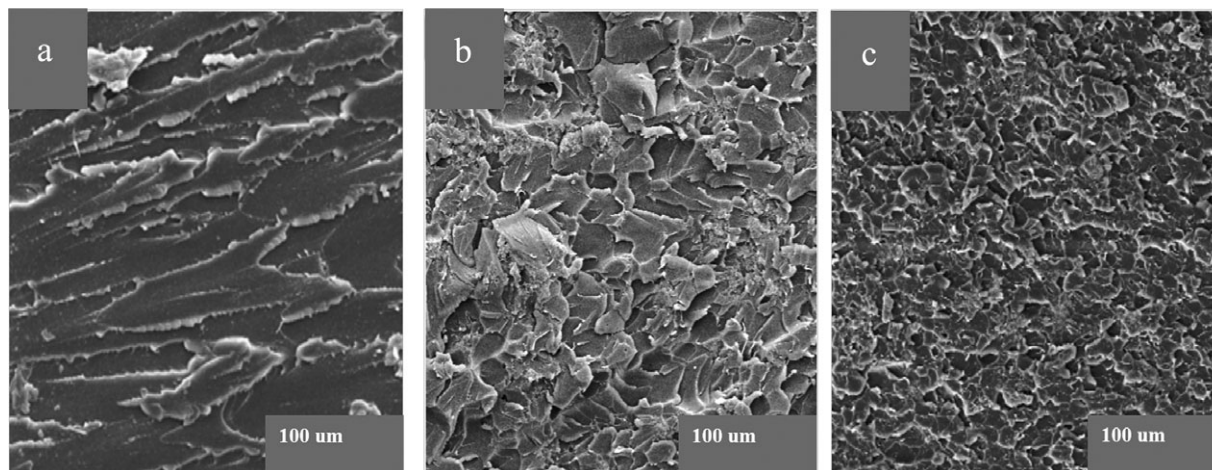
rougher [Figure 3(c)]. The amount of the dimples of the composite on per unit area is further more than BOZ-BMI resin. The increase in the dimples indicates that the fractured composite can consume more energy and the composite may have better toughness properties. The reinforced impact toughness of the HBPSi-SiO<sub>2</sub>/BOZ-BMI can be attributed to that HBPSi-SiO<sub>2</sub> easily dispersed uniformly in the resin matrix and bonded with BOZ, thus resulting in an organic-inorganic hybrid interpenetrating network. When the composites are impacted, much more microcracks are formed and much more energies are absorbed accordingly.<sup>30</sup> Consequently, the HBPSi-SiO<sub>2</sub>/BOZ-BMI composite shows the improved impact strength.

#### Tribological Properties of Materials

The friction coefficient of BOZ pure resin, BOZ-BMI resin and the composites with different amount of HBPSi-SiO<sub>2</sub> under dry conditions as a function of the sliding time is shown in Figure 4. Apparently, the frictional coefficient of BOZ-BMI resin is lower than BOZ pure resin during the sliding time, which demonstrates that the addition of BMI can decrease the frictional coefficient of BOZ. Moreover, the frictional coefficient of the composites decreases greatly by the addition of HBPSi-SiO<sub>2</sub>, and the formation time of transform film on the surface of counterpart steel ring is also shortened. The transform film formation of BOZ pure resin and BOZ-BMI resin need much more time, and transform film is nonuniform, thus resulting in the instability of frictional coefficient. Especially, the phenomenon stated above manifests more evidently on BOZ pure resin. When the content of HBPSi-SiO<sub>2</sub> is 3.0 wt %, the composite not only has the lower frictional coefficient, but also exhibits a more stable frictional coefficient during the friction process than the composites with other content of HBPSi-SiO<sub>2</sub>.

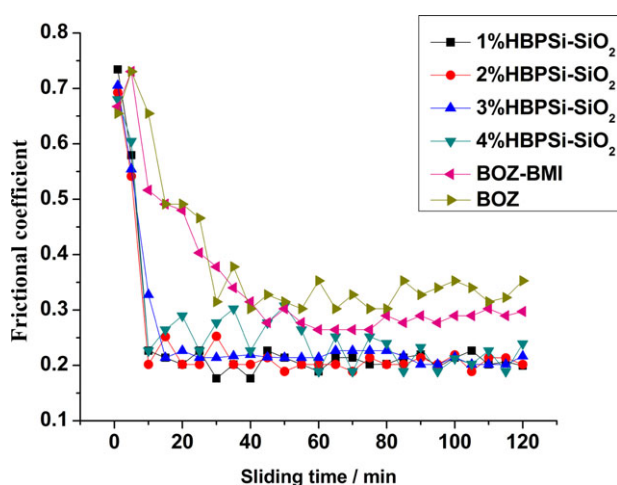
Figure 5 gives the wear rate of BOZ pure resin, BOZ-BMI resin and the composites with different content of HBPSi-SiO<sub>2</sub>. It can be concluded from the figure that BOZ-BMI resin exhibits lower wear rate than BOZ pure resin. The wear rate of BOZ-BMI is  $18.5 \times 10^{-6} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$ , decreases as much as 50.5% than that of BOZ pure resin, which wear rate is  $37.4 \times 10^{-6} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$ . The wear resistance of the composites is further improved by the addition of HBPSi-SiO<sub>2</sub>. When the content of HBPSi-SiO<sub>2</sub> is 3.0 wt %, the lowest wear rate of the composites is merely  $0.4 \times 10^{-6} \text{ mm}^3 \text{ N}^{-1} \text{ m}^{-1}$ , and decreases as much as 97.8% compared with BOZ-BMI resin.

The tribological properties of a composite depend on the internal strength and lubricating property of both matrix and reinforcement.<sup>31</sup> However, BMI with excellent tribological properties, which can largely enhance the tribological properties of



**Figure 3.** SEM of fracture surfaces taken from BOZ, BOZ-BMI, and the composites with 3.0 wt % HBPSi-SiO<sub>2</sub> in impact test (a: BOZ; b: BOZ-BMI; c: composites with 3.0 wt % HBPSi-SiO<sub>2</sub>).

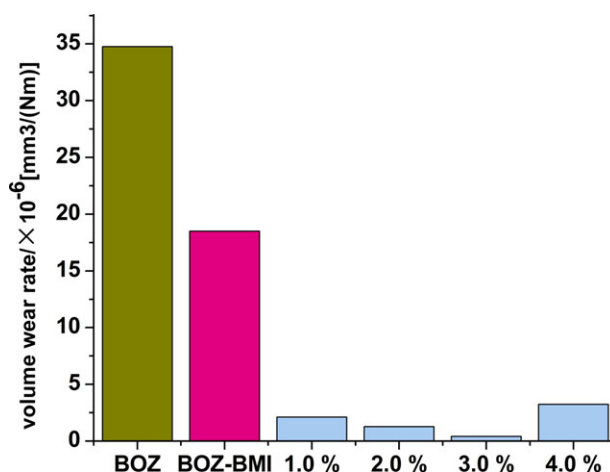
BOZ by cocuring. Meanwhile, during the cured process of the prepolymer, the heated allyl ether of the BMI formed allyl phenol, and the allyl phenol can react with BMI,<sup>21</sup> thus the matrix exhibits higher rheological property, which provides an optimal condition for the dispersion of nanoparticles. However, HBPSi-SiO<sub>2</sub> have good self-lubrication properties due to their highly branched, nonentangled structure of HBPSi, which enable the homodisperse of particles in the matrix. Furthermore, the epoxy group on the surface of HBPSi-SiO<sub>2</sub> can form chemical bond with BOZ by ring opening reaction (Figure 6). Consequently, the interfacial adhesion between HBPSi-SiO<sub>2</sub> particles and matrix is enhanced. However, when the amount of HBPSi-SiO<sub>2</sub> is excessive, HBPSi-SiO<sub>2</sub> entangled with each other, thus the advantages of HBPSi-SiO<sub>2</sub> cannot get into the full play. As a result, the tribology properties are also decrease.



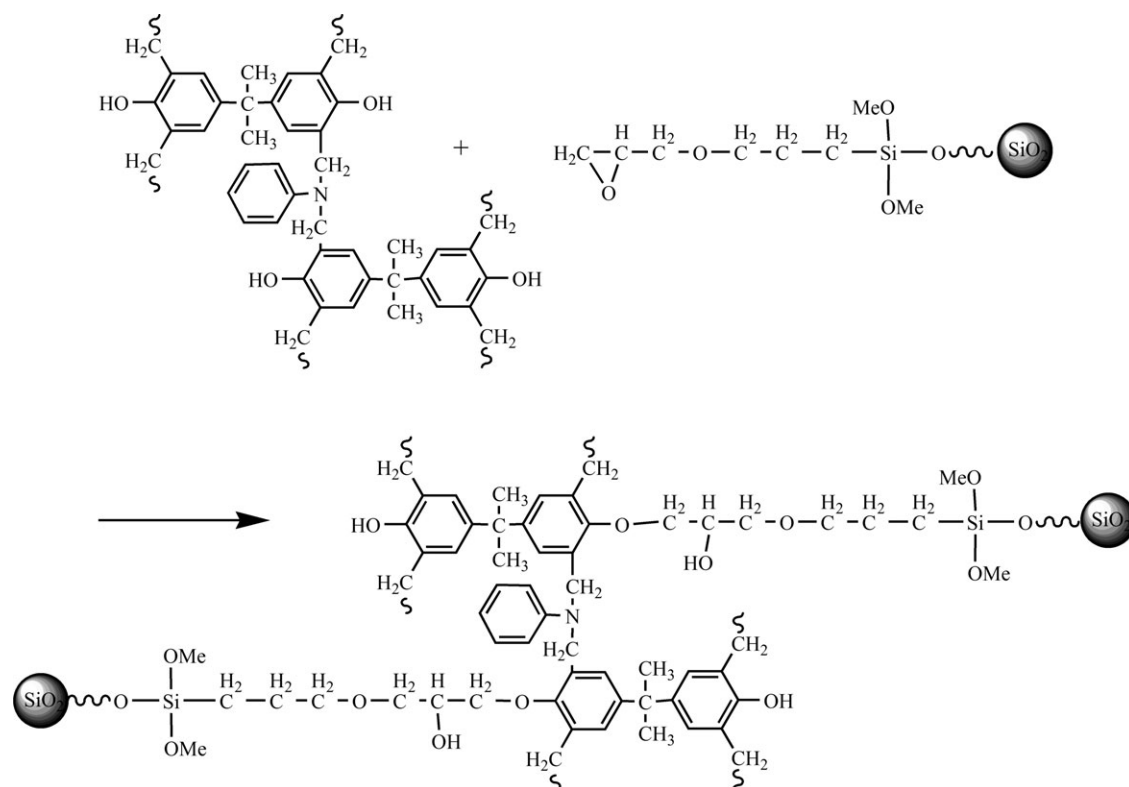
**Figure 4.** The friction coefficient of BOZ, BOZ-BMI, and the composites with different content HBPSi-SiO<sub>2</sub> (load: 196 N; sliding velocity: 0.42 m/s). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

### Wear Mechanism of Materials

To confirm the conclusions above, the morphologies of the wear surfaces of BOZ pure resin, BOZ-BMI resin and its composite filled with 3.0 wt % HBPSi-SiO<sub>2</sub> are selected to investigate the wearing mechanisms. The SEM images of the wear surface the composites under the same testing condition are shown in Figure 7. Obvious cracks and plate-like flake away can be seen on the wear surface of BOZ pure resin [Figure 7(a)], which is the characteristic of fatigue wear mechanism<sup>23</sup> and indicates that the wear resistance of BOZ is poor in sliding against the steel ring. However, for the BOZ-BMI resin [Figure 7(b)], the cracks on the wear surface of the composites are much smaller in comparison with BOZ pure resin, which wear mechanism, can be attributed to the adhesive wear. The result demonstrates that BOZ-BMI has the better antiwear properties than BOZ. In contrast, the wear surfaces of the composites filled with 3.0 wt % HBPSi-SiO<sub>2</sub> [Figure 7(c)] is completely different. Compared with the BOZ pure resin and BOZ-BMI resin, the wear of the



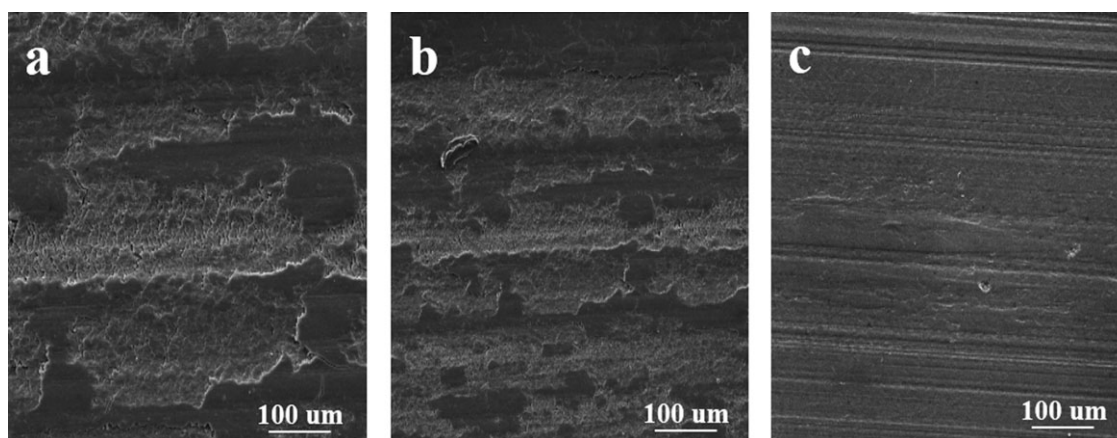
**Figure 5.** The volume wear rate of BOZ, BOZ-BMI, and the composites with different content HBPSi-SiO<sub>2</sub>. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]



**Figure 6.** The reaction between HBPSi-SiO<sub>2</sub> and BOZ.

composites is much milder, which is the characteristic of abrasive wear mechanism, it is also indicates that the antiwear ability of the composite is reinforced by HBPSi-SiO<sub>2</sub>. During the wear process of the composite, material transfer occurs and HBPSi-SiO<sub>2</sub> near the surface of resin matrix can be precipitated, then thin transfer film containing HBPSi-SiO<sub>2</sub> forms on the surface of counterpart steel ring. HBPSi-SiO<sub>2</sub> in the transfer film can play a role of “framework,” which inhibits the damage of the transfer film. Thus, the improvement of the wear resistance of the composites can be ascribed to the self-lubrication properties and “framework” role of HBPSi-SiO<sub>2</sub>. In addition, the good dispersion of HBPSi-SiO<sub>2</sub> in the

BOZ-BMI matrix possibly gives a uniform table and durable lubrication transfer film between the composite and the counterpart steel ring. The transfer film can protect the material during the sliding process, which results in lower frictional coefficient and higher wear resistance. These explanations are in agreement with the results described above that the content of 3.0 wt % HBPSi-SiO<sub>2</sub> composite possesses the highest wear resistance. The improved wear resistance of the content of 3.0 wt % HBPSi-SiO<sub>2</sub> composite can be attributed not only to the enhance functioning of BMI, but also to the fillers, especially to the HBPSi-SiO<sub>2</sub> homogeneously dispersed in resin matrix.



**Figure 7.** SEM of wear surface of BOZ, BOZ-BMI, and the composites with 3.0 wt % HBPSi-SiO<sub>2</sub> (a: BOZ; b:BOZ-BMI; c: composites with 3.0 wt % HBPSi-SiO<sub>2</sub>).

## CONCLUSION

BOZ pure resin, BOZ-BMI resin, and HBPSi-SiO<sub>2</sub>/BOZ-BMI composites are prepared by casting method. The mechanical properties of these materials are investigated. When the content of HBPSi-SiO<sub>2</sub> is 3.0 wt %, the impact strength of composites reaches the maximum value 15.46 kJ m<sup>-2</sup>, increasing 111.4% in comparison with BOZ pure resin. The tribological properties of these materials are also investigated. The results show that the tribological properties of the materials are greatly related to the content of HBPSi-SiO<sub>2</sub>. In detail, reasonable amount of HBPSi-SiO<sub>2</sub> added into BOZ-BMI resin decreases the frictional coefficient and the wear rate of the resins. Composite with the lowest frictional coefficient, wear rate and the best anti-wear properties are obtained with 3.0 wt % HBPSi-SiO<sub>2</sub>. The impact strength of the composite with 3.0 wt % HBPSi-SiO<sub>2</sub> is 15.46 kJ m<sup>-2</sup>, increases as much as 33.6% in comparison with BOZ-BMI resin, and increases 111.4% than BOZ pure resin. The lowest wear rate of the composites is 0.4 × 10<sup>-6</sup> mm<sup>3</sup> N<sup>-1</sup> m<sup>-1</sup>, decreases 97.8% compared with BOZ-BMI resin. The wear mechanism of the materials is also significantly changed with the addition of BMI and HBPSi-SiO<sub>2</sub>. The severe wear of the BOZ pure resin is owing to fatigue wear, the moderate wear of BOZ-BMI resin is attributed to adhesive wear, and the mild wear of the composites with HBPSi-SiO<sub>2</sub> is due to abrasive wear.

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