

Tribological Properties of Bismaleimide Composites with Surface-Modified SiO₂ Nanoparticles

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ABSTRACT: In this article, the surface of SiO₂ nanoparticles was modified by silane coupling agent *N*-(2-aminoethyl)- γ -aminopropylmethyl dimethoxy silane. The bismaleimide nanocomposites with surface-modified SiO₂ nanoparticles or unmodified SiO₂ nanoparticles were prepared by the same casting method. The tribological performance of the nanocomposites was studied on an M-200 friction and wear tester. The results indicated that the addition of SiO₂ nanoparticles could decrease the frictional coefficient and the wear rate of the composites. The nanocomposites with surface-modified SiO₂ nanoparticles showed better wear resistance and lower frictional coefficient than that with the unmodified nanoparticles SiO₂. The specific wear rate and the steady frictional coefficient

of the composite with 1.0 wt % surface-modified SiO₂ nanoparticles are only 1.8×10^{-6} mm³/N m and 0.21, respectively. The dispersion of surface-modified SiO₂ nanoparticles in resin matrix was observed with transmission electron microscope, and the worn surfaces of pure resin matrix and the nanocomposites were observed with scanning electron microscope. The different tribological behavior of the resin matrix and the filled composites should be dependent on their different mechanical properties and wear mechanism. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 110: 1375–1381, 2008

Key words: bismaleimide; SiO₂ nanoparticles; nanocomposites; friction and wear

INTRODUCTION

The polymer composites filled with nanoparticles have good friction and wear properties and have been used in many fields. In recent years, nanoparticles have been used as fillers to improve the tribological performance of polymeric composites, such as ZrO₂ nanoparticles/polyetheretherketone,¹ Al₂O₃ nanoparticles/polyphenylene sulfide,² ZnO nanoparticles/polytetrafluoroethylene,³ TiO₂ nanoparticles/epoxy,⁴ Al₂O₃ nanoparticles/polyimide,⁵ SiC nanoparticles/polyetheretherketone,^{6,7} etc. Besides, the synergistic effects of nano-Al₂O₃ and micro-CaSiO₃ in increasing wear resistance of epoxy resin⁸ and the different pretreatments of nanoparticles effecting on tribological performance of polymer composites were also investigated.⁹ One of the distinct advantages of nanocomposites over microcomposites lies in that the performance improvement is often achieved at relatively low concentration of the nanofillers. This is beneficial to the mechanical properties, processability, and esthetic appearance of the end-

products. Especially, SiO₂ nanoparticles have been used commonly in rubber as reinforcement agent because of its high hardness, high chemical stability, and high thermal conductivity. SiO₂ nanoparticles also have shown superiority in reducing the frictional coefficient and increasing wear resistance of polymer composites. Lai et al.¹⁰ recently found that the addition of SiO₂ nanoparticles improved the frictional coefficient and wear resistance of PA1010 composite coatings. And Li et al.¹¹ found that the frictional coefficient and wear rate of polyimide composite hybrid with SiO₂ nanoparticles was lower in contrast with that of pure polyimide. Su et al.¹² found that SiO₂ nanoparticles significantly improved the wear resistance and decreased the frictional coefficient of Nomex fabric phenolic composites. Also, Wang¹³ found that polyetheretherketone filled with SiO₂ nanoparticles had good friction and wear properties.

Bismaleimide (BMI) resin is the unique combination of high service temperature, good toughness and epoxy-like processing. Especially curing BMI with diallylbisphenol A results in the formation of a high-performance thermosetting resin, which are widely used in some of the most important areas ranging from military programs such as the Air Force to electronic engineering.¹⁴ And in the early years, it was found that BMI resin have good wear

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resistance properties by nature, even the erosion rate of the graphite-fiber-reinforced BMI compositions was stronger than BMI specimens.^{15–17} In our previous works,^{18–20} we found Si₃N₄ nanoparticles, SiC nanoparticles and ZrO₂ nanoparticles could significantly improve the wear resistance and decrease the frictional coefficient of BMI composites. Certainly, the performance of the composites filled with nanoparticles is influenced extensively by the filler morphology, size, volume fraction, and dispersion. Silane coupling agents are often applied to improve the adhesion of the matrix resin to the reinforcement particles.²¹ Recently, Liang et al.²² studied the friction and wear properties of potassium titanate whiskers reinforced BMI composites and the surfaces of whiskers treated by silane coupling agents and titanate coupling agents, respectively. The results showed that the wear-resistance of the matrix was improved by the incorporation of whiskers into the matrix, while the composite containing silane coupling agent treated whiskers had the best wear-resistance. Also, Our Group²⁰ currently found that BMI composites containing silane coupling agent treated ZrO₂ nanoparticles exhibited better tribological properties than the composites containing untreated ZrO₂ nanoparticles. This implies that the dispersion of nanoparticles in resin matrix is very important. In this article, we studied the effect of surface-modified SiO₂ nanoparticles by silane coupling agent on the friction and wear properties of BMI composites.

EXPERIMENTAL

Raw materials and its specifications

The SiO₂ nanoparticles were obtained from Zhejiang HISUN Materials Ltd. Co., China. Its diameter is 30 ± 5 nm, with the density being less than 0.11 g/cm³ and a surface area of 160 ± 20 m²/g. The content of SiO₂ nanoparticles is more than 99.5%. Hydroxide group content on the surface of the nanoparticles is above 19%. The ultraviolet radiation reflectivity is greater than 75%. *N*-(2-aminoethyl)- γ -aminopropyl methyltrimethoxy silane (JH-53) as coupling agent was supplied by Jingzhou Jiangnan Fine Chemical Co. Ltd., China. 4, 4'-Bismaleimidodiphenylmethane was obtained from Hubei Fenggang Chemicals, China. It's a commercial grade yellow powder containing more than 85% of maleimide double bond structure. *O*, *O'*-diallylbisphenol A (DABPA) was obtained from Sichuan Jianguo Material, China. It's industrious-grade, amber-colored viscous liquid at room temperature. The chemical structures of BMI and DABPA as well as JH-53 were represented in Figure 1.

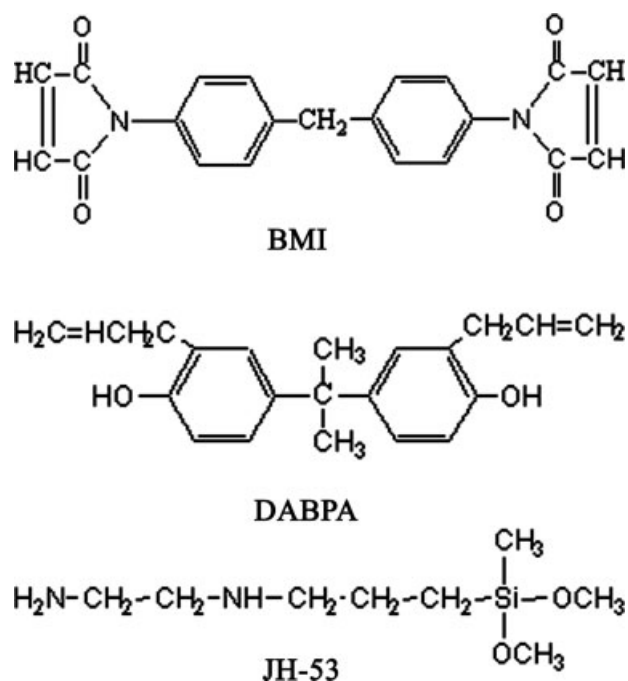


Figure 1 Chemical formulae of BMI, DABPA and JH-53.

Preparation of the BMI composites

The composites of BMI filled with SiO₂ nanoparticles were made by the casting method. First, SiO₂ nanoparticles were ultrasonically dispersed in acetone for about 20–30 min. Both BMI and DABPA were heated at 120°C for 30–40 min under vigorous stirring to form a clear homogeneous prepolymer. Then the fillers with desired proportion were carefully mixed with the prepolymer by a mechanical high shear dispersion process. The mixture consisting of prepolymer, the nanoparticles and acetone was heated to 120°C in a oil bath and kept at this temperature for 30 min with stirring to evaporate acetone. Finally, the mixture was poured into a preheated mold with release agent, and evacuated for additional 30 min at 120°C to remove air bubbles. All the samples were cured following the procedures shown below step by step: 2 h at 150°C, 2 h at 180°C, 2 h at 200°C, and 2 h at 220°C. A postcuring process was 250°C for 6 h.

The BMI composites filled with surface-modified SiO₂ nanoparticles were prepared in the same method as the above method except that the SiO₂ nanoparticles need to be modified by silane coupling agent JH-53 before used. A solution containing 1.0 g coupling agent of JH-53 in water/acetone (5 mL/95 mL) was adjusted pH equals to 5 by acetic acid in favor of hydrolyzation of JH-53 (see Fig. 2), then desired proportion SiO₂ nanoparticles were added into a flask. Under vigorous stirring, this suspension was ultrasonicated at room temperature for 20 min, and heated at 76°C for 1 h. After centrifugation,

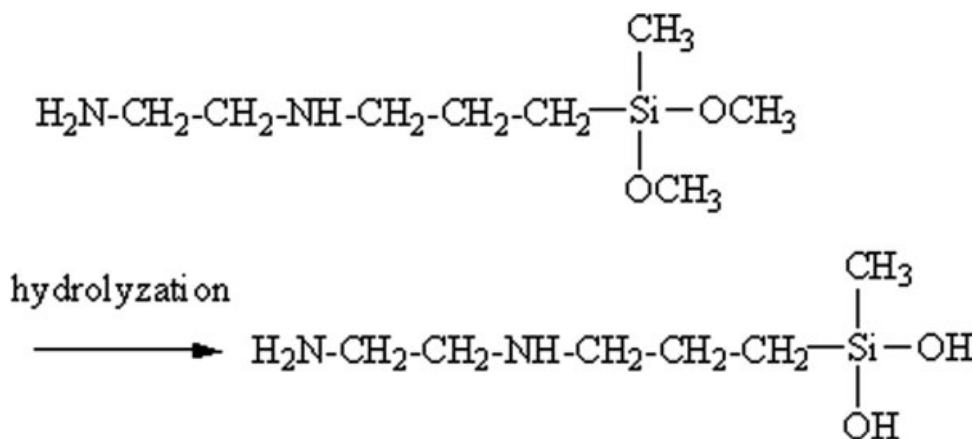


Figure 2 Hydrolysis reaction of JH-53.

washing with ethanol and drying in vacuum at 50°C. The SiO₂ nanoparticles treated by the coupling agent JH-53 were heated at 150–180°C for 2–3 h. So the surface-modified SiO₂ nanoparticles were obtained.

Contact angle measurements

Contact angle measurements on SiO₂ nanoparticles were performed by ref. 23. The wetting liquids were deionized water, glycerin, and *n*-heptane. In which *n*-heptane was used to determine the packing factor. The surface free energy and the London dispersive and polar components for the wetting liquids are listed in Table I.

The London dispersive and polar components of the surface free energy of SiO₂ nanoparticles were determined by measuring the contact angles of a variety of test liquids and the results analyzed by using the geometric mean.

$$\gamma_l(1 + \cos \theta) = 2(\gamma_s^d \gamma_l^d)^{1/2} + 2(\gamma_s^p \gamma_l^p)^{1/2} \quad (1)$$

where the subscripts *l* and *s* represent the liquid and solid states, respectively.

Measurement of the properties of materials

The mechanical properties of pure BMI resin and the composites were carried out by Chinese Standard methods. Flexural strength and impact strength were tested according to GB/T2570-1995 and GB/T2571-1995, respectively. Hardness was measured according to HG2-168-65 under a loading of 62.5 kg and the steel ball with diameter 5 mm.

The sliding wear properties were conducted on an M-200 model friction and wear tester. The contact schematic diagram of the frictional couple was the same as ref. 19. Sliding was performed under ambient conditions over a period of 2 h at a sliding speed

of 0.42 m s⁻¹ and under a load of 196 N. Before each test, the plain carbon steel ring and BMI matrix or its composite block was abraded with No.600 water-abrasive paper, and then the steel ring was cleaned with cotton dipped in alcohol, and the BMI composite blocks were also cleaned in alcohol and dried at 110°C for 2 h to remove the alcohol and moisture and then cooled in a vacuum desiccator at ambient temperature, providing for the initial weight measurement and friction test. At the end of each test, the blocks were cleaned and dried in the same way for the wear weight loss measurement. The density of the filled BMI samples was measured by Archimedes principle using absolute alcohol as the immersing medium. This mass loss can accurately be measured by determining the weight of the specimen before and after the experiment. A characteristic value, which describes the wear performance under the chosen conditions for a tribosystem, is the specific wear rate ω , which was calculated from the relationship

$$\omega = \frac{\Delta m}{\rho \cdot F_N \cdot L} \quad \left(\frac{\text{mm}^3}{\text{N m}} \right) \quad (2)$$

where Δm is the worn specimen mass (g), ρ is the composite specific gravity (g/cm³), F_N is the normal force (N) and *L* is the sliding distance (m).

TABLE I
Characteristics of the Wetting Liquids Used in this Work

Wetting liquid	γ_l^d (mJ m ⁻²)	γ_l^p (mJ m ⁻²)	γ_l (mJ m ⁻²)	η (mPa s)
Water	21.8	51.0	72.8	1
Glycerol	33.7	30.0	63.7	14.8
<i>n</i> -heptane	20.4	0.0	20.4	0.409

γ_l^d is the London dispersive component of the surface free energy; γ_l^p is the polar component of the surface free energy; γ_l is the total surface free energy; and η is the viscosity.

The morphologies of the worn surfaces and wear debris were observed using a AMRAY-1000B model scanning electron microscope (SEM). The distribution of surface-modified SiO₂ nanoparticles in BMI matrix was verified with a transmission electron microscope (TEM).

RESULTS AND DISCUSSION

The tribological properties of the BMI composites

Tribological properties of BMI/unmodified SiO₂ nanoparticles composites

Figure 3 shows the effect of the content of unmodified SiO₂ nanoparticles on the frictional coefficient and wear rate of composites. It can be seen that the steady frictional coefficient decreases with the content increase of the unmodified SiO₂ nanoparticles. This is to say, the higher the proportion of unmodified SiO₂ nanoparticles, the lower the steady frictional coefficient. For example, the frictional coefficient of the bulk matrix was 0.36. Although, the frictional coefficient of the composites with unmodified SiO₂ nanoparticles was reduced from 0.30 to 0.22 when unmodified SiO₂ nanoparticles content increased from 0.5 to 1.25 wt %. Also, it can be seen that the wear resistance of the composites is improved by the filling of the unmodified SiO₂ nanoparticles. The specific wear rate of the composites is also decreased with the increase of the proportion of unmodified SiO₂ nanoparticles. The specific wear rate of the composites can reach the lowest value of $2.1 \times 10^{-6} \text{ mm}^3/\text{N m}$ when the proportion of unmodified SiO₂ nanoparticles is 1.0 wt %. The specific wear rate of the composites is slightly increased to $2.5 \times 10^{-6} \text{ mm}^3/\text{N m}$ when the proportion of unmodified SiO₂ nanoparticles was 1.25 wt %. So, the optimum content of SiO₂ nanoparticles should be 1.0 wt %. And the content of SiO₂ nanoparticles is 1.0 wt % in all materials systems studied.

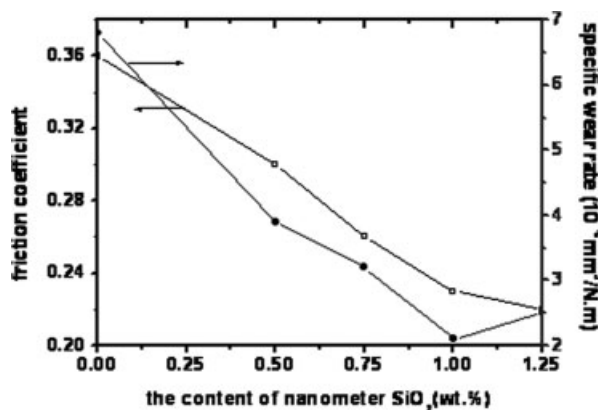


Figure 3 Tribological properties of BMI/unmodified SiO₂ nanoparticles composites.

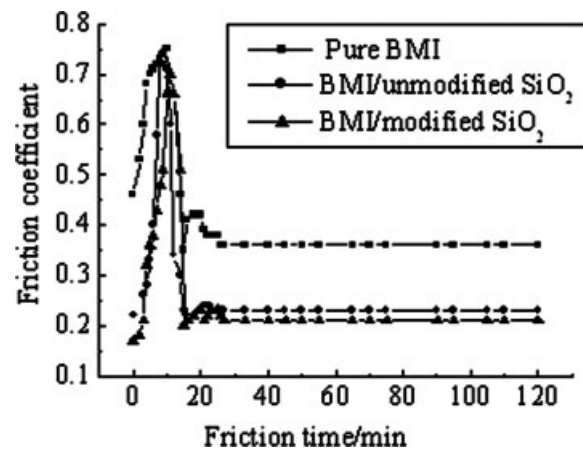


Figure 4 Frictional coefficient as a function of the friction time for the BMI resin matrix and the filled composite (load: 196 N; sliding velocity: 0.42 m s^{-1}).

Tribological properties of BMI/modified SiO₂ nanoparticles composites

Figure 4 shows the frictional coefficient as a function of the sliding time for the matrix and the composite with unmodified 1.0 wt % SiO₂ nanoparticles and the composite with surface-modified 1.0 wt % SiO₂ nanoparticles. It can be seen that the frictional coefficient of all samples attained to a steady-going value in a short time after about 20 min in process of the friction test and appropriate SiO₂ nanoparticles can significantly decrease the frictional coefficient of BMI composites. For example, the steady frictional coefficient of the bulk matrix was 0.36. The steady frictional coefficient of the BMI composites with 1.0 wt % unmodified SiO₂ nanoparticles was 0.23. While, the steady frictional coefficient of the composites with surface-modified SiO₂ nanoparticles was 0.21 at the same content. This is to say, the surface-modified SiO₂ nanoparticles by JH-53 was better than the unmodified SiO₂ nanoparticles in reducing the steady frictional coefficient of BMI composites.

Figure 5 shows the specific wear rate of the BMI matrix and the composite with unmodified 1.0 wt % SiO₂ nanoparticles and the composite with surface-modified 1.0 wt % SiO₂ nanoparticles. It can be seen that the specific wear rate of the composites containing SiO₂ nanoparticles exhibits remarkable change in comparison with that of the pure BMI matrix. The specific wear rate, $6.7 \times 10^{-6} \text{ mm}^3/\text{N m}$, of the pure BMI matrix is decreased to that, $2.1 \times 10^{-6} \text{ mm}^3/\text{N m}$, of the composite containing unmodified SiO₂ nanoparticles when the content of SiO₂ nanoparticles is 1.0 wt %. And the composites containing the surface-modified SiO₂ nanoparticles have better wear resistance than the composites containing the unmodified SiO₂ nanoparticles. The specific wear rate of the composites containing 1.0 wt % surface-modified SiO₂ nanoparticles is only $1.8 \times 10^{-6} \text{ mm}^3/\text{N m}$. That is

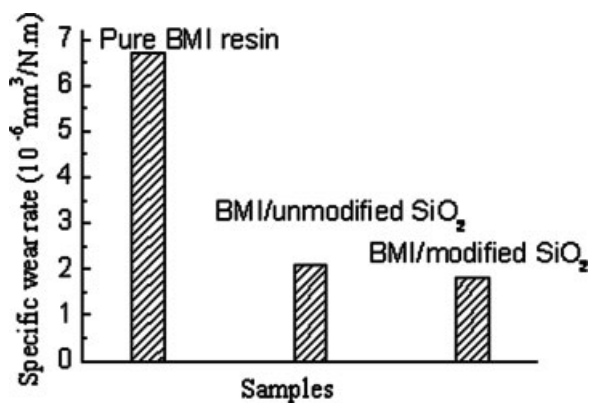


Figure 5 Specific wear rate of the BMI matrix and the filled composites.

to say, the surface-modified SiO₂ nanoparticles compared with the unmodified SiO₂ nanoparticles exhibited better effect in increasing the wear resistance as well as decreasing the frictional coefficient of BMI composites. It should be noted that the content of SiO₂ nanoparticles also influence the tribological properties of the BMI composites. The wear resistance of the composites decreases when nanoparticles proportion exceeds the optimum value. As a matter of fact, we found it was impossible to prepare good samples with SiO₂ nanoparticles above 1.0 wt % because the system including resin prepolymer and SiO₂ nanoparticles so viscous that it is difficult to pour it into the casting mold.

The wearing mechanisms of the BMI composite

It is widely known that the tribological behavior of polymer can sometime be greatly improved by nanoparticles, but the mechanism for nanoparticles action and interaction among the polymer matrix, the nanoparticles and the counterface are not well understood. Especially, the relationship between the tribological properties and mechanical properties of composites filled with nanoparticles has not been adequately studied. To understand the influence of factors on the friction and wear behavior of the composites filled with SiO₂ nanoparticles, hardness and mechanical properties of the composites were investigated.

TABLE II
Hardness, Impact Strength and Flexural Strength of Pure BMI Matrix and the Filled Composite

Samples	Hardness (kg mm ⁻²)	Impact strength (kJ m ⁻²)	Flexural strength (MPa)
Pure BMI resin	25.38	9.6	106.2
BMI/unmodified SiO ₂	27.07	12.3	109.1
BMI/ modified SiO ₂	27.09	13.8	116.9

Table II shows the harness, flexural strength, and impact strength of pure BMI and the composites with SiO₂ nanoparticles. It is seen that SiO₂ nanoparticles can increase the hardness of pure BMI. While, the surface-modified SiO₂ nanoparticles compared with the unmodified SiO₂ nanoparticles have no obvious difference in improving hardness. On the other hand, it is seen that the flexural strength and impact strength of the composite filled SiO₂ nanoparticles are significantly increasing compared with pure BMI, and the composite with surface-modified SiO₂ nanoparticles have better mechanical properties than the composite with unmodified SiO₂ nanoparticles. The reasons is that the surface-modified SiO₂ nanoparticles can easily disperse uniformly in the resin matrix (see Fig. 6), and tend to occupy small "holes" in the resin matrix and to act as a bridge to make more molecular interconnected, resulting in a reduction of the total free volume and limiting the chain segmental motions and reduces the flexibility of the matrix reins.²⁴ Consequently, nanocomposites with surface-modified SiO₂ nanoparticles show a distinct improvement in mechanical properties. On the other hand, the -NH₂ at surface-modified SiO₂ nanoparticles by JH-53 may react with the C=C bond in BMI, resulting in increasing the interface strength between SiO₂ nanoparticles and the resin matrix, as well as an increase in the crosslinking

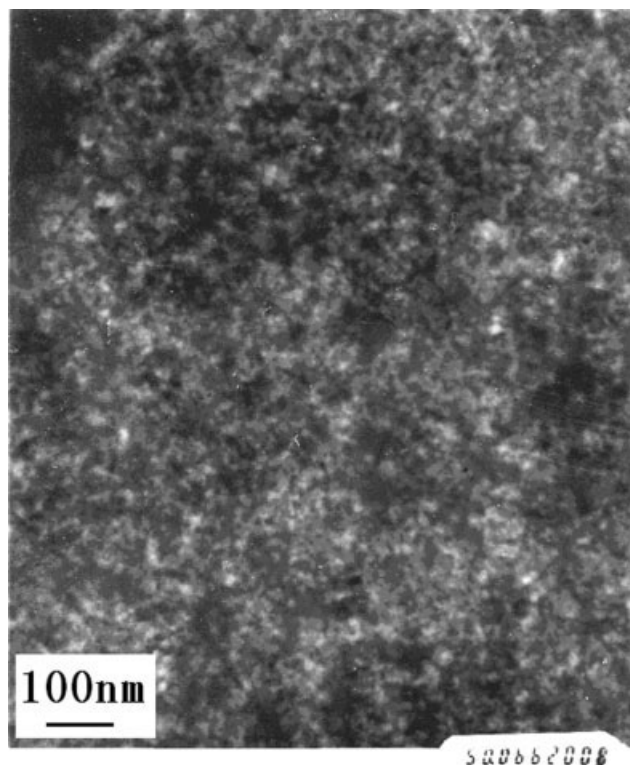


Figure 6 TEM photographs of the composites with surface-modified 1.0 wt % SiO₂ nanoparticles (Amplification: 80,000).

TABLE III
Contact Angles and the Surface Free Energy of SiO₂ Nanoparticles

Nanoparticle SiO ₂	Contact angles on water (°)	Contact angles on glycerol (°)	γ_s^d (mJ m ⁻²)	γ_s^p (mJ m ⁻²)	γ_s (mJ m ⁻²)
Modified	74.9	81.3	30.78	4.52	35.3
Unmodified	60.4	77.5	50.02	2.13	52.1

density. The mechanical properties are just consistent with the improvement in tribological properties. It can also be seen that the hardness of the composite with modified SiO₂ nanoparticles is almost unchanged compared with the composite with unmodified nanoparticles. This implies that the hardness of the composites is just one of the factors influencing the tribological behavior of materials. And the mechanical properties are more important than hardness to determine the tribological behavior of materials. The higher the flexural strength is, the better the wear resistibility is.

Table III lists the contact angle data of SiO₂ nanoparticles on water and glycerol and the results of the surface free energies of SiO₂ nanoparticles calculated from eq. (1). It can be seen that the water contact angles on the surface-modified SiO₂ nanoparticles increased considerably compared with the unmodified. This is due to the fact that the —OH groups at the surface of SiO₂ nanoparticles could react with —OH groups in hydrolyzation of JH-53 (see Fig. 7). Organic functional groups of JH-53 will be grafted to the surface of SiO₂ nanoparticles, resulting in increased hydrophobe properties. It can also be seen that the surface free energies of SiO₂ nanoparticles decreased from 52.1 to 35.3 mJ m⁻² by modified with JH-53. The low surface free energies are favorable to the dispersion of the SiO₂ nanoparticles.

Figure 8 shows the SEM photographs of the wear surfaces on the pure BMI matrix and the composites with unmodified SiO₂ nanoparticles and the composites with surface-modified SiO₂ nanoparticles under amplification of 200 times. It can be seen that the obvious triangular shape opens in the wear direction

and some cracks across the wear tracks on the pure BMI block [see Fig. 8(a)] in accordance with an earlier finding,²⁰ which indicates that, for the pure BMI matrix, the fatigue-crack is the main wear mechanism due to acting pressure and shear loads induced by the movement of the steel counterpart during the friction process. In contrast, the surface of the worn composites containing SiO₂ nanoparticles appears to be completely different. For the composites containing 1.0 wt % unmodified SiO₂ nanoparticles [see Fig. 8(b)], some of slightly plucked marks are visible resulting from possibly the combination between the plastic flow of the BMI resin matrix and lowed reaction of SiO₂ nanoparticles under the high temperature during the sliding process. While, the composites with surface-modified SiO₂ nanoparticles, the worn surfaces are characterized with a little plucked marks [see Fig. 8(c)]. It agrees well with the friction and wear behaviors of the composites with surface-modified SiO₂ nanoparticles. Therefore, the change of wear mechanism is another reason of improvement in tribological properties of the composites containing 1.0 wt % SiO₂ nanoparticles. Certainly, the different wear mechanism of composites with specific proportion SiO₂ nanoparticles also related to the different distribution of SiO₂ nanoparticles in BMI matrix.

CONCLUSIONS

The frictional coefficient and the specific wear rate of the BMI resin can be decreased by filled SiO₂ nanoparticles. The surface-modified SiO₂ nanoparticles compared with the unmodified SiO₂

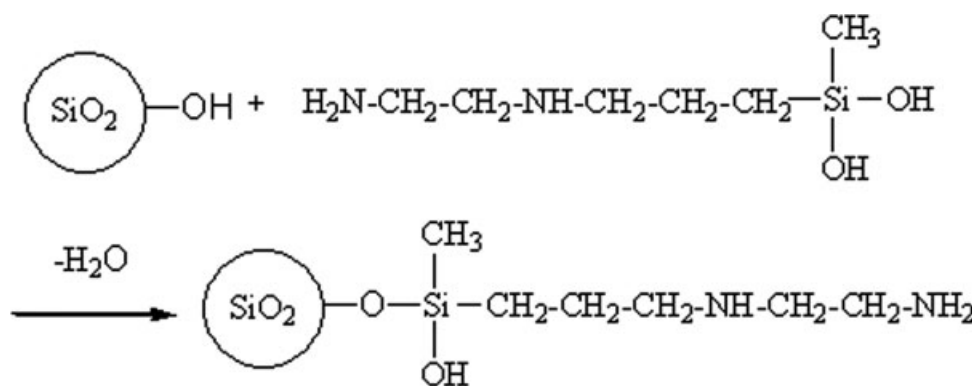


Figure 7 Reaction of —OH groups at surface of SiO₂ nanoparticles with JH-53.

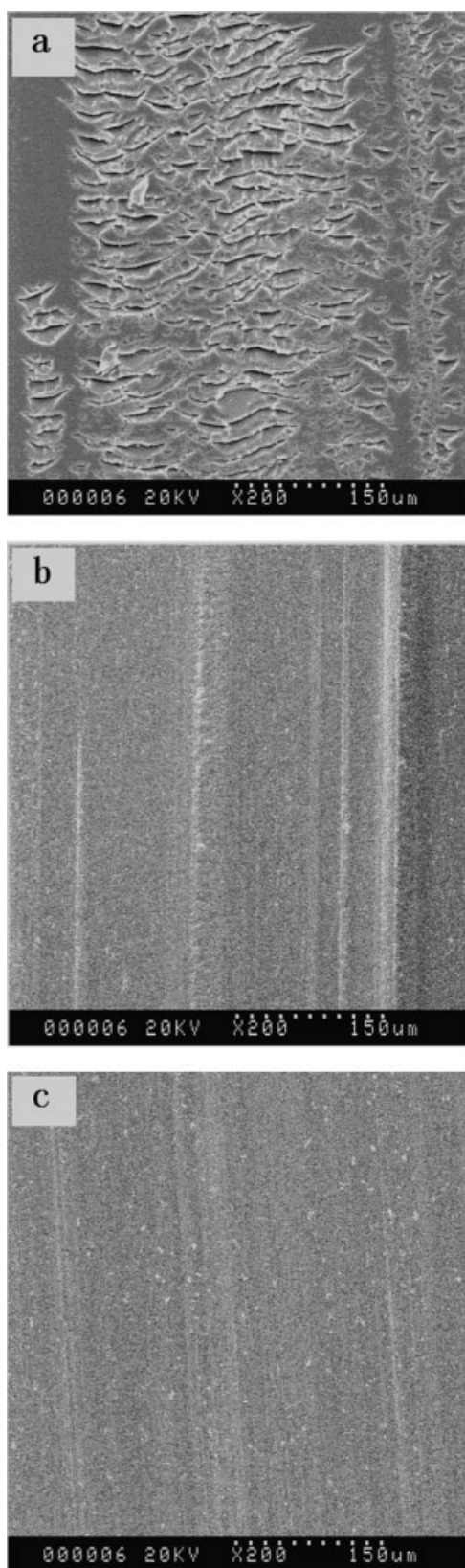


Figure 8 SEM photographs of the worn surface of the BMI matrix and the filled composites.

nanoparticles exhibited better improvement in increasing the wear resistance as well as decreasing the frictional coefficient of BMI composites. The specific wear rate and the steady frictional coefficient of the composite with surface-modified SiO₂ nanoparticles are only $1.8 \times 10^{-6} \text{ mm}^3/\text{N m}$ and 0.21, respectively. The severe fatigue wear of the neat resin matrix can be changed into slight adhesive wear with the addition of the suitable content of SiO₂ nanoparticles. The surface-modified of SiO₂ nanoparticles by *N*-(2-aminoethyl)- γ -aminopropylmethyl dimethoxy silane can distribute at relative uniform in resin matrix. The different tribological behavior of the resin matrix and the filled composites should be dependent on their different mechanical properties and wear mechanism.

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