Tribological Characteristics of Nanometer Si_3N_4 Filled Poly(ether ether ketone) Under Distilled Water Lubrication

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ABSTRACT: Nanometer Si_3N_4 filled poly(ether ether ketone) (PEEK) composite blocks with different filler proportions were prepared by compression molding. Their friction and wear properties under distilled water lubrication, as well as under ambient dry conditions, were investigated on a block on ring machine by running a plain carbon steel (AISI 1045 steel) ring against the PEEK composite block. The worn surfaces of nanometer Si_3N_4 filled PEEK and the transfer film were observed by scanning electron microscopy (SEM) and electron probe microanalysis (EPMA). The results showed that distilled water could reduce the friction coefficient of nanometer Si_3N_4 filled PEEK but with the sacrifice of a large reduction in wear resistance. The SEM and EPMA pictures of the worn surfaces indicated that the wear mechanisms of nanometer Si_3N_4 filled PEEK under distilled water lubrication and ambient dry rubbing conditions were different. Under water lubrication, the dominant wear mechanism of the filled PEEK was severe abrasive wear with surface fracture. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 79: 1394–1400, 2001

Key words: nanometer Si_3N_4 filled poly(ether ether ketone) composite; water lubrication; tribological characteristics; morphologies; worn surface

INTRODUCTION

Poly(ether ether ketone) (PEEK) and its composites are widely used in dry friction conditions. Their friction and wear properties were investigated by many researchers.[1–4] But up to now, only a few results on the tribological characteristics of PEEK composites under water lubrication were reported, although sometimes they are used in water. It is well known that many plastics and their composites wear much more in water than in air.[5–8] Yamada and Tanaka investigated the wear of poly(tetrafluoroethylene) (PTFE) composites in water and pointed out that one of the causes of their high wear in water was the easier separation between the fillers and PTFE matrix in an aqueous environment.[9] Wang et al.[10] revealed that nanometer Si_3N_4 as a filler in PEEK was very effective in reducing the friction and wear of PEEK under ambient dry conditions.

The purpose of this work was to investigate the tribological properties of nanometer Si_3N_4 filled PEEK 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 was believed that this work would be helpful for understanding the wear mechanism of nanometer Si_3N_4 filled PEEK under distilled wa-

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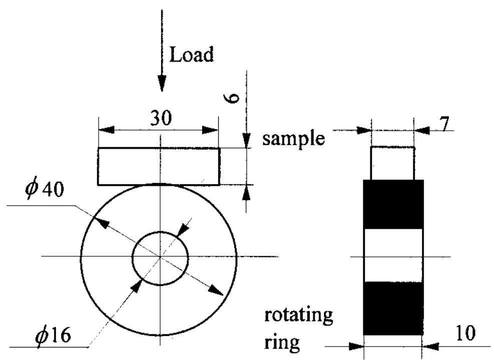


Figure 1 The contact schematic diagram for the frictional couple.

ter lubrication and for providing guidance to the tribological application of PEEK.

EXPERIMENTAL

PEEK fine powders with a diameter of 100 μm were produced by Jilin University of China. The nanometer Si_3N_4 was smaller than 50 nm and was produced by Shenyang Institute of Metal Research. The polymer powder was fully mixed with nanometer Si_3N_4 by being ultrasonically dispersed in alcohol for about 15 min. Then the mixture was dried at 110°C for 6 h to remove the alcohol and moisture. Finally, the mixture was molded into block specimens by compression molding in which the mixture was heated at a rate of 10°C/min 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 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. 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 ± 5%. Before each test, the plain carbon steel ring (HRC 48–50 hardness) and the PEEK or its composite block were abraded with number 900 water-abrasive paper. Then the steel ring with a surface roughness of 0.05 μm was cleaned with cotton dipped in acetone. The PEEK or its composite blocks with a surface roughness of 0.1 μm were cleaned with acetone and dried at 110°C for 2 h to remove the acetone and moisture and then cooled in a vacuum desiccator to 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 PEEK samples was measured by Archimedes principle using absolute alcohol as the immersing medium. The weight loss of the filled PEEK specimens was determined on a balance with a sensitivity of 0.05 mg and was then converted into the volume loss by using the filled PEEK density. The wear rate $\tilde{\omega}$ was calculated from the relationship

$$\omega = V/(XL)[\text{mm}^3/(\text{Nm})]$$

where V is the volume loss (mm^3), X is the sliding distance (m), and L is the applied load (N). Three replicate friction and wear tests were carried out to minimize data scattering, and the average of the three replicate test results is reported in this work. The deviation of the data of the replicate friction coefficient was ±5%. The morphologies of the worn surfaces were observed using scanning electron microscopy (SEM) and electron probe microanalysis (EPMA).

RESULTS AND DISCUSSION

Friction and Wear Properties of Nanometer Si_3N_4 Filled PEEK

Figure 2 shows the friction coefficient of nanometer Si_3N_4 filled PEEK as a function of the Si_3N_4

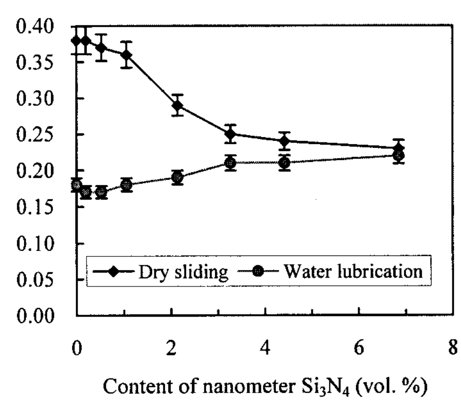


Figure 2 The friction coefficient of nanometer Si_3N_4 filled PEEK as a function of Si_3N_4 content under water lubrication and ambient dry conditions (196-N load, 0.445 m/s sliding velocity).

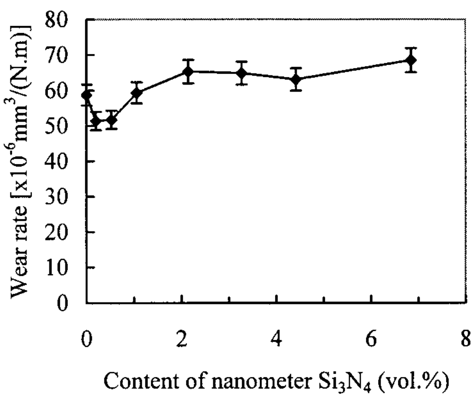


Figure 3 The wear rate of nanometer Si_3N_4 filled PEEK as a function of Si_3N_4 content under water lubrication (196-N load, 0.445 m/s sliding velocity).

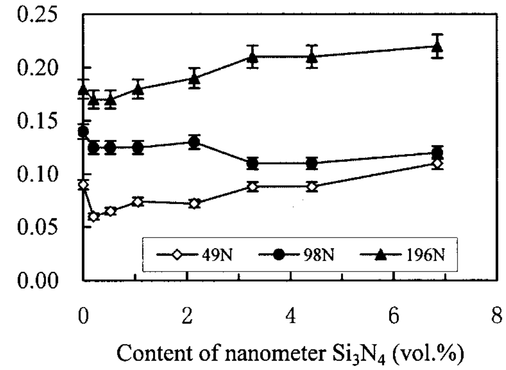


Figure 5 The effect of the load on the friction coefficient of PEEK composites filled with various contents of nanometer Si_3N_4 under water lubrication (0.445 m/s sliding velocity).

content under water lubrication and ambient dry sliding conditions. It is seen that under ambient dry conditions nanometer Si_3N_4 reduced the friction coefficient effectively. The friction coefficient decreased gradually with increasing Si_3N_4 content and finally reached a minimum value as the Si_3N_4 content reached 6.8 vol %. In contrast, under water lubrication the friction coefficient of nanometer Si_3N_4 filled PEEK increased continuously with increasing nanometer Si_3N_4 content. The friction coefficient was higher than that of unfilled PEEK when the Si_3N_4 content was above 1.1 vol %, although it was still considerably lower in comparison with the filled PEEK under ambient dry conditions. This indicated that under water lubrication nanometer Si_3N_4 as a filler in PEEK could not reduce the friction coefficient of the filled PEEK.

Figures 3 and 4 show the wear rate of nanometer Si_3N_4 filled PEEK under water lubrication

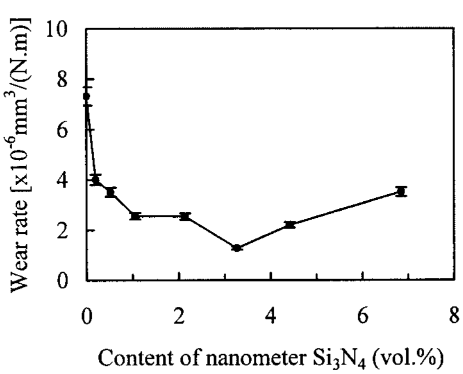


Figure 4 The wear rate of nanometer Si_3N_4 filled PEEK as a function of Si_3N_4 content under ambient dry sliding (196-N load, 0.445 m/s sliding velocity).

and ambient dry conditions as a function of the nanometer Si_3N_4 content. It can be seen that under ambient dry conditions nanometer Si_3N_4 filled PEEK exhibited a considerably decreased wear rate than the unfilled PEEK. The wear rate of the composite was affected by the filling of the nanometer Si_3N_4 in a complicated fashion. When the content of nanometer Si_3N_4 was below 3.3 vol %, the wear rate was continuously decreased with increasing Si_3N_4 content. The lowest wear rate was obtained with 3.3 vol % nanometer Si_3N_4. When the nanometer Si_3N_4 content was above 3.3 vol %, the wear rate increased with increasing Si_3N_4 content, although it remained lower than that of the unfilled PEEK. Meanwhile, it can be seen that the unfilled PEEK and filled PEEK composites both showed much higher wear rates under water lubrication than under dry sliding. Besides, PEEK composites with filler volume fractions of 0.2 and 0.5 vol % gave the lowest wear rate under water lubrication; those with higher filler volume fractions showed slightly increased wear rates. Under water lubrication the wear rate of nanometer Si_3N_4 filled PEEK was higher in comparison with the unfilled PEEK when the content of nanometer Si_3N_4 was above 1.1 vol %. This indicated that under water lubrication the nanometer Si_3N_4 as a filler in PEEK could not improve the wear resistance of the filled PEEK.

Figures 5 and 6 show the effect of load on the coefficient of friction and wear rate of PEEK composites filled with various contents of Si_3N_4 under water lubrication. Note that the friction coefficient decreased with decreasing load. The lowest friction coefficient in this work was obtained under a load of 49 N. Obviously, in water the wear

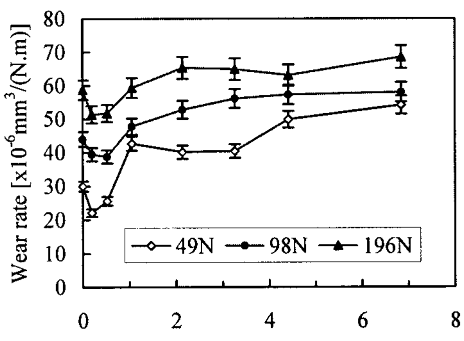


Figure 6 The effect of the load on the wear rate of PEEK composites filled with various contents of Si_3N_4 under water lubrication (0.445 m/s sliding velocity).

rate of the filled PEEK under various loads increased with increasing Si_3N_4 content, while the wear rate was higher in comparison with the unfilled PEEK when the content of Si_3N_4 was above 1.1 vol %. The lowest wear rate was also obtained under a load of 49 N. Thus, it can be concluded that nanometer Si_3N_4 as a filler in PEEK under water lubrication does not cause improvement in the tribological characteristics of PEEK.

SEM and EPMA Observation of Worn Surfaces

To understand the effect of nanometer Si_3N_4 on the friction and wear behaviour of the filled PEEK under water lubrication, the worn surfaces were studied by SEM and EPMA. Figure 7 shows SE micrographs of the worn surfaces for the frictional couple of a carbon steel ring and a 4.4 vol % nanometer Si_3N_4 filled PEEK block under water lubrication. It can be seen that severely ploughed and plucked marks appeared on the block surface, while the surface of the counterpart ring was very rough and a discontinuous thick transfer film was seen. Figure 7(a,e,g) proves that surface fracture took place on the block and that fracture debris transferred to the steel ring surface.

Figure 8 gives the SEM and EPMA pictures of the worn surfaces under ambient dry conditions for the frictional couple of a carbon steel ring and a 4.4 vol % nanometer Si_3N_4 filled PEEK block. The scuffing on the block surface was found to be considerably abated, while the surface of the counterpart steel ring appeared to be smoother. The micrographs showed that a uniform and tenacious transfer film was formed on the counterpart steel ring surface during the friction process. Thus, it can also be inferred that the morphologies of the worn surfaces are relevant to the wear rates of nanometer Si_3N_4 filled PEEK under water lubrication and ambient dry sliding conditions.

DISCUSSION

The incorporation of nanometer Si_3N_4 into PEEK under ambient dry conditions caused a significant improvement in the tribological characteristics. However, under distilled water lubrication the friction coefficient of the filled PEEK could be decreased but with the sacrifice of a large increase in wear rate. For nanometer Si_3N_4 filled PEEK under dry friction a uniform and tenacious transfer film was formed on the counterpart ring surface during the friction process. With the formation of the uniform and tenacious transfer film, subsequent sliding occurred between the surface of the nanometer Si_3N_4 filled PEEK composite block and the transfer film. Thus, a considerably lower wear rate was obtained. The corresponding wear mechanism was slight fatigue and transfer. In contrast, under water lubrication it was seen that obvious scuffing was observed on the block surface, the surface of the counterpart ring seemed to be rough, and a discontinuous thick transfer film was seen. This indicated the inability of this material to form its transfer film on the steel ring surface under water lubrication. The transfer film in this case was unable to provide protection to the filled PEEK from the carbon steel ring surface. The surface of the filled PEEK was ploughed severely by the rough counterpart steel ring. Severe surface fracture took place on the filled PEEK block. All of these caused a fairly high wear rate of the filled PEEK under distilled water lubrication.

CONCLUSIONS

From above, the following conclusions can be drawn:

1. Under ambient dry conditions, nanometer Si_3N_4 filled PEEK exhibited a considerably lower friction coefficient and wear rate than unfilled PEEK. A uniform and tenacious transfer film was formed on the counterpart steel ring surface during the friction process.
2. Distilled water lubrication could reduce

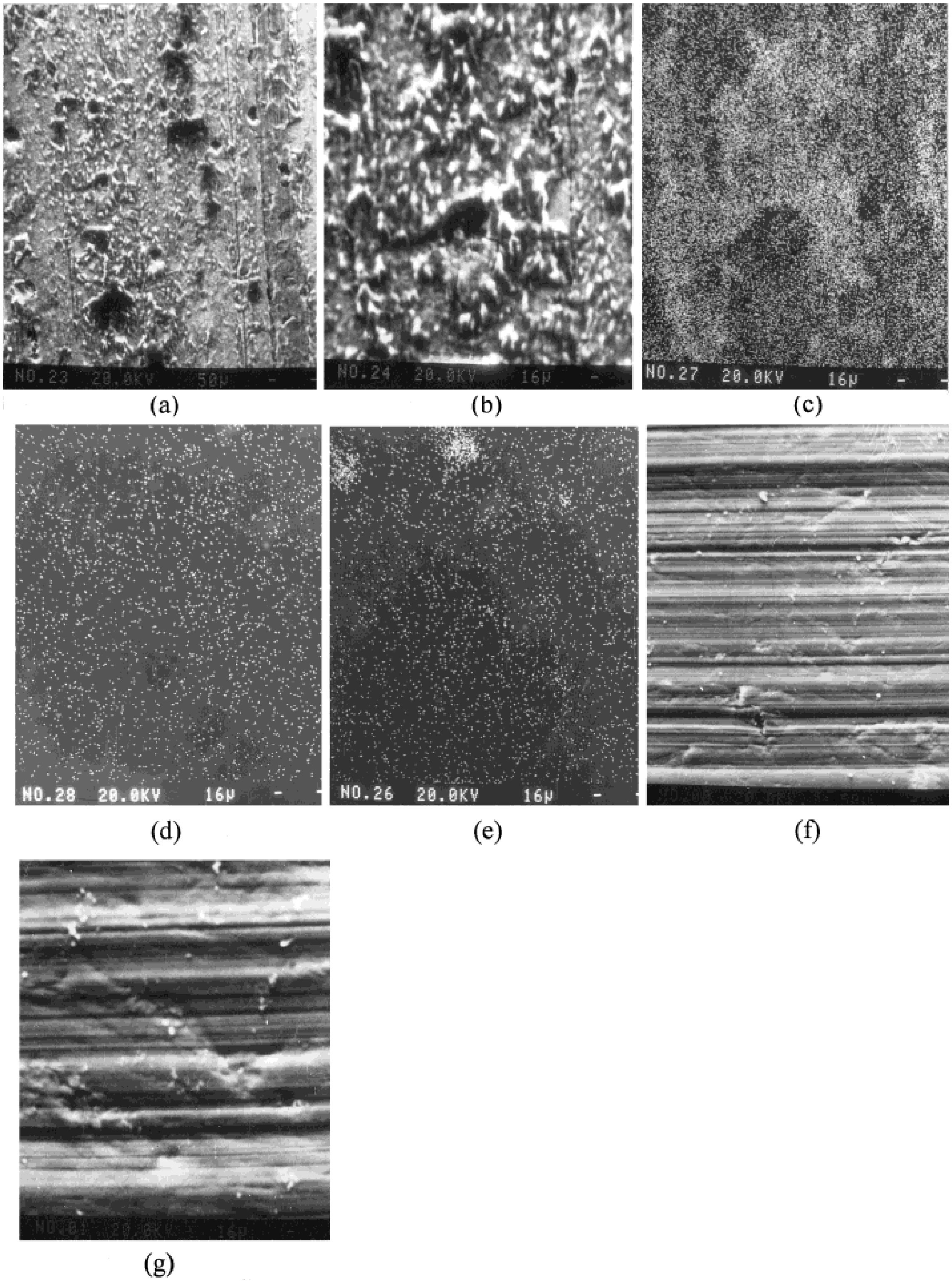
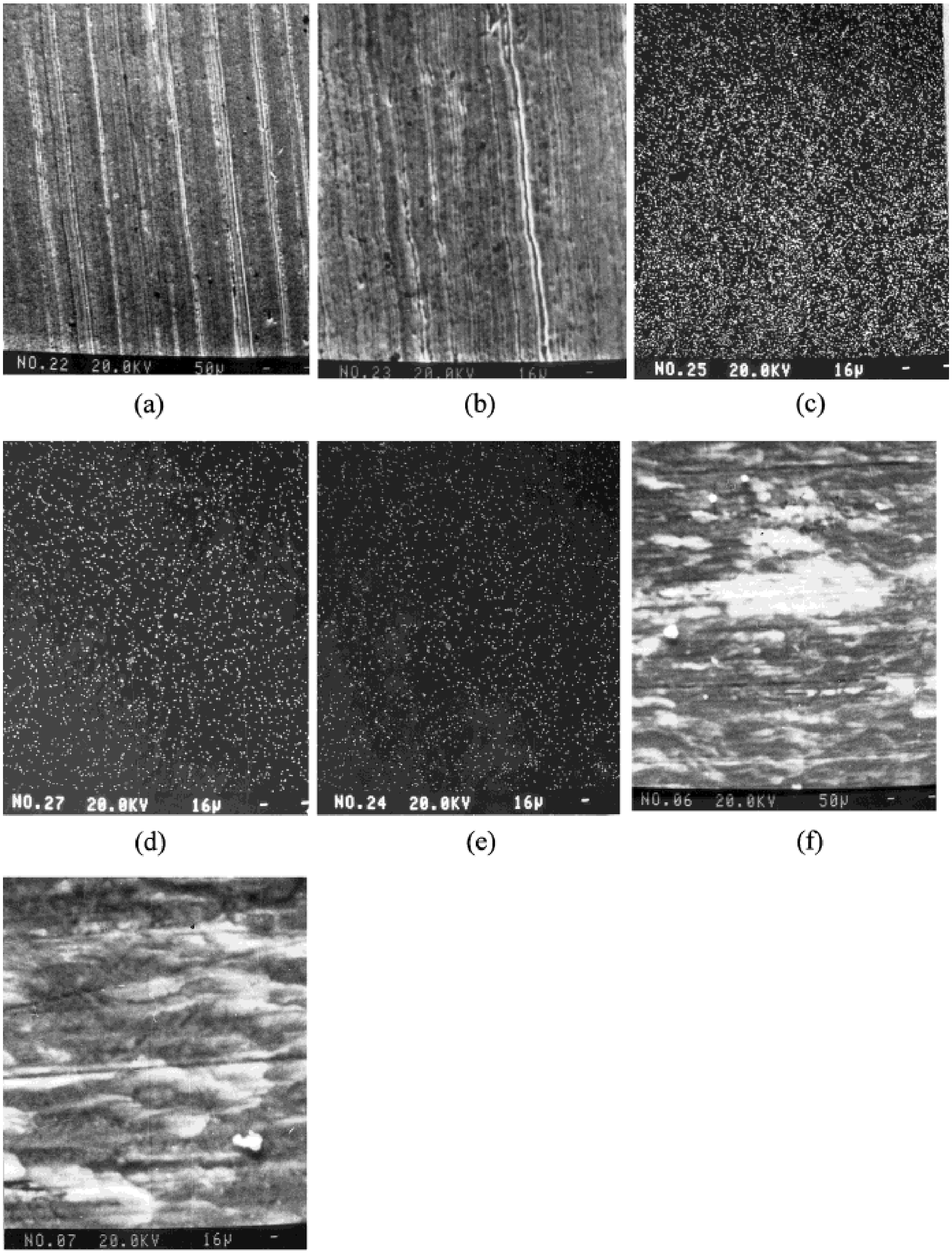


Figure 7 SEM pictures of the worn surfaces for the frictional couple of the carbon steel ring and the 4.4 vol % nanometer Si_3N_4 filled PEEK under water lubrication (196-N load, 0.445 m/s sliding velocity, 90-min test duration): (a) the worn surface of the steel ring, (b) a magnified picture of the steel ring, (c) the distribution of Si in the steel ring, (d) the distribution of N in the steel ring, (e) the distribution of C in the steel ring, (f) the worn surface of the filled PEEK, and (g) a magnified picture of the filled PEEK.



(g)

Figure 8 SEM pictures of the worn surfaces for the frictional couple of the carbon steel ringand the 4.4 vol % nanometer Si_3N_4 filled PEEK block under ambient dry conditions (196-N load, 0.445 m/s sliding velocity, 90-min test duration): (a) the worn surface of the counterpart steel ring, (b) a magnified picture of the steel ring, (c) the distribution of Si in the steel ring, (d) the distribution of N in the steel ring, (e) the distribution of C in the steel ring, (f) the worn surface of the filled PEEK, and (g) a magnified picture of the filled PEEK.

the friction coefficient of nanometer Si_3N_4 filled PEEK but with the sacrifice of a large reduction in wear resistance.

3. Under water lubrication the worn surfaces of nanometer Si_3N_4 filled PEEK were very rough and a discontinuous thick transfer film was formed on the surface of the counterpart steel ring. The major wear mechanism of the filled PEEK was severe abrasive wear.

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