The Tribological Properties of SiC Whisker-Reinforced Polyetheretherketone

QIHUA WANG, QUNJI XUE, WEICHANG SHEN

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: SiC whisker-reinforced polyetheretherketone (PEEK) composites with different filler proportions were made into block specimens by compression molding. The friction and wear properties of the composites were investigated on a block-on-ring machine by running a plain carbon steel (AISI 1045 steel) ring against the composite block under ambient conditions. The morphologies of the wear traces and wear debris were observed by scanning electron microscopy (SEM). It was found that SiC whisker-reinforced PEEK exhibited considerably lower friction coefficient compared with pure PEEK, while SiC whisker as a filler at a content of 1.25 to 2.5 wt % was very effective in reducing the wear rate of PEEK. Especially, the lowest wear rate was obtained with the composite containing 1.25 wt % SiC whisker. The SEM pictures of the wear traces indicated that PEEK composites undertook abrasive wear that was enhanced with increasing SiC whisker content, while for the frictional couple of carbon steel ring/composite block (reinforced with 1.25 wt % filler), a thin, uniform, and tenacious transfer film was formed on the ring surface. It was also supposed that the differences in the content of SiC whisker as filler could cause the differences in the wear mechanisms of SiC whisker-reinforced PEEK composites. © 1998 John Wiley & Sons, Inc. J Appl Polym Sci 69: 2341–2347, 1998

Key words: whisker; reinforced polyetheretherketone (PEEK) composite; friction and wear properties; microcracking; abrasive wear

INTRODUCTION

The aromatic thermoplastic polyetheretherketone (PEEK) is suitable for use at high temperature and also has relatively favorable processing capability. With the combination of outstanding thermal stability, good resistance to solvent attack, resistance to damage by irradiation, and good wear resistance, it has been becoming a potential candidate for dry friction units under severe conditions. The friction and wear properties of PEEK and its composites have been focused on by many researchers.[1–4] So far, it has been found that the incorporation of some appropriate reinforcing agents into PEEK has a beneficial effect on its strength and tribological properties. Briscoe et al.[5] said that by adding polytetrafluoroethylene (PTFE) the friction of PEEK was considerably reduced with the sacrifice of small loss in wear resistance. Bahadur et al.[6] investigated the action of various copper compounds as fillers on the tribological behavior of PEEK, and revealed that CuS and CuF_2 as fillers were very effective in reducing the wear of PEEK when sliding occurred against steel surfaces. Voss et al.[7] reported that short glass fibers slightly improved the wear resistance of PEEK, and carbon fibers were superior to glass fibers in enhancing the wear resistance. Wang et

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Correspondence to: Q. Wang.

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al.[8] revealed that nanometer SiC as filler was very effective in reducing the friction coefficient and wear rate of PEEK.

The purpose of this work is to investigate the friction and wear properties of SiC whisker-reinforced PEEK composites with different filler proportions. As a comparison, the friction and wear properties of nonreinforced PEEK were also evaluated under the identical test conditions. It was believed that this work would be helpful for understanding the function of SiC whisker as a filler in PEEK and for providing guidance to the tribological application of PEEK.

EXPERIMENTAL DETAILS

The PEEK fine powders (ICI grade 450P, $\eta = 0.62$) in a diameter of 100 μm were provided by Jilin University of China. The β-SiC whiskers, which were 0.1–1 μm in diameter and 15–80 μm in length (from Shanghai Institute of Ceramics of Chinese Academy of Sciences), were fully mixed with the PEEK powders by ultrasonically dispersing them in alcohol for about 15 min. Then the mixture was dried at 110°C for 6 h to remove the alcohol and moisture. The dried mixture was then made into block specimens in a size of $30 \times 7 \times 6$ mm by compression molding. During the compression molding process the mixture was heated at a rate of 10°C min^{-1} to a maximum temperature of 345°C. The pressure was held at 5 MPa until the temperature of the mixture increased to 320°C. For the rest of the heating cycle the pressure was raised to 15 MPa. The mixture was held at 345°C for 8 min and was then cooled to 100°C in the mold. After releasing of the mold, the resultant block specimen was availed for friction and wear test.

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. Sliding was performed under ambient conditions 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% ± 5%. Before each test, the plain carbon steel ring (hardness of HRC 48–50) and the PEEK or its composite blocks were abraded with No. 900 water-abrasive paper. Then the steel ring was cleaned with cotton dipped in acetone, and the PEEK or its composite blocks were cleaned with acetone followed by drying. At the end of each test, the blocks were cleaned with acetone followed by drying, then provided for wear weight loss measurement. The density of the reinforced PEEK samples was measured by Archimedes' principle using absolute alcohol as immersing medium. The weight loss of the reinforced PEEK specimens was determined on a balance with a sensitivity of 0.05 mg and was then converted into volume loss by using the reinforced PEEK density. The wear rate ω was calculated from the relationship

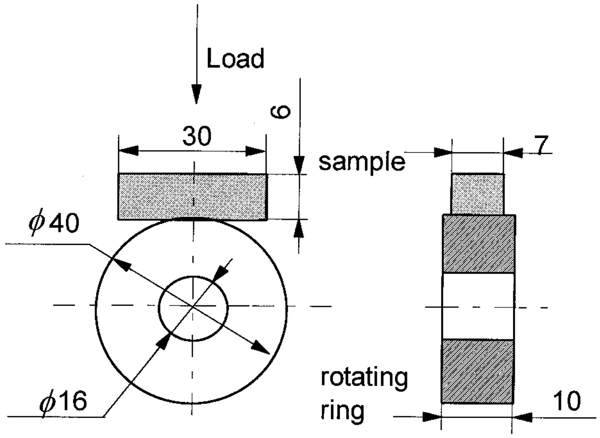

Figure 1. The contact schematic diagram for the frictional couple.

$$\omega = V/(L \cdot F)[\mathrm{mm}^3/(\mathrm{N} \cdot \mathrm{m})]$$

where V is the volume loss in cubic millimeters, L is the sliding distance in meters, and F is the applied load in Newtons. Also, three replicate friction and wear tests were carried out so as to minimize data scattering, and the average of the three replicate test results are reported in this work. The deviation of the data of the replicate friction and wear test was 15%. The morphologies of the wear traces and wear debris were observed using a JEM-1200EX model scanning electron microscope (SEM), with energy-dispersive X-ray analysis (EDAX) attached to the SEM.

RESULTS AND DISCUSSION

Friction and Wear Poperties

Figure 2 shows the friction coefficient and wear rate of SiC whisker-reinforced PEEK as a func-

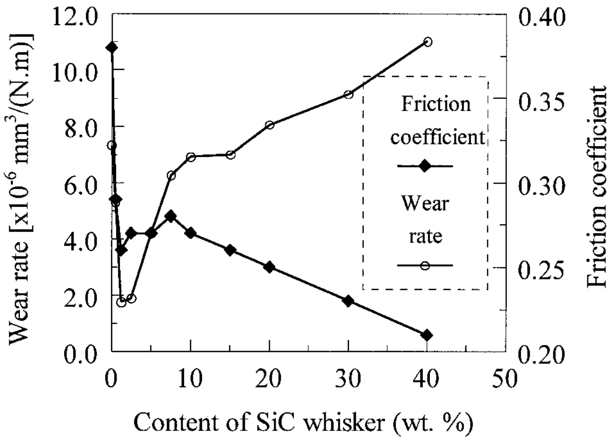

Figure 2. Effect of the content of SiC whisker on the friction coefficient and wear rate of the reinforced PEEK composite (load: 196 *N*; sliding velocity: 0.445 m/s).

tion of SiC whisker content. It can be seen that the SiC whisker-reinforced PEEK composites exhibited a considerably decreased friction coefficient than the pure PEEK. In other words, the friction coefficient decreased sharply when only a little SiC whisker (0.5 to 1.25 wt %) was filled to PEEK. As the SiC content was between 2.5 and 10 wt %, the variation in the friction coefficient was indistinctive. As the SiC content was above 10 wt %, the friction coefficient decreased gradually with the increasing SiC content. The lowest friction coefficient of the composite was obtained as the SiC content reached 40 wt % in this work. In the meantime, the reinforced composite with lower SiC whisker content (below 5.0 wt %) exhibited a considerably decreased wear rate than the unfilled PEEK. Here, the relatively higher wear rate of the unfilled PEEK can primarily be understood by considering that the wear debris of pure PEEK was obviously larger in size than that of reinforced PEEK, as can be seen by naked eye. The wear rate of the composites decreased sharply as the SiC whisker content was below 2.5 wt %. The lowest wear rate of the composites was obtained at the SiC whisker content between 1.25 and 2.5 wt %. When the filler content was above 2.5 wt %, the wear rate gradually increased with an increasing SiC whisker content. As the SiC whisker content reached above 15.0 wt %, the wear rate of the composites was even higher than that of the unfilled PEEK. This indicates that higher content of SiC whisker as filler in PEEK was unsuitable for desired tribological behaviors of the composites. Taking into account both the friction coefficient and the wear rate of the composites prepared in this work, it might be rational to suggest that the optimal content of SiC whisker as filler in PEEK be recommended as 1.25 to 2.5 wt %.

Scanning Electron Microscopy (SEM) Observation

Figure 3 shows the scanning electron micrographs of the worn surfaces of pure PEEK and its composite blocks with various contents of SiC whisker. It can be seen that the plucked and ploughed marks appeared on the wear scar of pure PEEK block, while the worn surfaces of the SiC whisker-reinforced PEEK composites were obviously different. For 1.25 wt % SiC whisker-reinforced PEEK [see Fig. 3(b)], the abated scuffing with lots of microcracks perpendicular to the sliding direction appeared on the wear scar. For those PEEK composites with higher content of SiC whisker [see Fig. 3(c) and (d)], the severe plucked and ploughed marks were seen on the wear surfaces. Especially for 30.0 wt % SiC whisker-reinforced PEEK the formation and transfer of wear debris could be found on the worn surface. EDAX analysis indicated the wear debris transfer consisted mainly of an Fe element. In other words, the counterpart carbon steel ring was indeed abraded and partly transferred to the wear surface of 30.0 wt % SiC whisker-reinforced PEEK. This further indicates that a higher content of SiC whisker as a filler in PEEK was unfavorable for tribological properties. Besides, it can also be inferred that the morphologies of the wear traces are relevant to the wear rates of SiC whisker-reinforced PEEK, that is, the more severely abraded and ploughed the worn surface was corresponding to higher wear rate.

The wear traces formed on the steel ring surfaces by running the plain carbon steel ring against the pure PEEK and its composite blocks with different contents of SiC whisker are shown in Figure 4. It can be seen that a thick, lumpy, and incoherent transfer film was formed on the counterpart steel ring surface by running the steel ring against the pure PEEK block, while a thin, uniform, and coherent transfer film was formed by running the steel ring against the 1.25 wt % SiC whisker-reinforced PEEK composite block.

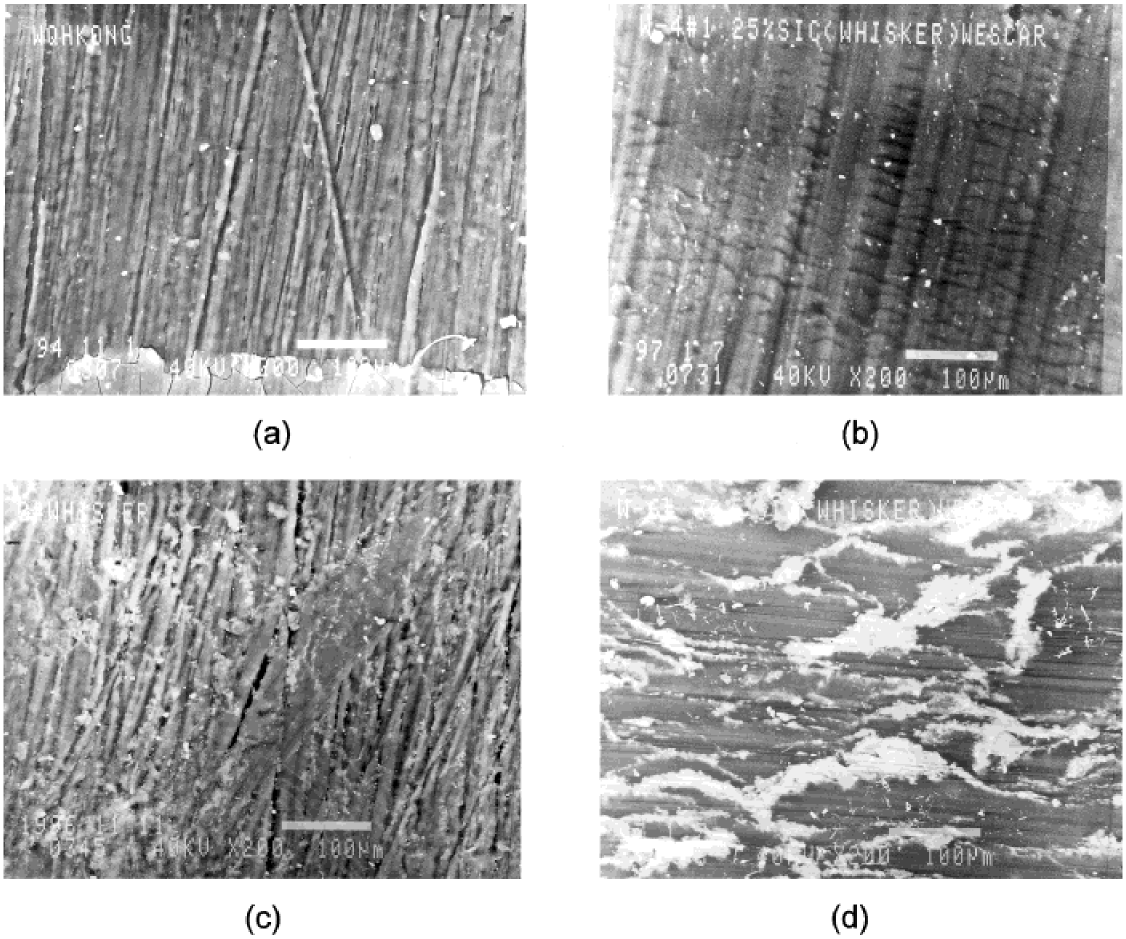

Figure 3. SEM micrographs of the worn surfaces of pure PEEK and the reinforced PEEK composites with various contents of SiC whisker (load: 196 *N*; sliding velocity: 0.445 m/s; test duration: 90 min). (a) pure PEEK; (b) 1.25 wt % SiC whisker/PEEK; (c) 10 wt % SiC whisker/PEEK; (d) 30 wt % SiC whisker/PEEK.

Figure 4(c) and (d) indicated that almost no transfer film was formed on the wear surfaces of the counterpart rings. In particular, with the increasing SiC whisker content, the more and more severe plucked and ploughed marks appeared on the wear surfaces of the counterpart steel rings. This indicated that for the higher SiC whisker content-reinforced PEEK, the counterpart steel ring was worn and ploughed more severely. It is necessary to emphasize again that there exists obvious difference among the morphologies of the wear traces on the pure PEEK and reinforced PEEK blocks (see Fig. 3). The obvious scuffing on the pure PEEK and those composite blocks with higher content of SiC whisker indicated the severe ploughing of the pure PEEK and its composites surfaces by the counterpart ring surface and the wear debris during the friction process. In contrast, only slight scuffing was observed on the surface of the 1.25 wt % SiC whisker-reinforced composite block. This also indicates that a uniform and tenacious transfer film was formed on the counterpart ring surface with the frictional couple of steel ring/1.25 wt % SiC whisker-filled PEEK block, being in agreement with what was seen in Figure 4.

Figure 5 gives the SEM micrographs of wear debris produced in wear tests of pure PEEK and its composite blocks with various contents of SiC whisker. It is found that the wear debris of the composites are much smaller than that of pure PEEK. Especially, the size of composite wear de-

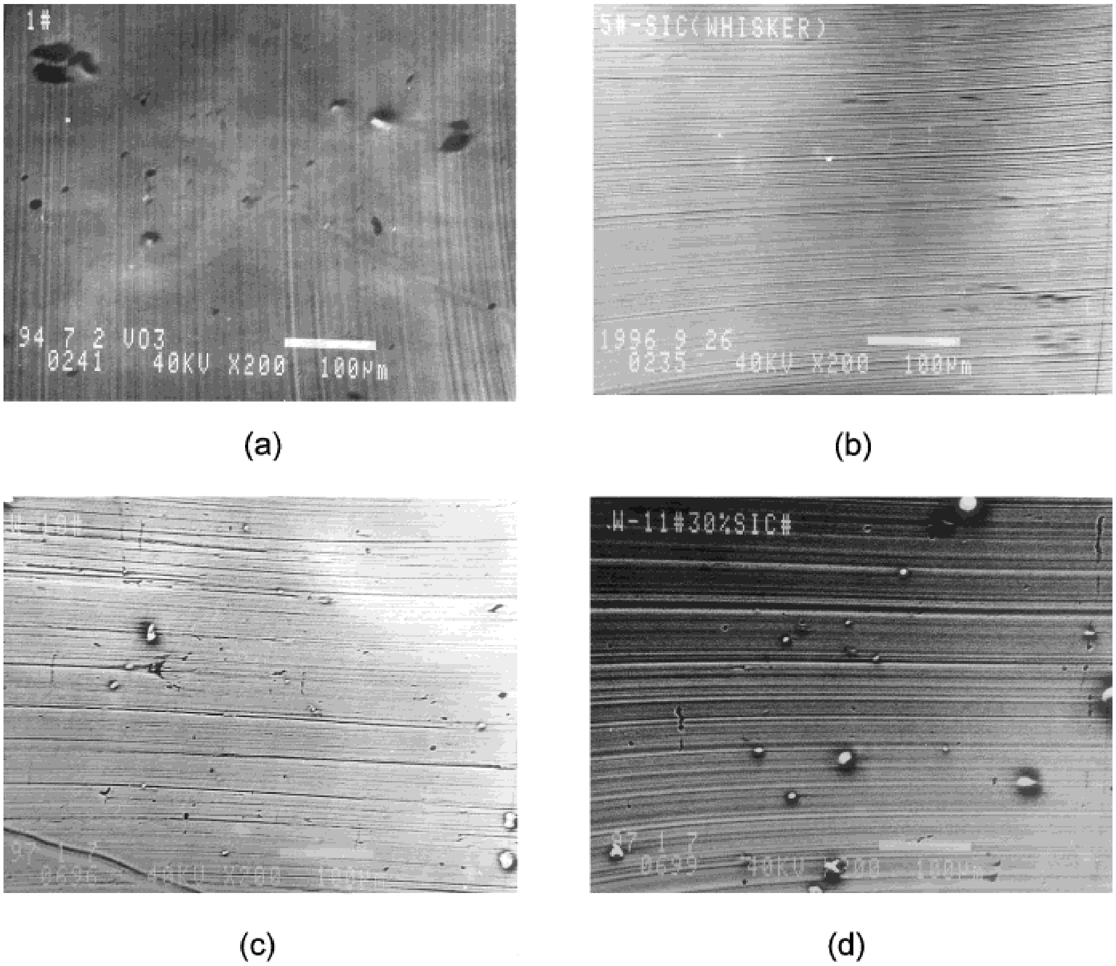

Figure 4. SEM micrographs of the wear traces of steel rings by running the steel ring against the pure PEEK block and the reinforced PEEK blocks with various contents of SiC whisker (load: 196 *N*; sliding velocity: 0.445m/s; test duration: 90 min) (a) pure PEEK; (b) 1.25 wt % SiC whisker/PEEK; (c) 10 wt % SiC whisker/PEEK; (d) 30 wt % SiC whisker PEEK/PEEK.

bris decreased sharply with an increase of SiC whisker content. EDAX analysis revealed that almost no Fe element existed in the pure PEEK wear debris. Conversely, for the wear debris of PEEK composites with various contents of SiC whisker, a lot of the elemental Fe was found in them. Especially, the content of elemental Fe existed in wear debris increased greatly with the increasing SiC whisker content. This indicates again that the higher content of the SiC whisker-reinforced PEEK could cause more severe wear of the counterpart carbon steel compared with pure PEEK and lower content of SiC whisker-reinforced PEEK.

DISCUSSION

The incorporation of SiC whisker in PEEK at an appropriate content caused a significant improvement in the tribological characteristics. The composite reinforced with 1.25 wt % SiC whisker exhibits considerably lower friction coefficient and minimized wear rate. It has been recognized that friction-induced fatigue tearing, local melting in the flash temperature zone, chemical degradation, and poor quality of the transfer film are responsible for the higher wear rate of pure PEEK.[9] Contrary to the above, a thin and uniform transfer film could be formed on the counterpart ring sur-

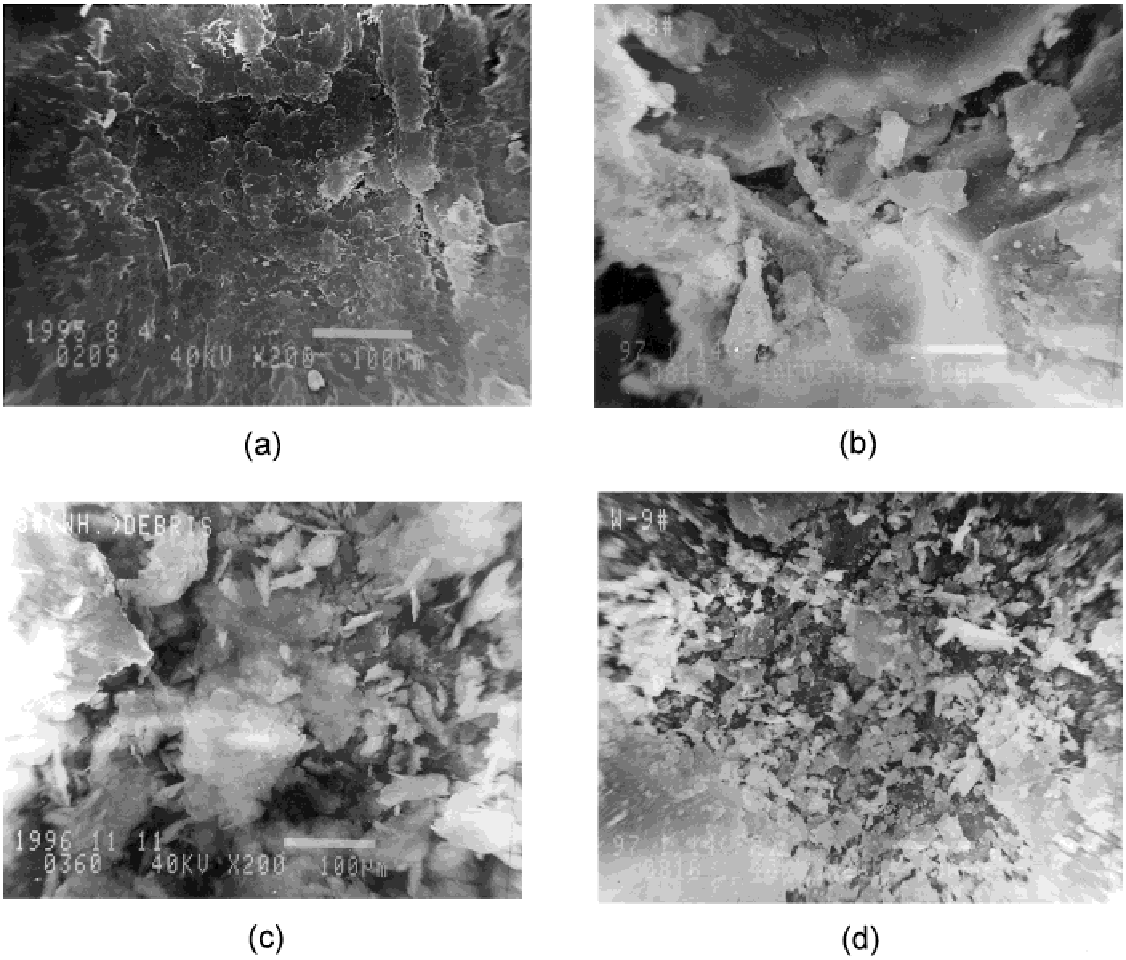

Figure 5. SEM micrographs of the wear debris of pure PEEK and the reinforced composites with various contents of SiC whisker (a) pure PEEK; (b) 1.25 wt % SiC whisker PEEK/PEEK; (c) 10 wt % SiC whisker/PEEK; (d) 30 wt % SiC whisker/PEEK.

face during the friction process of 1.25 wt % SiC whisker-reinforced PEEK/carbon steel ring system. With the formation of the uniform and tenacious transfer film, the subsequent sliding occurred between the surface of the 1.25 wt % SiC whisker reinforced PEEK composite block and the transfer film. Subsequently, a lowered wear rate was achieved. The corresponding wear mechanism is mild fatigue with microcracking. However, for the higher SiC whisker content-reinforced PEEK, the counterpart steel ring abraded by the composite block asperity transferred to the composite surface. This caused abrasive wear during further sliding. Because the abrasive wear became more severe with increasing SiC whisker content, a much higher wear rate was recorded for the composites containing higher content of SiC whisker filler.

CONCLUSIONS

The tribological properties of PEEK composites reinforced with various contents of the SiC whisker have been investigated under ambient dry sliding conditions by rotating a plain carbon steel ring against the composite block. It was found that SiC whisker as a reinforcing agent at an appropriate content greatly reduced the friction coefficient and wear rate of PEEK. The lowest wear rate of the composites was obtained at a SiC whisker content of 1.25 wt %. The corresponding dominant wear mechanism of the composite is mild fatigue wear with microcracking. On the other hand, at a higher content of the SiC whisker in the composites (above 10.0 wt %), severe abrasive wear caused much higher wear rate of the composites.

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