

# The Tribological Behavior of Micrometer and Nanometer TiO<sub>2</sub> Particle-Filled Poly(phthalazine ether sulfone ketone) Composites

Xin Shao,<sup>1</sup> Weimin Liu,<sup>2</sup> Qunji Xue<sup>2</sup>

<sup>1</sup>College of Materials Science and Engineering, Liaocheng University, Liaocheng 252000, People's Republic of China

<sup>2</sup>State Key Laboratory of Solid Lubrication, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou 730000, People's Republic of China

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**ABSTRACT:** Micrometer and nanometer TiO<sub>2</sub> particle-filled poly(phthalazine ether sulfone ketone) (PPESK) composites with various filler volume fractions from 0.5 to 7.5 vol % were prepared by heating compression molding. The friction and wear behaviors of the PPESK composites were evaluated using the block-on-ring test rig by sliding PPESK-based composite blocks against a mild carbon steel ring under dry friction conditions. The wear debris and the worn surfaces of the PPESK composites filled with micrometer and nanometer TiO<sub>2</sub> particles were investigated by using a scanning electron microscope (SEM), while the structures of PPESK composites and wear debris were analyzed with IR spectra. Experimental results show that antiwear properties of the PPESK composites can be improved greatly by filling nanometer TiO<sub>2</sub> particles, and the friction coefficient decreases when the filler volume fraction is below 2.5%, but when the filler volume fraction is above 2.5% the friction coefficient increases gradually with increasing filler volume fraction. In the case of micrometer TiO<sub>2</sub> filler, wear rates increase with increasing filler volume fractions under iden-

tical test conditions, and the friction coefficients are less sensitive to the filler volume fraction. It was also found that the wear mechanism of micrometer TiO<sub>2</sub> particle-filled PPESK is mainly severe adhesion and abrasive wear, while that of nanometer TiO<sub>2</sub> particle-filled PPESK is mainly slight abrasive wear. In the former case, there are no transfer film formed on the surface of the counterpart steel, and wear debris are in the form of long and large ribbon. While in the latter case, the wear debris was granule and their size was about 10 μm. In case of 1 vol % nanometer TiO<sub>2</sub> particle-filled PPESK composites, the transfer film was fairly thinner and smoother, and the transfer film provided better coverage on the surface of steel ring, while that of 7.5 vol % was thicker and discrete. These account for the different friction and wear behavior of micrometer and nanometer TiO<sub>2</sub> particle-filled PPESK composite. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 92: 906–914, 2004

**Key words:** poly(phthalazine ether sulfone ketone); TiO<sub>2</sub> particles; composites; friction and wear; behavior

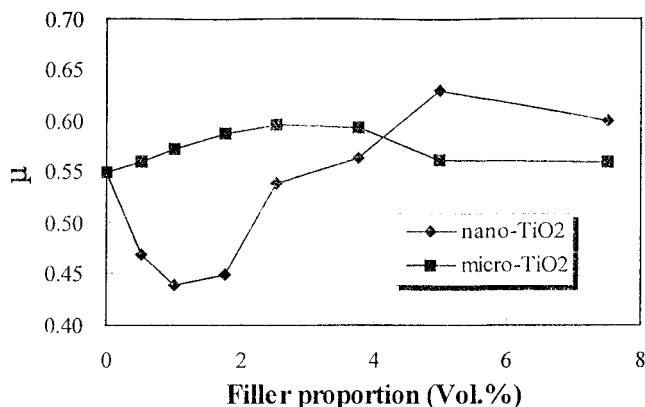
## INTRODUCTION

Polymers and polymer-based composites have been found numerous applications for reducing friction and improving wear resistance.<sup>1–3</sup> Their use is always indicated where fluids are ineffective or cannot be tolerated because of the possibility of contamination of the product or the environment, or the lack of opportunity for maintenance. The majority of polymer-based materials used in industry as bearing materials commonly incorporate fillers and reinforcements, not only to improve the tribological, mechanical, and thermal properties, but also to reduce the materials costs and to improve the processability of the polymer.<sup>4–6</sup> When a polymer composite is used in sliding wear, the filler particle size, its aspect ratio, hardness, concentration, as well as the nature of the interface be-

tween the polymer and the particles, are all important.<sup>4,7</sup>

Many efforts have been made to investigate the tribological behaviors of the micrometer particle-filled polymer composites, and several mechanisms have been suggested to demonstrate the action mechanisms of fillers.<sup>8,9</sup> It should be noticed not much literature is available that has dealt with nanoparticle-filled polymers for tribological applications;<sup>10–12</sup> however, what has been reported seems to show promise. The nanoparticles have the capability of blending well with polymer, which could lead to strengthening of the transfer film. Another difference between a nanoparticle and a microscale particle of the same material is that the nanoparticle has a much higher surface area-to-volume ratio. This ratio may be important for bonding of the particle to the polymer matrix in the composite material. These ideas proposed here led to the study of different effects on the tribological properties of polymer composites made with micrometer and nanometer particles as a filler for sliding applications.

Correspondence to: X. Shao (shxin\_2000@sina.com).



**Figure 1** Effect of filler volume fraction on the friction coefficient of PPEsk composites filled with micrometer and nanometer TiO<sub>2</sub> particles. Sliding velocity 0.43 m/s, load 200 N, duration 90 min.

In recent years there has been growing interest in the development of high-performance polymers for the aerospace, automobile, electrical, and nuclear industries. This is because these types of polymers are technologically important matrices, high-temperature films and adhesives, and cable insulation. Poly(phthalazine ether sulfone ketone) copolymer, with an equivalent molar ratio of sulfone to ketone, coded as PPEsk, is an attractive matrix material owing to its outstanding high temperature stability, excellent mechanical properties, radiation, and hydrolytic resistance, and easy processing.<sup>13</sup>

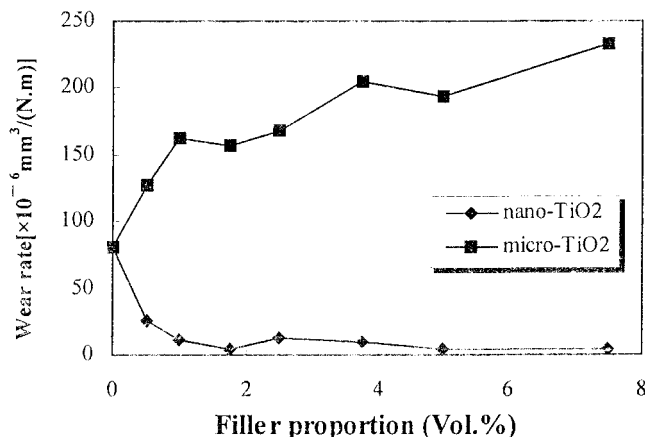
In this study, micrometer and nanometer TiO<sub>2</sub> particle-filled poly(phthalazine ether sulfone ketone) composites were prepared by heating compression molding. An MM-2000 friction and wear tester was used to examine the tribological behaviors of the composites. Scanning electron microscopy (SEM) was used to investigate the morphological features of the worn surfaces and the wear debris. IR spectra were taken to characterize the structure of wear debris and PPEsk composites. It is expected that this effort will be helpful in understanding the different effects of the micrometer and nanometer TiO<sub>2</sub> particles on the tribological behaviors of PPEsk composites under identical experimental conditions.

## EXPERIMENTAL

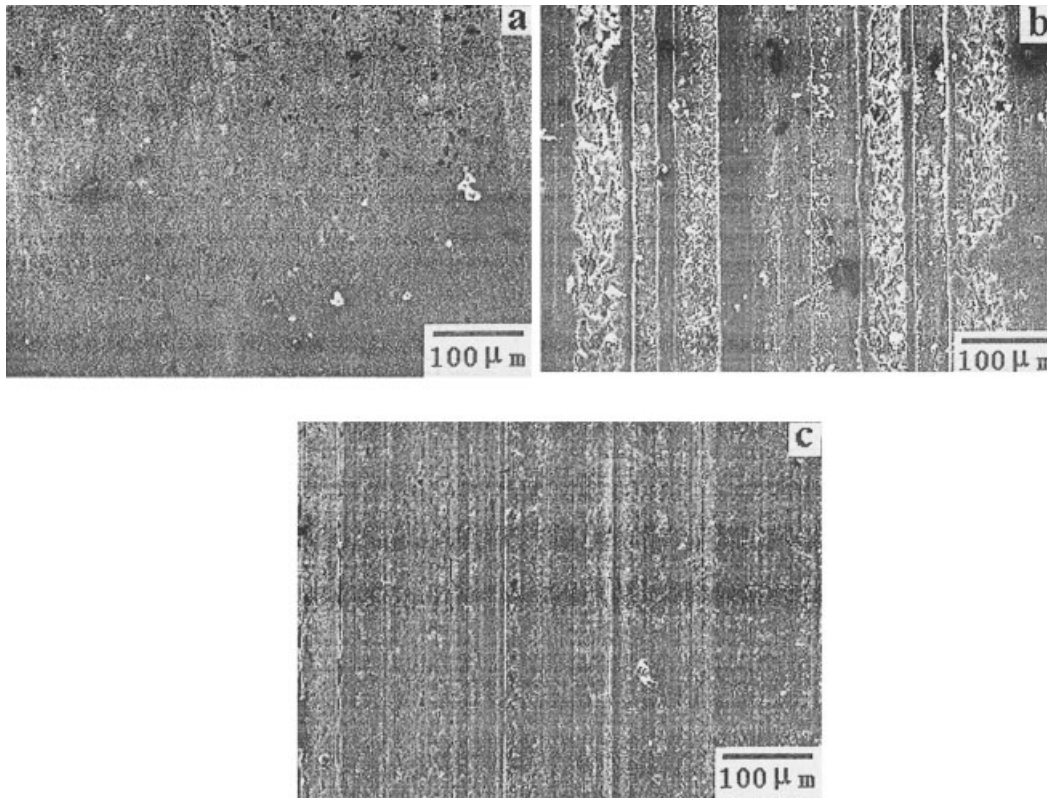
Commercial PPEsk fine powders with a diameter of 40 μm used as the matrix material were provided by Dalian University of Technology (China). The PPEsk was synthesized from bis(4-fluorophenyl) ketone, bis(4-chlorophenyl) sulfone, and 4-(4-hydroxybenzyl)-2,3-phthalazine-1-one through nucleophilic substitution polycondensation.<sup>13</sup> The TiO<sub>2</sub> micrometer particles (<75 μm) were supplied by the Beijing Chemical Factory of China. The TiO<sub>2</sub> nanoparticles in a size of 40

nm were produced by the Zhengyuan Nanomaterial Corporation of Shandong (China). Fillers in varying volume fractions, from 0.5 to 7.5%, were used in this work. The mixture of PPEsk and filler was fully mixed by ultrasonically dispersing them in acetone for about 20 min and then dried at 100°C for 120 min to remove the acetone. The mixture was then compressed in a mold to a pressure of 30 MPa and temperature 340°C at a rate of 5°C · min<sup>-1</sup> and held at 340°C for 30 min. The color of samples changed to darker compared with neat PPEsk. Finally, the mold was cooled to room temperature in air. After releasing of the mold, the resultant composite blocks in a size of 30 × 7 × 6 mm were obtained.

The friction and wear tests were performed in an MM-2000 model friction, and wear tester made by the Xuanhua Tester Factory (China). The details of the contact configuration of the frictional pair have been described previously.<sup>11</sup> The upper block made of PPEsk or filled PPEsk composites and the lower ring made of mild carbon steel (AISI 1045 steel) were all finely, ground with abrasive papers in SiC sand paper until a surface roughness of Ra 0.52–0.88 μm was reached. Both the blocks and the rings were cleaned with acetone, then dried and served for friction and wear tests. In this work, a sliding speed of 0.43 m s<sup>-1</sup>, a load of 200 N, and ambient conditions were used. The friction coefficients of the nanometer and micrometer TiO<sub>2</sub> particle-filled PPEsk composites sliding against AISI 1045 steel were recorded with a computer, connecting with the tester by a sensor. At the end of each test, a total sliding distance of 2330 m for the steel ring, the widths of wear scar were measured to an accuracy of 0.01 mm on a digital-reading optical microscope; then the wear volume loss of the composite specimen was calculated. The wear rate  $\omega$  of each specimen was calculated from the relationship:  $\omega = V/$



**Figure 2** Effect of filler volume fraction on the wear rate of PPEsk composites filled with micrometer and nanometer TiO<sub>2</sub> particles. Sliding velocity 0.43 m/s, load 200 N, duration 90 min.



**Figure 3** SEM micrographs of (a) original surface of pure PPESK, (b) worn surface of pure PPESK, and (c) steel ring of sliding against unfilled PPESK. (Sliding velocity 0.43 m/s, load 200 N, duration 90 min.)

( $SP$ ), where  $V$  is the wear volume,  $S$  the sliding distance, and  $P$  the applied load. Three replicate tests were carried out for each specimen, and the average of the three replicate test results is reported in this work. The deviation of the friction and wear test data was 15%. Wear volume,  $V$ , is calculated using the following equation:

$$V = \left[ \frac{\pi R^2}{180} \cdot \arcsin\left(\frac{b}{2R}\right) - \frac{b}{2} \sqrt{R^2 - \left(\frac{b}{2}\right)^2} \right] \cdot B \cdot \Delta \cdot \Lambda \quad (1)$$

where  $R$  is radius of steel ring,  $b$  and  $B$  are the width of the wear scar and the block, respectively.

The morphologies of the wear traces of micrometer and nanometer  $\text{TiO}_2$  particle-filled PPESK composite and the wear debris generated during the composites sliding against mild carbon steel were observed with a JSM-5600LV scanning electron microscope (SEM). IR spectra were taken on a Bio-Rad FTS-165 IR spectrometer, with operated from 4000 to 500  $\text{cm}^{-1}$ , to characterize the structure of wear debris and PPESK composites.

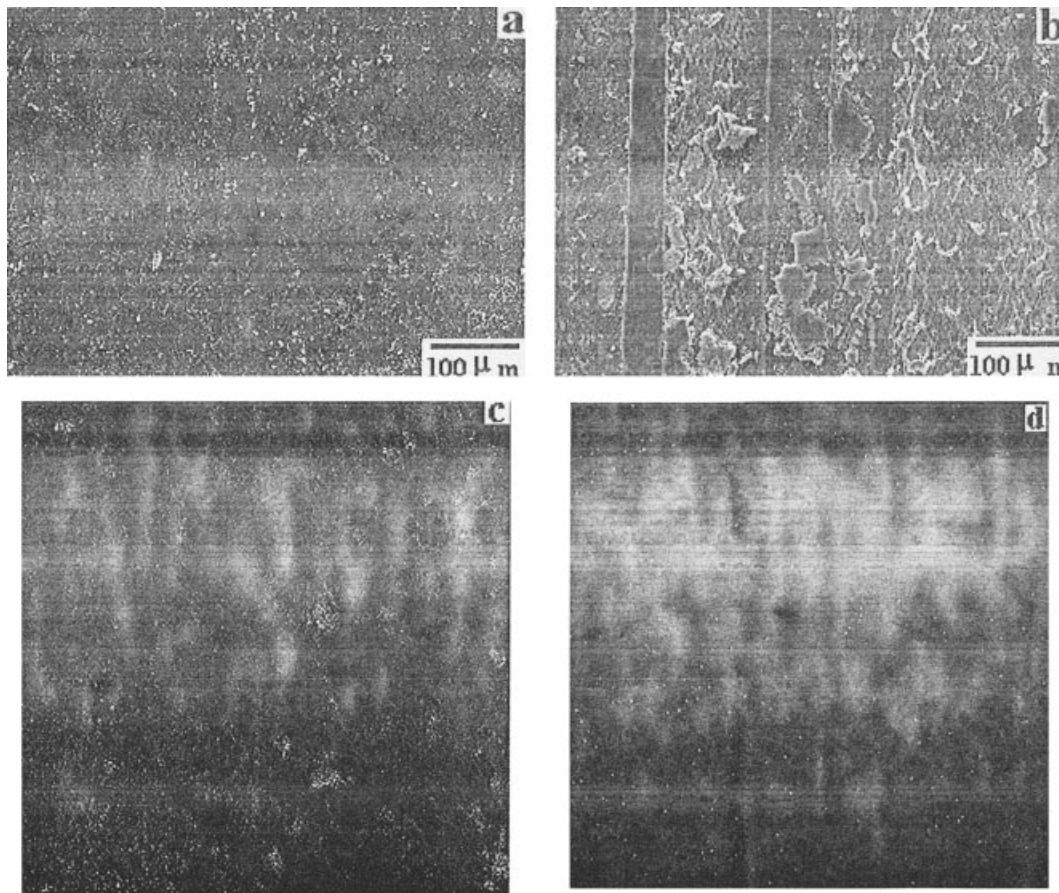
## RESULTS AND DISCUSSION

### Friction and wear properties

Figure 1 shows plots of the friction coefficient vs. filler volume fraction for micrometer and nanometer  $\text{TiO}_2$

particle-filled PPESK composites in dry sliding friction. It is seen from Figure 1 that the friction coefficient of nanometer  $\text{TiO}_2$  particle-filled PPESK composite first decreases greatly with the increase of the filler volume fraction and then increases as the filler volume fraction of the nanometer  $\text{TiO}_2$  particle increases. Namely, at a nanometer  $\text{TiO}_2$  particle volume fraction below 1%, the friction coefficient almost linearly decreases with an increase of the  $\text{TiO}_2$  volume fraction. When the filler volume fraction of the nanometer  $\text{TiO}_2$  particle in PPESK is 1%, the friction coefficient is the lowest (about 0.43). Furthermore, when the volume fraction is above 2.5%, the friction coefficient increases gradually with increasing filler volume fraction, which is higher than that of unfilled PPESK (which is 0.59). This indicated that nanometer  $\text{TiO}_2$  particles are beneficial to reduce the friction of PPESK when the filler volume fraction is below 2.5%. Contrary to the above case, the friction coefficient of micrometer  $\text{TiO}_2$  particle-filled PPESK composite is less sensitive to the filler volume fraction, as shown in Figure 1. It is apparent that irrespective of the micrometer  $\text{TiO}_2$  particle volume fraction in PPESK the friction coefficient is higher than that of unfilled PPESK under the same test conditions, but the rise in value is very small.

The wear rate of nanometer and micrometer  $\text{TiO}_2$  particle-filled PPESK composites against filler volume fraction is plotted in Figure 2. In case of the nanometer



**Figure 4** SEM micrographs of (a) original surface of micrometer TiO<sub>2</sub> particle-filled PPESK; (b) worn surface of micrometer TiO<sub>2</sub> particle-filled PPESK; (c) the Ti element distribution corresponding to (b); and (d) the Fe element distribution corresponding to (b). (Sliding velocity 0.43 m/s, load 200 N, duration 90 min.)

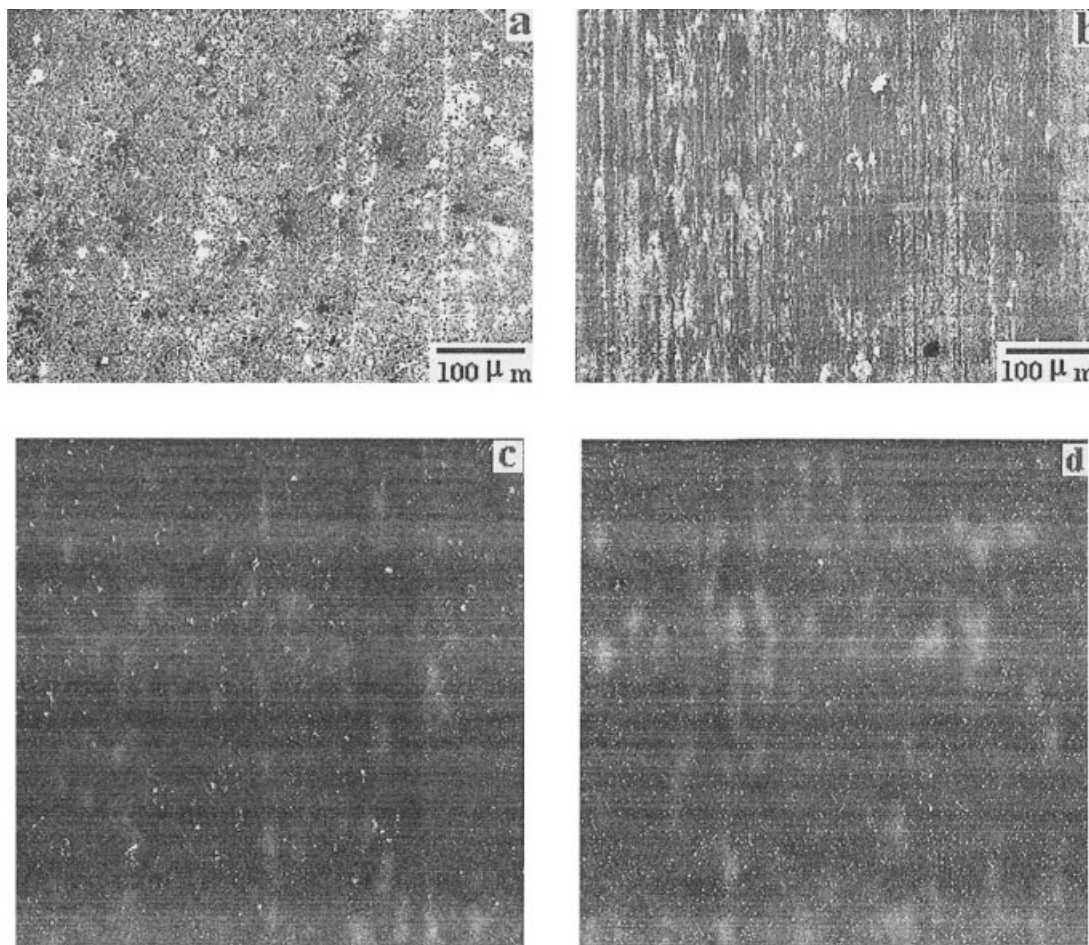
TiO<sub>2</sub> particle, the wear rate of composite is observed to fall greatly with small additions of nanometer TiO<sub>2</sub> in PPESK, and thereafter the reduction in wear rate is very small with the increase of the filler volume fraction. The flattening of the curve between 1 and 7.5% filler volume fractions indicates that most of the beneficial wear-reducing effect has been achieved with these volume fractions. In case of 1.75%, the wear rate of nanometer TiO<sub>2</sub> particle-filled PPESK composites is about  $4.86 \times 10^{-6} \text{ mm}^3/\text{N} \cdot \text{m}$ , which is only about 6% than that of unfilled PPESK ( $80.12 \times 10^{-6} \text{ mm}^3/\text{N} \cdot \text{m}$ ). A volume fraction higher than 7.5% is thus not desirable because with higher filler proportions the composites tend to become fragile. In the case of the micrometer TiO<sub>2</sub> filler, wear rates of composites increased with increasing filler volume fractions under identical test conditions. At filler volume fractions between 1 and 7.5%, the wear rates of micrometer TiO<sub>2</sub> particle-filled PPESK composites are higher by a factor of 1.5 to 3 than that of unfilled PPESK. In combination of Figure 1 and 2, it is interesting to note that nanometer TiO<sub>2</sub> particle-filled PPESK exhibits a lower friction coefficient and wear rate than that of

unfilled PPESK, while those of micrometer TiO<sub>2</sub> particle-filled PPESK are higher than that of unfilled PPESK. Such a difference could be attributed to the different specific surface area and surface reactivity of nanometer and micrometer fillers, and thereby the different bonding strength between the matrix and the fillers.

#### Investigation of wear mechanisms

To understand the different effects of the micrometer and nanometer TiO<sub>2</sub> particles on the tribological behavior of PPESK under identical experimental conditions, the wear traces and the wear debris were studied by means of scanning electron microscopy. Figure 3(b) shows the worn surface of unfilled PPESK after sliding against a mild carbon steel ring. Comparison of this with the unrubbed surface of pure PPESK [shown in Fig. 3(a)] shows that severe adhesion was the dominant wear mechanism for unfilled PPESK.

The micrograph of the worn surface of the micrometer TiO<sub>2</sub> particle-filled PPESK composite is shown in Figure 4(b). It can be seen that obvious marks of

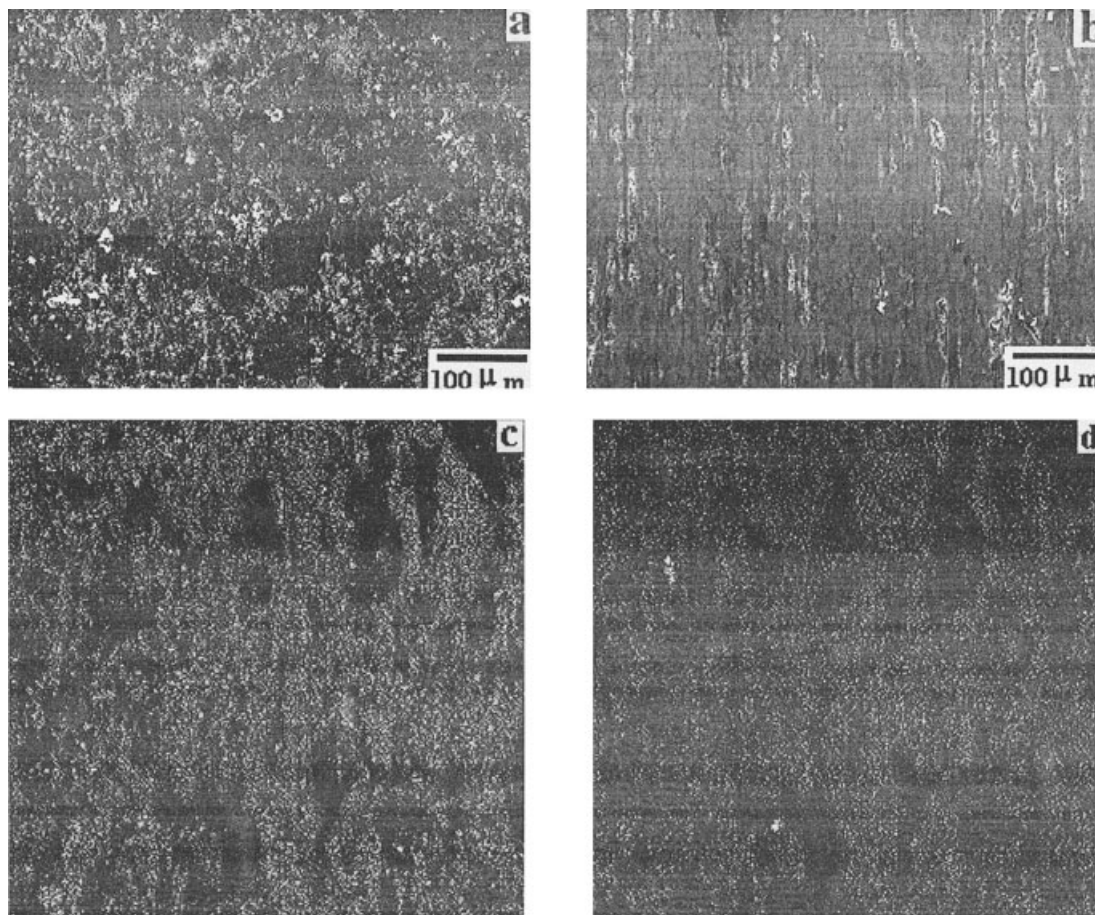


**Figure 5** SEM micrographs of (a) original surface of 1 vol % nanometer TiO<sub>2</sub> particle-filled PPESK; (b) worn surface of 1 vol % nanometer TiO<sub>2</sub> particle-filled PPESK; (c) the Ti element distribution corresponding to (b); and (d) the Fe element distribution corresponding to (b). (Sliding velocity 0.43 m/s, load 200 N, duration 90 min.)

severe adhesion appeared on the worn surface of the composite after sliding micrometer TiO<sub>2</sub> particle-filled PPESK against mild carbon steel; meanwhile, many stripped flake-like debris can be seen adhered to the worn surface. In this case, the bonding strength between polymer matrix and micrometer filler was very weak, and the micrometer TiO<sub>2</sub> particle served as an abrasive, which makes a higher friction coefficient and wear rate. It has been widely recognized that in metal-polymer systems transfer films play an important role in determining the tribological behavior. The micrometer TiO<sub>2</sub> particle-filled PPESK composite hardly formed any transfer film on the steel ring [shown in Fig. 8(a)]. This indicates the inability of this filled composite to form transfer film. The absence of transfer film resulted in virtually no protection of the composite block surface from the hard asperities on the steel ring, so the friction coefficient and wear rates of this composite were higher than those of unfilled PPESK, as shown in Figure 1 and 2. The friction coefficient in the case of micrometer particle-filled PPESK is presumably higher because of partial bubbling be-

tween the exposed inorganic filler particles and the metal counterface, thereby reducing the lubricating effect of the polymeric material. The wear debris of micrometer TiO<sub>2</sub> particle-filled PPESK seem to be in the form of a long ribbon [Fig. 7(b)]. The debris were probably generated from the worn surface during the sliding micrometer TiO<sub>2</sub> particle-filled PPESK composite against the mild carbon steel. Thus, it can also be inferred that the bonding strength between PPESK and micrometer TiO<sub>2</sub> is weak, which leads to higher wear rates of composites. Similarly, the corresponding wear mechanism was severe adhesion and abrasive wear in this case.

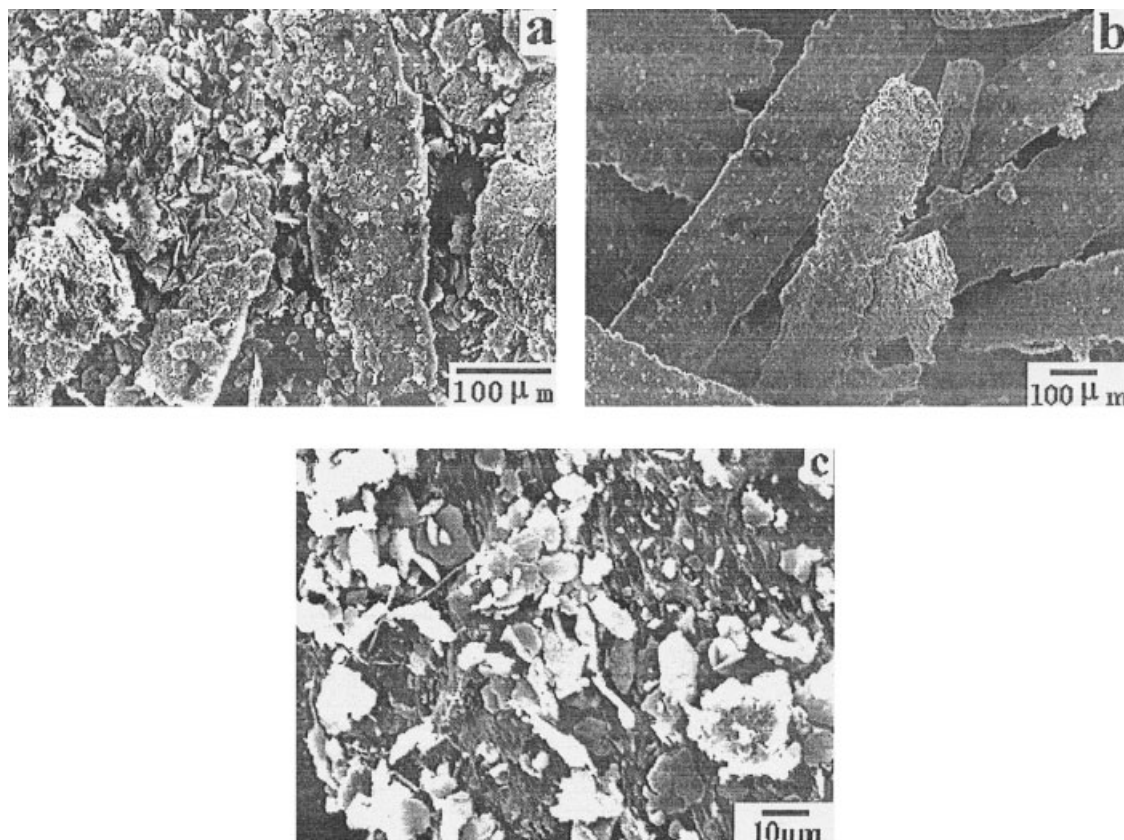
Figures 5(b) and 6(b) give the micrographs of the worn surfaces of 1 and 7.5% nanometer TiO<sub>2</sub> particle-filled PPESK composites. These two nanometer particle cases were selected for this analysis because of the extreme friction behavior, as 1% volume fraction was most effective in reducing friction and 7.5% volume fraction in increasing it. Unlike the above cases, there are no adhesion and flake-like debris on the worn surfaces of the nanometer TiO<sub>2</sub> particle-filled PPESK



**Figure 6** SEM micrographs of (a) original surface of 7.5 vol % nanometer TiO<sub>2</sub> particle-filled PPESK; (b) worn surface of 7.5 vol % nanometer TiO<sub>2</sub> particle-filled PPESK; (c) the Ti element distribution corresponding to (b); and (d) the Fe element distribution corresponding to (b). (Sliding velocity 0.43 m/s, load 200 N, duration 90 min.)

composites. As shown in Figure 5(b), only slight scuffing can be seen on the wear traces of the 1% nanometer TiO<sub>2</sub> particle-filled PPESK. Especially for 7.5% nanometer TiO<sub>2</sub> particle-filled PPESK, the worn surface was smoother [shown in Fig. 6(b)]. As is well known, the nanoparticle has a much smaller diameter of particle, a much greater surface area-to-volume ratio, and more physical and chemical defect on surface, which could lead to the combination of particle and polymer by physical or chemical pattern. These may be important for increasing the binding of the particle to the polymer matrix at polymer-active position in the composite material. This probably accounts for the better wear resistance of the nanometer TiO<sub>2</sub> particle-filled PPESK composite. As shown in Figure 7(c), the wear debris of nanometer particle-filled PPESK was granule and their sizes were about 10 μm, which was probably generated from the transfer film due to repetitive sliding of the ring, thereby generating the granular structure. Thus, it is concluded that nanometer particles increase bond strength between the filler and polymer, while the micrometer particles worsen

the bonding of filler and polymer, and the wear debris is easy to peel from the composite. The transfer film formed on the steel counterface by the 1% nanometer TiO<sub>2</sub> particle-filled PPESK composite is shown in Figure 8(c). It may be seen that the transfer film was fairly thinner and smoother, and the transfer film provided better coverage on the surface of the steel ring. This is obviously the reason why the friction coefficient and wear rate for this material was very small in comparison with unfilled PPESK. Contrary to the above, the transfer film was thicker and covered the steel surface partially in the case of 7.5% [Fig. 8(e)]. The higher friction coefficient was caused probably because of the polishing action between nanometer TiO<sub>2</sub> particles on the friction couple. It can thus be inferred that TiO<sub>2</sub> particles as fillers in PPESK lead to a change of the wear mechanism of the composites. In other words, the wear mechanism of the micrometer TiO<sub>2</sub> particle-filled PPESK is mainly severe adhesion and abrasive wear, while that of nanometer TiO<sub>2</sub> particle-filled PPESK is mainly slight abrasive wear. This would confirm that the filling of nanometer TiO<sub>2</sub> particle in



**Figure 7** SEM micrographs of the wear debris of (a) unfilled PPESK; (b) micrometer  $\text{TiO}_2$  particle-filled PPESK; and (c) nanometer  $\text{TiO}_2$  particle-filled PPESK. (Sliding velocity 0.43 m/s, load 200 N, duration 90 min.)

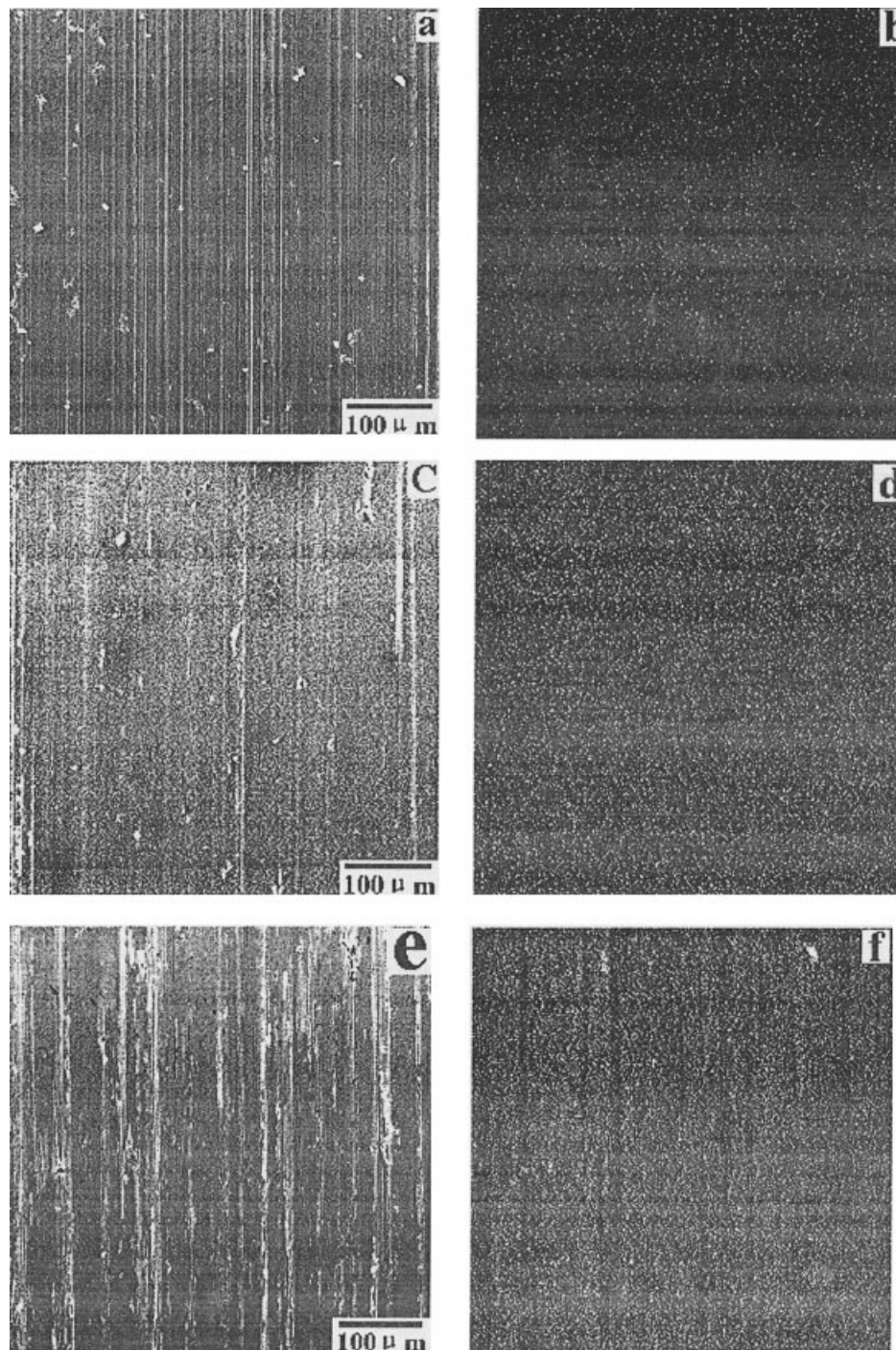
moderate volume fraction can modify the tribological properties of PPESK composite. Due to the polishing action of nanometer  $\text{TiO}_2$ , when volume fraction is higher ( $>1\%$ ), the friction coefficient increases.

Figure 9 shows the FTIR transmission spectra of PPESK composites and wear debris generated during sliding PPESK composites against the mild steel ring. From IR spectra of unfilled PPESK and wear debris of unfilled PPESK [Fig. 9(a) and (b)], it can be seen that after friction the C—O—C peak ( $1240\text{ cm}^{-1}$ ) became strong and the Ar—C=O ( $1666\text{ cm}^{-1}$ ) peak became weak; this indicated that the double band in Ar—C=O is active and easy to react with Fe (steel ring). So the transfer film of unfilled PPESK is thick and lumpy [Fig. 3(c)]. Although the IR spectra of micrometer  $\text{TiO}_2$  particle-filled PPESK and the wear debris were almost the same, this perhaps indicated that the bonding strength between the polymeric material and the particles is weak. There is almost no transfer film on the surface of the steel ring due to the heavy abrasive of  $\text{TiO}_2$  particles; consequently, the wear rates of micrometer  $\text{TiO}_2$  particle-filled PPESK composites are higher than that of unfilled PPESK. Similarly, from IR spectra of nanometer  $\text{TiO}_2$  particle-filled PPESK and the wear debris, it can be seen that after friction the

C—O—C peak became strong and the Ar—C=O peak became weak; this indicated that the double band in Ar—C=O is active and easy to react with Fe (steel ring). It can thus be inferred that the bonding strength between polymeric material and nanoparticle is strong, so the transfer film adheres to the steel ring well. In addition, the addition of the nanometer particle could lead to strongly combine the particle and polymer. In this case, the transfer film was continuous, uniform, and thin, and the wear rates of these composites were considerably lower than that of unfilled PPESK. The disappearance of the peak at  $1030\text{ cm}^{-1}$  and the appearance of the peak at  $1047\text{ cm}^{-1}$  after friction only show the shift of Ar—C—O—C ( $1075\text{--}1020\text{ cm}^{-1}$ ). This could account for the wear rates of  $\text{TiO}_2$  particle-filled PPESK composites.

## CONCLUSIONS

From a dry sliding test on micrometer and nanometer  $\text{TiO}_2$  particle-filled PPESK composites, the following conclusions can be drawn: (1) micrometer  $\text{TiO}_2$  particle-filled PPESK composites exhibited increased friction coefficient and wear rate compared with unfilled PPESK. The wear rates of the micrometer  $\text{TiO}_2$  parti-

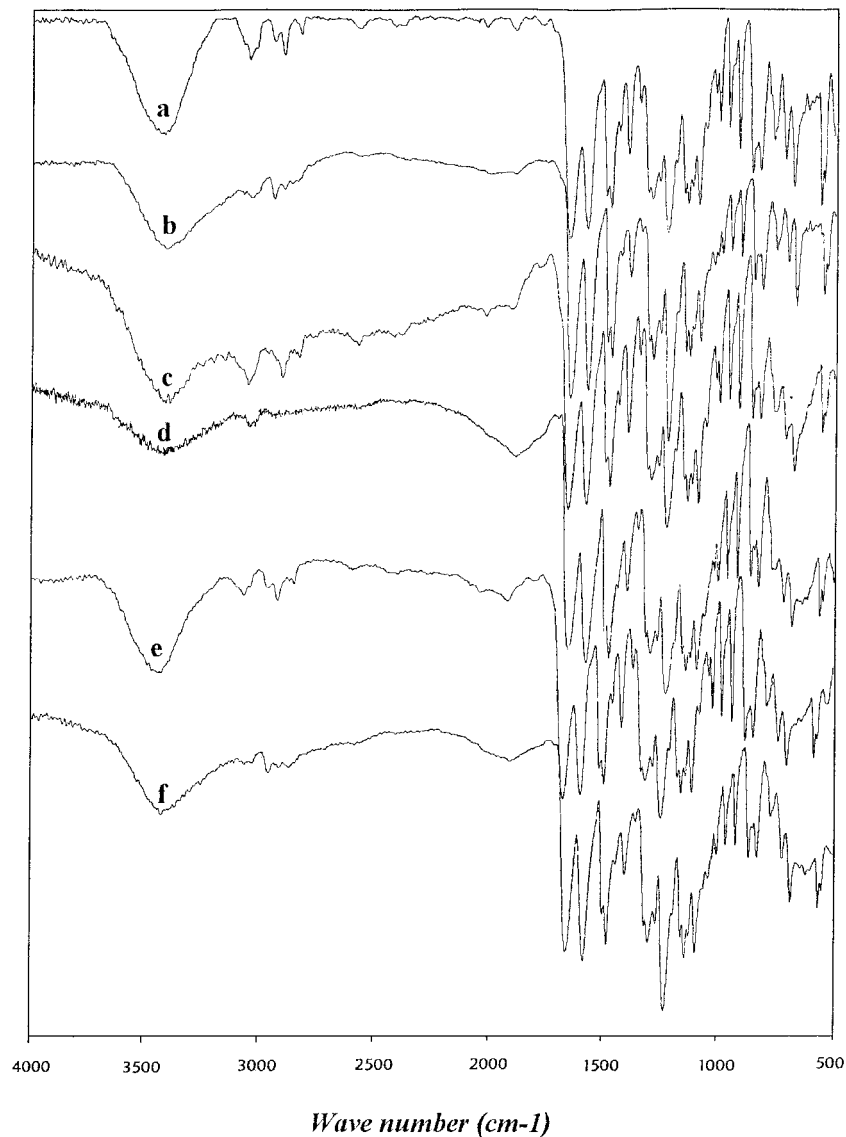


**Figure 8** The scanning electron micrographs of steel rings sliding against of (a) micrometer TiO<sub>2</sub> particle-filled PPESK; (b) the distribution of Ti element corresponding to (a); (c) 1 vol % nanometer TiO<sub>2</sub> particle-filled PPESK; (d) the distribution of Ti element corresponding to (c); (e) 7.5 vol % nanometer TiO<sub>2</sub> particle-filled PPESK; (f) the distribution of Ti element corresponding to (e). (Sliding velocity 0.43 m/s, load 200 N, duration 90 min.)

cle-filled PPESK composite increased with increasing filler volume fractions, and the friction coefficient is less sensitive to the filler volume fraction. (2) The filling nanometer TiO<sub>2</sub> particle could greatly increase the wear resistance of PPESK in all cases under filler volume fractions from 0.5 to 7.5%. The nanometer TiO<sub>2</sub> particle has good friction reduction at a filler

volume fraction below 2.5%, while too a high filler volume fraction corresponds to higher friction coefficient. (3) TiO<sub>2</sub> particles as fillers in PPESK exhibit a distinctive size effect in modifying the wear mechanisms of the composites. In other words, the wear of micrometer TiO<sub>2</sub> particle-filled PPESK is characteristic of severe adhesion, while that of nanometer TiO<sub>2</sub> par-





**Figure 9** IR spectra of PPESK composites and wear debris. (a) unfilled PPESK; (b) debris of unfilled PPESK; (c) 7.5 vol % micrometer  $\text{TiO}_2$  particle-filled PPESK; (d) debris of (c); (e) 7.5 vol % nanometer  $\text{TiO}_2$  particle-filled PPESK; (f) debris of (e).

ticle-filled PPESK is characteristic of slight abrasive wear. In the case of micrometer  $\text{TiO}_2$  particle-filled PPESK, the bonding strength between the polymeric material and the particles is weak, while that of the nanometer  $\text{TiO}_2$  particle-filled PPESK is strong.

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