Effect of Nanometer SiC Filler on the Tribological Behavior of PEEK under Distilled Water Lubrication

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ABSTRACT: The composites of polyetheretherketone (PEEK) filled with nanometer SiC of different proportions were prepared by compression molding. The tribological behaviors of the composites under lubrication of distilled water were investigated and compared with that under dry sliding, on an M-200 friction and wear test rig, by running a plain carbon steel (AISI 1045 steel) ring against the composite block. The worn surfaces of nanometer SiC filled-PEEK and the transfer film were observed by means of scanning electron microscopy (SEM) and electron probe microanalysis (EPMA). As the results, nanometer SiC as the filler greatly improves the wear resistance of PEEK under dry sliding and distilled water lubrication, though the composites show different dependence of wear resistance on the filler content. Nanometer SiC-filled PEEK showed signs of slight scuffing under distilled water lubrication, while a thin, uniform, and tenacious transfer film was formed on the surface of the counterpart steel ring. On the contrary, unfilled PEEK under lubrication of water showed signs of severe plowing and erosion, while the worn surface of the counterpart ring was very rough, and a discontinuous PEEK transfer film was formed. Thus, the different friction and wear behaviors of unfilled PEEK and nanometer SiC-filled PEEK can be attributed to the different characteristics of the corresponding transfer films. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 78: 609–614, 2000

Key words: nanometer SiC; filled polyetheretherketone (PEEK) composite; water lubrication; wear resistance

INTRODUCTION

With its outstanding thermal stability and good resistance to solvent attack and wear, polyetheretherketone (PEEK), a semicrystalline thermoplastic, is becoming a potential candidate for dry friction units under severe conditions, such as bearing components. The friction and wear properties of PEEK and its composites have been focused on by many researchers.[1–3] It has been found that the incorporation of some appropriate reinforcing agents into PEEK has a beneficial effect on its strength and tribological properties.[4–6] However, a few results on the tribological characteristics of PEEK composites under water lubrication have been reported, though the composites can sometimes be used in aqueous environment. It is well known that many plastics and their composites wear much more in water than in air.[7–9] Yamada and Tanaka investigated the wear of polytetrafluoroethylene (PTFE) composites in water, and pointed out that one of the causes of the high wear of PTFE composites in water was the easier separation between fillers and PTFE matrix in an aqueous environment.[10] Watanabe

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investigated the friction and wear mechanisms of various PTFE composites sliding against stainless steel in an aqueous environment, and pointed out that the glass fiber composites wore much more in water, probably owing to increased mating surface roughness resulting from the adhered glass powders.[11]

We reported in our previous work that nanometer SiC as filler in PEEK was very effective in reducing the friction and wear of PEEK under ambient dry conditions.[12] This article deals with the tribological properties of nanometer SiC-filled PEEK composites sliding against a carbon steel counterface under water lubrication. As a comparison, the friction and wear properties of unfilled PEEK were also evaluated under the identical test conditions. It is believed that this work would be helpful for understanding the wear mechanism of nanometer SiC-filled PEEK under distilled water lubrication and for providing guidance to the tribological application of PEEK.

EXPERIMENTAL

PEEK powders of about 100-μm diameter were provided by Jilin University of China. Nanometer SiC filler in a size below 80 nm was produced by the Shenyang Institute of Metal Research of the Chinese Academy of Sciences. The polymer powder was fully mixed with nanometer SiC by dispersing in alcohol in the presence of ultrasonic wave agitation. Then the mixture was dried at 110°C for 6 h to remove the alcohol and moisture, and then the ingredients were mixed in a blade mixer. Finally, the mixture was molded into block specimens by compression molding, in which the mixture was heated at a rate of 10°C min^{-1} to 340°C, held there for 8 min, and then cooled in the mold to 100°C.

The friction and wear tests were conducted on an M-200 model friction and wear tester. The contact schematic diagram of the frictional couple is shown in Figure 1. During the test, the friction force between the tested block and the counterpart steel ring was measured with a torque shaft equipped with strain gauges. The lubrication was produced by a dripping process. Sliding was performed under distilled water lubrication over a period of 1.5 h at a sliding speed of 0.445 m/s. The ambient temperature was around 20°C, and the relative humidity was 50% ± 10%. Before each test, the plain carbon steel ring (HRC 48-50) and the PEEK or its composite blocks were abraded

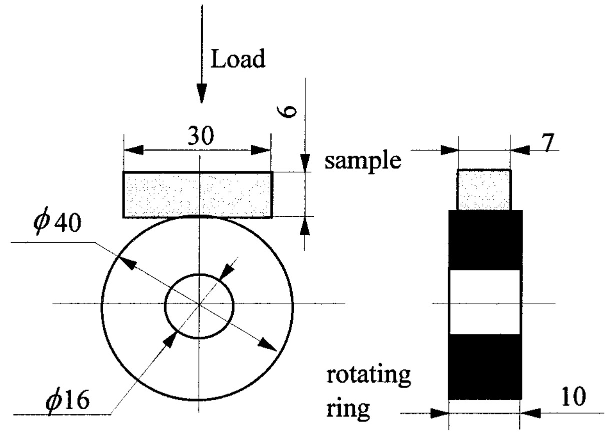

Figure 1 The contact schematic diagram for the frictional couple.

with No. 900 water-abrasive paper. Then the steel ring was cleaned with cotton dipped in acetone. The PEEK or its composite blocks were cleaned with acetone and dried at 110°C for 2 h to remove the acetone and moisture, then cooled in vacuum desiccator to ambient temperature, finally provided for weight measurement and friction and wear tests. At the end of each test, the blocks were cleaned and dried with the procedures same as above, then provided for wear weight loss measurement. The density of the filled PEEK samples was measured by Archimedes' principle using absolute alcohol as immersing medium. The weight loss of the filled PEEK specimens was determined on a balance to an accuracy of 5×10^{-8} kg, and was then converted to volume loss by accounting for the density of filled PEEK. The wear rate ϖ was calculated as below:

$$\varpi = V/(X.L) \quad [\mathrm{mm^3/(N{\cdot}m)}]$$

where V is the volume loss in cubic millimeters, X refers to the sliding distance in meters, and L to the applied load in Newtons. Three replicate friction and wear tests were carried out so as to minimize data scattering, and the average of the three replicate test results is reported in this article. The morphologies of the worn surfaces were observed on a scanning electron microscope (SEM) and an electron probe microanalyzer (EPMA).

RESULTS

Friction and Wear Properties of Nanometer SiC-Filled PEEK

Figure 2 shows the friction coefficient of nanometer SiC-filled PEEK against AISI 1045 steel as a

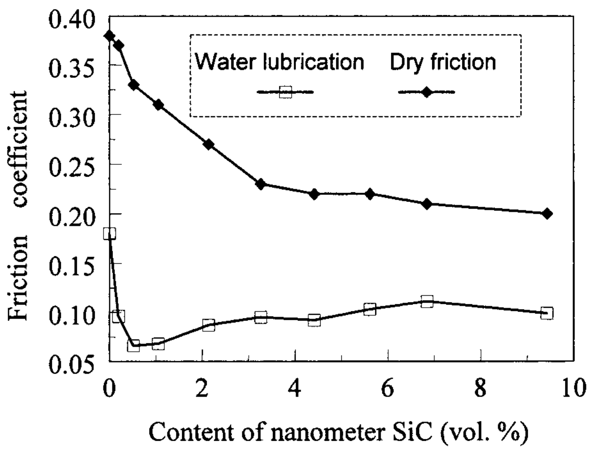

Figure 2 Effect of the content of nanometer SiC on the friction coefficients of filled PEEK under water lubrication and dry friction conditions (load: 196 *N;* sliding velocity: 0.445 m/s).

function of SiC content under water lubrication and ambient dry sliding conditions. It can be seen that unfilled PEEK sliding against AISI 1045 steel registers high friction coefficients under both dry sliding and water lubricated conditions. Especially, both unfilled PEEK and filled PEEK composites show much higher friction coefficients under dry sliding than under water lubrication. Besides, PEEK composites with a filler volume fraction of 0.5 and 1.0% give the lowest friction coefficient under water lubrication; those with higher filler volume fractions show slightly increased friction coefficients, which fluctuate around 0.1. Different from the case in water lubrication, nanometer SiC-filled PEEK composites sliding against AISI 1045 steel under dry sliding condition register gradually decreased friction coefficients at filler volume fractions below 3.0%. Then the friction coefficients assume less dependence on the filler volume fractions. Namely, PEEK composites with filler volume fractions between 4.0 and 9.4% sliding against AISI 1045 steel give friction coefficients around 0.2 under dry sliding conditions. Therefore, it can be concluded that nanometer SiC as filler is effective in improving the friction-reduction behavior of PEEK under both dry sling and water-lubricated conditions, though the composites show differences in the friction-reduction behaviors under the two testing conditions.

Figure 3 shows the wear rates of nanometer SiC-filled PEEK under water lubrication and ambient dry sliding as functions of nanometer SiC content. It is seen that the inclusion of nanometer SiC is effective in decreasing the wear rate of PEEK. This is especially so under water-lubricated condition. In other words, nanometer SiC as a filler is more effective in increasing the wear resistance of PEEK in an aqueous environment. The composites with lower filler volume fractions (below 4.4%) exhibit a decreased wear rate under a dry sling compared with unfilled PEEK; at SiC volume fraction between 1.1 and 4.4%, the wear rate reaches the lowest, and stays nearly unchanged; above 4.4%, the wear rate almost linearly increases with increasing SiC volume fraction. However, above a filler volume fraction 4.5%, the wear rate of filled PEEK under dry sliding rises significantly, and finally surpasses that of the unfilled one. This indicates that the friction-induced thermal and mechanical effects (leading to enhanced softening and plastic deformation of the polymer matrix and detachment of the filler from the polymer matrix) is ineligible under dry friction condition when the filler volume fraction comes to a certain high value. Contrary to the above, irrespective of the filler volume fractions in the composites, such effects are not observed under water-lubricated condition. We assume this is because water as lubricant medium also functions as a cooling agent, and hence, abate the thermal and mechanical effects. Subsequently, the wear rate of filled PEEK under water-lubricated conditions decreases gradually with the increase of nanometer SiC filler volume fraction, the lowest wear rate in this case is obtained as a filler volume fraction 9.4%. Thus, it is concluded that nanometer SiC as filler is more

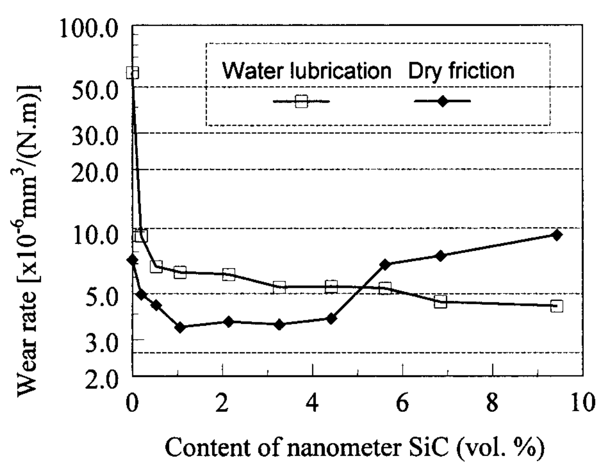

Figure 3 Effect of the content of nanometer SiC on the wear rate of the filled PEEK under water lubrication and dry friction conditions (load: 196 *N;* sliding velocity: 0.445 m/s).

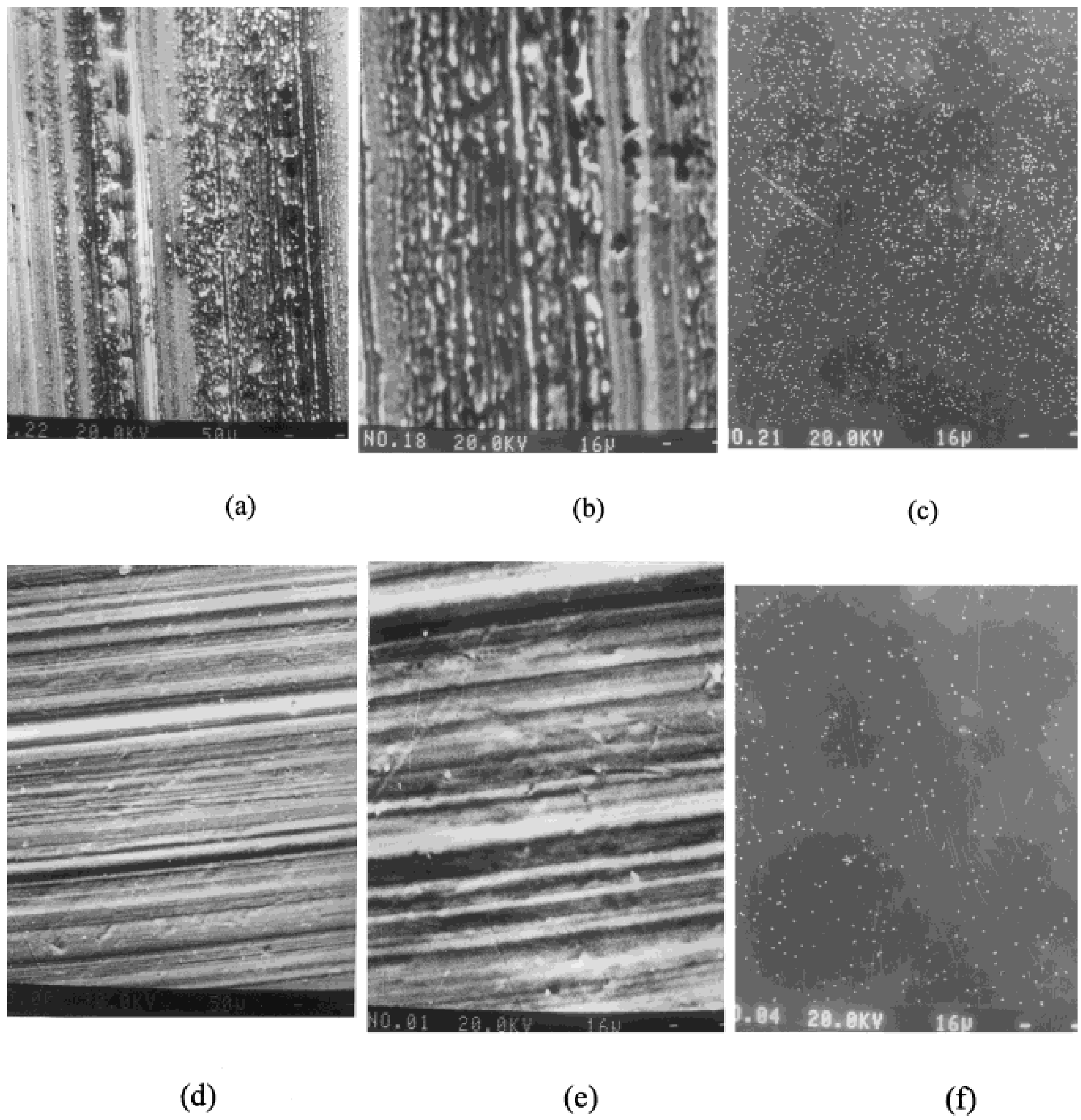

Figure 4 SEM pictures of the worn surfaces of unfilled PEEK block and counterpart steel ring under water lubrication (load: 196 *N;* sliding velocity: 0.445 m/s; test duration: 90 min). (a) worn surface of steel ring; (b) magnified picture of (a); (c) distribution of C in (b); (d) worn surface of unfilled PEEK; (e) magnified picture of (d); and (f) distribution of Fe in (e).

effective in increasing the wear resistance of PEEK under water-lubricated condition.

SEM and EPMA Observation of the Wear Tracks

To understand the effect of nanometer SiC on the friction and wear behavior of the filled PEEK under water lubrication, the worn tracks of unfilled PEEK and nanometer SiC-filled PEEK were observed and analyzed by means of SEM and EPMA. Figure 4 shows the scanning electron micrographs of the wear tracks for the frictional couple of carbon steel ring/pure PEEK block under water lubrication. It can be seen that the worn surface of unfilled PEEK block shows obvious signs of severe scuffing, accompanied by a lot of pits due to water erosion of the composite block [see Fig. 4(d) and (e)]. That of the counterpart steel ring shows signs of adhesion, which corresponds to the transfer of the composite onto the steel surface and the resultant formation of a discontinuous transfer film,

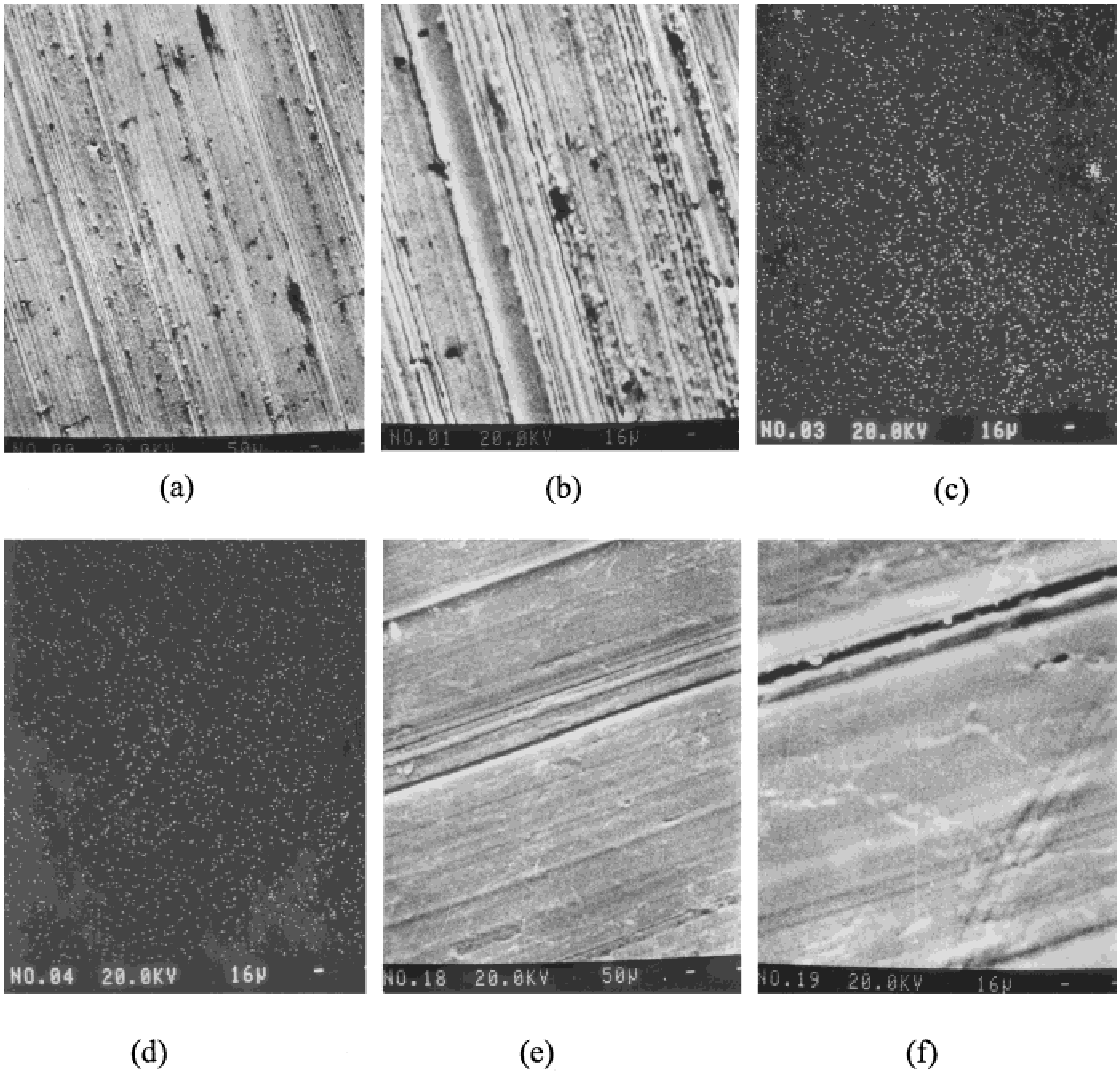

Figure 5 SEM pictures of the worn surfaces of the filled PEEK with 4.4% nanometer SiC and counterpart ring under water lubrication (conditions same as in Fig. 4). (a) worn surface of steel ring; (b) magnified picture of (a); (c) distribution of Si in (b); (d) distribution of C in (b); (e) worn surface of PEEK–SiC composite; and (f) magnified picture of (e).

accompanied by many erosion pits as well [see Fig. 4(a) and 4(b)]. At the same time, the wear debris of the composite transfers onto the worn surface of the steel ring, but the inverse transfer of the steel counterpart wear debris onto the composite surface is almost invisible. In other words, transfer, adhesion, and severe water erosion account for the wear of unfilled PEEK sliding against AISI 1045 steel under water-lubricated condition.

Figure 5 gives the SEM and EPMA pictures of the wear tracks for the frictional couple of carbon steel ring/4.4% nanometer SiC-filled PEEK block under water lubrication. In comparison with what was mentioned above for unfilled PEEK, it is obvious that the inclusion of nanometer SiC in PEEK is effective in abating the adhesion, scuffing, and water erosion of both the composite block and the steel counterpart ring [see Fig. 5(a), (b), (e), and (f)]. Meanwhile, the worn surfaces of both the composite and steel counterpart ring in this case are more smooth as well [see Fig. 5(a) and (e)], though the transfer of composite ingredients onto the steel surface are still observed [see Fig. 5(c) and (d)]. In other words, the inclusion of nanometer SiC in PEEK is very much effective in abating the adhesion, scuffing, and water erosion of both the composite block and the steel counterpart ring. This accounts for the better wear resistance of PEEK composites compared with the unfilled one. However, such an inclusion assumes less effect on the transfer behavior of the polymer-based composites.

DISCUSSION

Under distilled water lubrication the incorporation of nanometer SiC into PEEK caused a great improvement in the tribological characteristics. The PEEK, filled with nanometer SiC, exhibited very lower friction coefficient and wear rate in comparison with unfilled PEEK under water lubrication. For the unfilled PEEK specimen, under water lubrication it was seen that severely ploughed, and plucked marks appeared on the block surface, while the surface of the counterpart ring was rough, and a discontinuous PEEK transfer film was seen. This indicates that unfilled PEEK was hard to form a transfer film on the steel ring surface under water lubrication, or at least the transfer film in this case was unable to provide protection to unfilled PEEK from the abrasion by the asperities on the carbon steel ring surface. Subsequently, the severe scuffing by the rough counterpart surface and water erosion accounted for the high wear rate of unfilled PEEK in aqueous environment. Contrary to the above, nanometer SiC-filled PEEK sliding against the same counterpart under water lubrication formed a thin and uniform transfer film on the counterpart surface, and the adhesion, scuffing, and water erosion were greatly abated as well. Consequently, a considerably lowered wear rate of the filled PEEK was obtained.

CONCLUSIONS

The tribological properties of PEEK composites filled with nanometer SiC of various volume fractions have been investigated under distilled water lubrication and dry sliding conditions by rotating a plain carbon steel ring against the composite block. It has been found that nanometer SiC as filler in PEEK greatly improves the wear and friction properties of filled PEEK under water lubrication. This is because the adhesion, scuffing, and water abrasion of both the composite and the counterpart steel are effectively abated by the inclusion of nanometer SiC filler.

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