Friction and Wear Characteristics of Metal Sulfides and Graphite-Filled PTFE Composites Under Dry and Oil-Lubricated Conditions

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Received 4 May 1998; accepted 17 June 1998

ABSTRACT: Five kinds of polytetrafluoroethylene (PTFE)-based composites, pure PTFE, PTFE + 30(v)% MoS_2, PTFE + 30(v)% PbS, PTFE + 30(v)% CuS, and PTFE + 30(v)% graphite (GR) composites, were first prepared. Then the friction and wear properties of these PTFE composites, sliding against GCr15-bearing steel under both dry and liquid paraffin-lubricated conditions, were studied by using an MHK-500 ring-on-block wear tester. Finally, the worn surfaces and the transfer films of the PTFE composites formed on the surface of GCr15 bearing steel were investigated by using a scanning electron microscope (SEM) and an optical microscope, respectively. Experimental results show that filling with MoS_2, PbS, CuS, or graphite to PTFE can reduce the wear of the PTFE composites by two orders of magnitude compared to that of pure PTFE under dry friction conditions. However, the friction and wear-reducing properties of these PTFE composites can be greatly improved by lubrication with liquid paraffin. Investigations of transfer films show that MoS_2, PbS, CuS, and graphite promote the transfer of the PTFE composites onto the surface of GCr15-bearing steel under dry friction conditions, but the transfer of the PTFE composites onto the surface of GCr15-bearing steel can be greatly reduced by lubrication with liquid paraffin. SEM examinations of worn surfaces show that with lubrication of liquid paraffin, the creation and development of the cracks occurred on the worn surfaces of the PTFE composites under load, which reduces the load-supporting capacity of the PTFE composites. This would lead to the deterioration of the friction and wear properties of the PTFE composites under higher loads ($>600N$). © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 72: 751–761, 1999

Key words: metal sulfides and graphite; PTFE composites; oil lubrication; friction and wear; frictional surfaces

INTRODUCTION

It is well known that a low friction coefficient, high thermal stability, and chemical resistance combine to make polytetrafluoroethylene (PTFE) a particularly attractive polymer for sliding applications, but its poor mechanical strength, excessive viscoelastic deformation under load, and a high rate of wear usually limit greatly the use of PTFE in practice. It has been found that almost any type of inorganic fillers can greatly reduce the wear of PTFE.[1] Graphite and metal sulfides MoS_2, PbS, and CuS are important inorganic fillers for polymers, and their effects on the friction and wear behaviors of PTFE-based composites

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Journal of Applied Polymer Science, Vol. 72, 751–761 (1999)

CCC 0021-8995/99/060751-11

Table I Chemical Compositions of GCr15-Bearing Steel (Wt %)

C	Mn	Si	Cr	P	S	Fe
0.950–1.050	0.200–0.400	0.150–0.350	1.300–1.650	<0.027	<0.020	Remainder

under dry friction conditions have been studied by Gong et al.[2–4]

However, with enlargement of application fields of PTFE-based composites in practice, more and more PTFE-based composites have been used in fluid environments. It has been found that the wear of many polymers in water is much higher than that in air, and the wear of the PTFE composites filled with only glass fibers is much greater than that of other PTFE composites in water.[5–7] However, until now, much less information has been available on the friction and wear behavior of metal sulfides or graphite-filled PTFE composites under oil-lubricated conditions. Therefore, it is essential to study the friction and wear behavior of metal sulfides or graphite-filled PTFE composites under oil-lubricated conditions.

The aim of this work is to study the friction and wear behavior of the PTFE composites filled with metal sulfides or graphite under both dry and oil-lubricated conditions and to give some insight into the friction and wear mechanisms of the PTFE composites under oil-lubricated conditions. It is expected that this study may be helpful to the application of the PTFE composites under oil-lubricated conditions in practice.

EXPERIMENTAL

Materials used for preparing PTFE composites include PTFE powder with a grit size of about 30 μm, MoS_2, PbS, CuS, and graphite (GR) powders about of 76 μm. First, the GR and the metal sulfides MoS_2, PbS, and CuS powders were mixed completely with the PTFE powder, respectively. Secondly, these mixtures were molded into the blocks by compression molding under the pressure of 50 MPa. Finally, these PTFE composite blocks were sintered at 380°C for 3 h in air and then cooled freely to the room temperature. Five kinds of the PTFE-based composites, pure PTFE, PTFE + 30(v)% MoS_2, PTFE + 30(v)% PbS, PTFE + 30(v)% CuS, and PTFE + 30(v)% GR composite, were prepared in this work.

The friction and wear tests were carried out on an MHK-500 ring-block wear tester (Timken Wear Tester) with a steel ring, which is 49.2 mm in diameter and 13.0 mm in length, rotating on a PTFE composite block, which is 12.3 $\times$ 12.3 $\times$ 18.9 mm in size. The surfaces of the PTFE composite blocks were polished with number-800-grade SiC abrasive paper to a surface roughness of R_a = 0.2–0.4 μm. Meanwhile, the steel ring, made of GCr15-bearing steel (its chemical composition is listed in Table I), was polished with number-900-grade SiC abrasive paper to a surface roughness of R_a = 0.15 μm.

The friction and wear tests were performed at room temperature with a sliding speed from 1.0 to 2.5 m s and loads from 100 to 400N for the dry friction conditions or 200 to 1000N for the oil-lubricated conditions. The lubricating oil used in the experiments was liquid paraffin (its typical characteristics are listed in Table II), which was added to the rubbing surfaces at a rate of 30 drops per min during the tests. Each friction and wear test was performed for 30 min. Before each test started, the surfaces of the PTFE composite block and the GCr15-bearing steel ring were cleaned by rubbing with a soft cloth dipped in acetone and then dried in air. In this work, three to five samples were tested at each condition; the friction coefficient and wear were the average values of

Table II Typical Characteristics of Liquid Paraffin

Viscosity ($\times 10^{-6}$ m^2 s^{-1})					
40°C	100°C	Viscosity Index	Flash Point (°C)	Boiling Point (°C)	Main Composition
21.49	4.42	117	226	>300	Paraffin

Table III Friction and Wear Results of the PTFE Composites Filled with Metal Sulfides or Graphite Sliding Against GCr15-Bearing Steel Under the Dry Friction Condition

Material	Friction Coefficient	Wear (mg)
PTFE	0.257	385.4
PTFE + 30(v)% GR	0.245	5.8
PTFE + 30(v)% MoS_2	0.284	8.8
PTFE + 30(v)% PbS	0.395	3.3
PTFE + 30(v)% CuS	0.454	4.4

Conditions are as follows: Sliding speed: 1.5 m/s; load, 100*N;* time, 30 min.

these tests for each condition. The relative error of the data is about 10%. The wear was detected by the weight loss of the PTFE composite blocks after each test to an accuracy of 0.1 mg. The friction coefficient was determined by measuring the friction torque, while the friction torque was detected by a torque measuring system. The friction coefficient was the average value of those in the steady stage of friction (the last 10 min) for each test. Finally, the worn surfaces of the PTFE composites and the transfer films formed on the surface of GCr15-bearing steel ring were investigated by using an SEM and an optical microscope, respectively.

RESULTS AND DISCUSSION

Friction and Wear Properties Under Dry Friction Conditions

The friction and wear results of metal sulfides and graphite-filled PTFE composites sliding against GCr15-bearing steel under dry friction conditions are listed in Table III. The results in Table III show that GR reduces the friction coefficient of the PTFE composite, but metal sulfides MoS_2, PbS, and CuS increase the friction coefficients of the PTFE composites. As for MoS_2-, PbS-, or CuS-filled PTFE composites, the friction coefficient of the PTFE + 30(v)% MoS_2 composite is the lowest, and that of the PTFE + 30(v)% CuS composite is the highest. Meanwhile, the results in Table III show that the antiwear properties of the PTFE composites can be greatly improved by filling PTFE with MoS_2, PbS, CuS, or graphite, and the wear of the PTFE composites can be decreased by two orders of magnitude compared to that of pure PTFE. However, the wear-reducing action of PbS is the most effective, that of CuS is the second, then the graphite, and that of MoS_2 is the worst.

Variations of friction coefficients and wear with load for metal sulfides or graphite-filled PTFE composites sliding against GCr15-bearing steel under dry friction conditions are shown in Figures 1 and 2, respectively. It can be seen from Figure 1 that the friction coefficients of metal

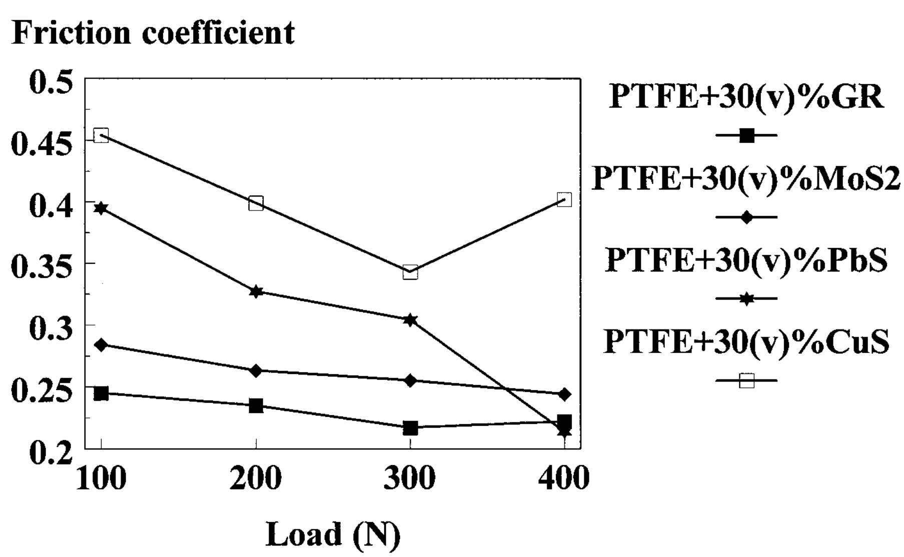

Figure 1 Variations of friction coefficients with load for metal sulfides or graphite-filled PTFE composites sliding against GCr15-bearing steel under dry friction conditions (sliding speed, 1.5 m/s).

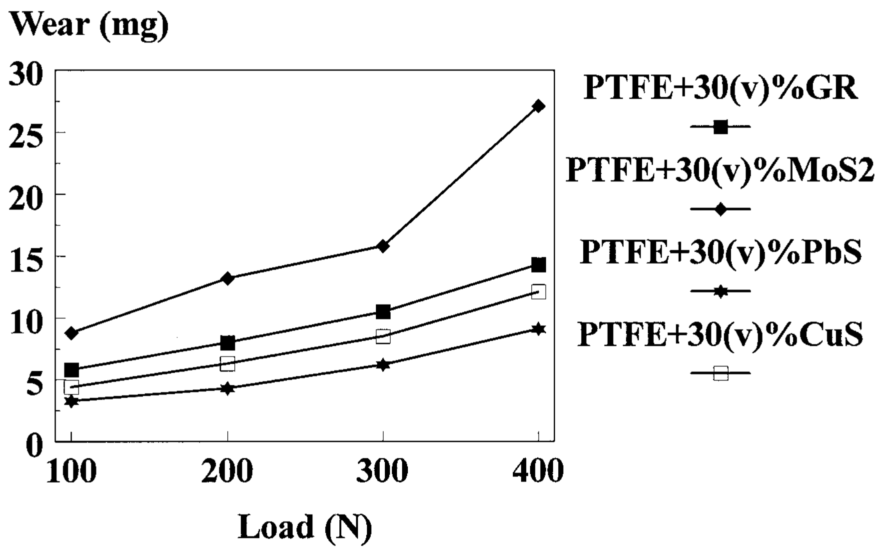

Figure 2 Variation of wear with load for metal sulfides or graphite-filled PTFE composites sliding against GCr15-bearing steel under dry friction conditions (sliding speed, 1.5 m/s).

sulfides or graphite-filled PTFE composites decrease with the increase of load under dry friction conditions. Under different loads in dry friction conditions, the friction property of the PTFE + 30(v)% GR composite is the best, that of the PTFE + 30(v)% MoS_2 composite is the second, then the PTFE + 30(v)% PbS composite and that of the PTFE + 30(v)% CuS composite is the worst. The results in Figure 2 show that the wear of metal sulfides or graphite-filled PTFE composites increases with the increase of load. Under different loads in dry friction conditions, the wear-reducing property of the PTFE + 30(v)% PbS composite is the best, that of the PTFE + 30(v)% CuS composite is the second, then the PTFE + 30(v)% GR composite and that of the PTFE + 30(v)% MoS_2 composite is the worst.

Friction and Wear Properties Under Oil Lubricated Conditions

The variations of friction coefficients and wear with load for metal sulfides or graphite-filled PTFE composites sliding against GCr15-bearing steel under lubrication with liquid paraffin are shown in Figures 3 and 4, respectively. Comparison of the friction results in Figure 3 to those in Figure 1 shows that the friction properties of metal sulfides or graphite-filled PTFE composites can be greatly improved by lubrication with liquid paraffin, and the friction coefficients of the PTFE composites can be decreased by one order of magnitude. Under lubrication of liquid paraffin, the friction coefficients of the PTFE composites first decrease with the increase of load and then increase with the increase of load when the load is higher than 600N. But the friction property of pure PTFE is better than those of metal sulfides or graphite-filled PTFE composites under oil lubricated conditions. Comparison of the wear results in Figure 4 to those in Figure 2 shows that the wear-reducing properties of metal sulfides or graphite-filled PTFE composites can be greatly improved by lubrication with liquid paraffin, and the wear of the PTFE composites increases with the increase of load. However, when the load is higher than 600N, the wear-reducing properties of the PTFE + 30(v)% PbS and PTFE + 30(v)% CuS composites are better than those of the other PTFE composites under lubrication of liquid paraffin.

The variations of friction coefficient and wear rate with sliding speed for the PTFE + 30(v)% PbS composite sliding against GCr15-bearing steel under lubrication of liquid paraffin are shown in Figures 5 and 6, respectively. The results in Figures 5 and 6 show that the friction coefficient and the wear rate of the PTFE + 30(v)% PbS composite first decrease with the increase of sliding speed and then increase as the sliding speed increases. At the sliding speed of 2.0 m s in liquid paraffin lubrication, the friction

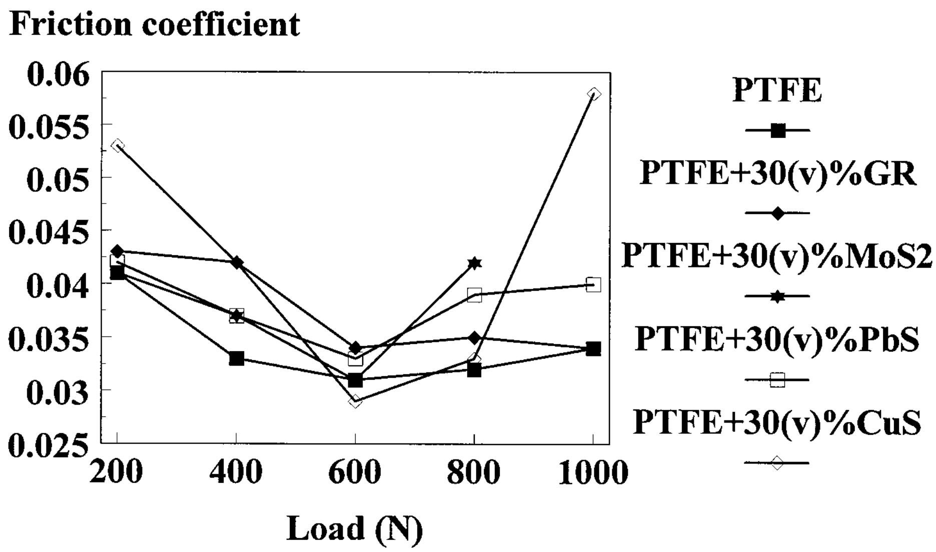

Figure 3 Variations of friction coefficients with load for metal sulfides or graphite-filled PTFE composites sliding against GCr15-bearing steel under lubrication of liquid paraffin (sliding speed, 2.5 m/s).

and wear-reducing properties of the PTFE composite are the best. It is believed that, with the increase of sliding speed in liquid paraffin lubrication, a layer of lubricating oil film can be formed more easily on the frictional surfaces, then the lubrication condition at the rubbing surfaces can be greatly improved. Therefore, the friction coefficient and wear rate of the PTFE composite decrease with the increase of sliding speed. However, with a further increase of sliding speed, the temperature rise at rubbing surfaces results in the reduction of mechanical strength and load carrying capacity of the PTFE composite; this can lead to the increase of friction and wear of the PTFE composite.

When the sliding speed is constant, the variations of friction coefficients with load for the PTFE composites filled with metal sulfides or

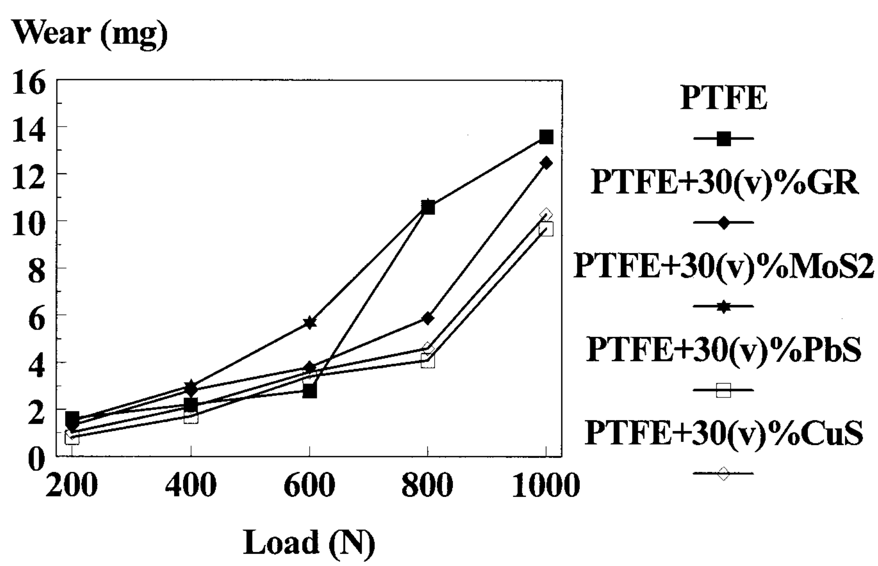

Figure 4 Variation of wear with load for metal sulfides or graphite-filled PTFE composites sliding against GCr15-bearing steel under lubrication of liquid paraffin (sliding speed, 2.5 m/s).

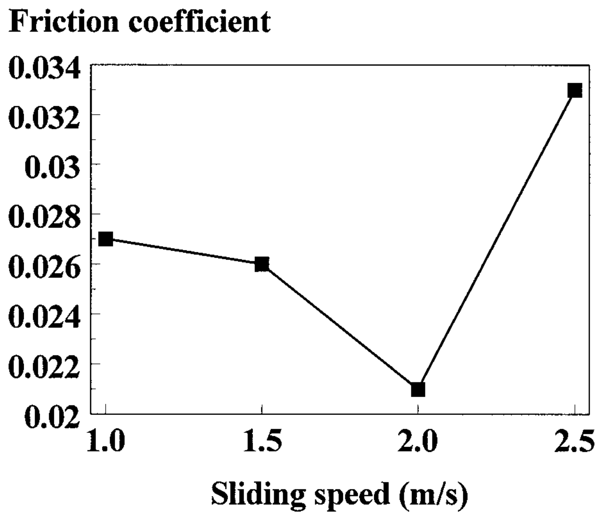

Figure 5 Variation of friction coefficient with sliding speed for the PTFE + 30(v)% PbS composite sliding against GCr15-bearing steel under lubrication of liquid paraffin (load, 600N).

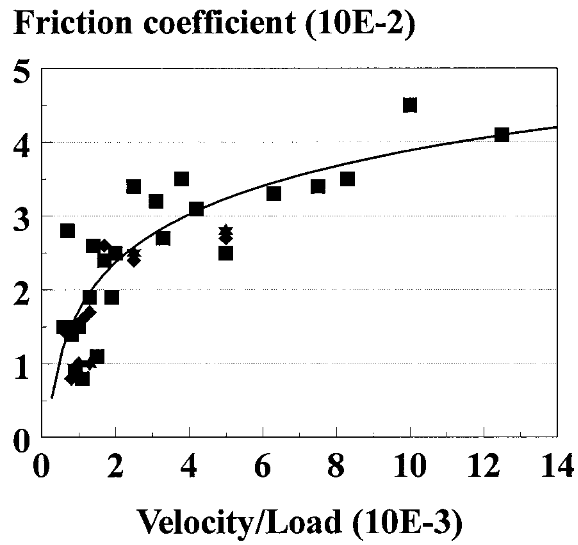

Figure 7 Variation of friction coefficient as a function of velocity/load for pure PTFE sliding against GCr15-bearing steel under the lubrication of liquid paraffin.

graphite in liquid paraffin lubrication can be explained by the Stribeck's curves of friction coefficients against the Sommerfeld variable $\eta N/P$, where η is the viscosity of liquid paraffin, N is the rotation speed of the GCr15-bearing steel ring, and P is the load applied.[8,9] At a constant sliding speed, the temperature at frictional surfaces increases with the increase of load, while the viscosity of liquid paraffin decreases with the increase of temperature but increases with the increase of load. Therefore, under the given conditions in this work, the effect of viscosity on the Sommerfeld variable $\eta N/P$ is so small, compared to the effect of load on it, that the $\eta N/P$ can be approximated to N/P. Figure 7 gives the variation of friction coefficient as a function of velocity/load (N/P) for pure PTFE sliding against GCr15-bearing steel under lubrication of liquid paraffin. It can be seen from Figure 7 that the friction coefficient of PTFE decreases with the increase of load at a constant sliding speed. Therefore, it can be deduced that the variations of friction coefficients with load for metal sulfides or graphite-filled PTFE composites under lower loads (<600N) in liquid paraffin lubrication can also be explained by Stribeck's curve, as given in Figure 7.

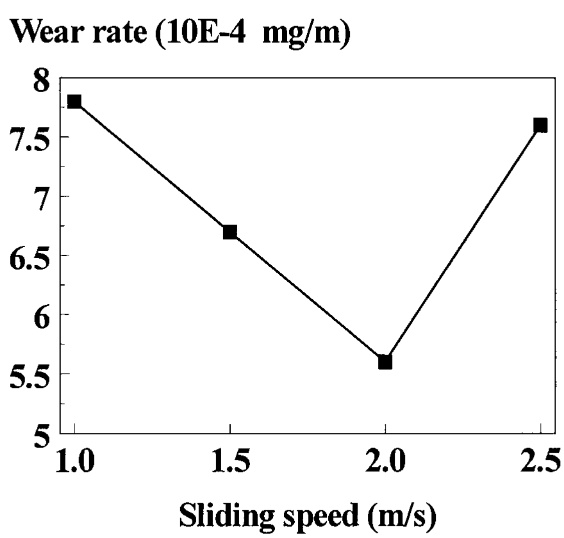

Figure 6 Variation of wear rate with sliding speed for PTFE + 30(v)% PbS composite sliding against GCr15-bearing steel under lubrication of liquid paraffin (load, 600N).

SEM Examination of Worn Surfaces

It was found in the experiments that when the load increased to a certain value under lubrication of liquid paraffin, some obvious cracks or serious deformation occurred on the worn surfaces of the PTFE composites. At this time, the wear of the PTFE composites increases sharply. These loads, under which obvious cracks or serious deformation occurred on the worn surfaces of the PTFE composites, are considered as the limit loads of the PTFE composites under the given

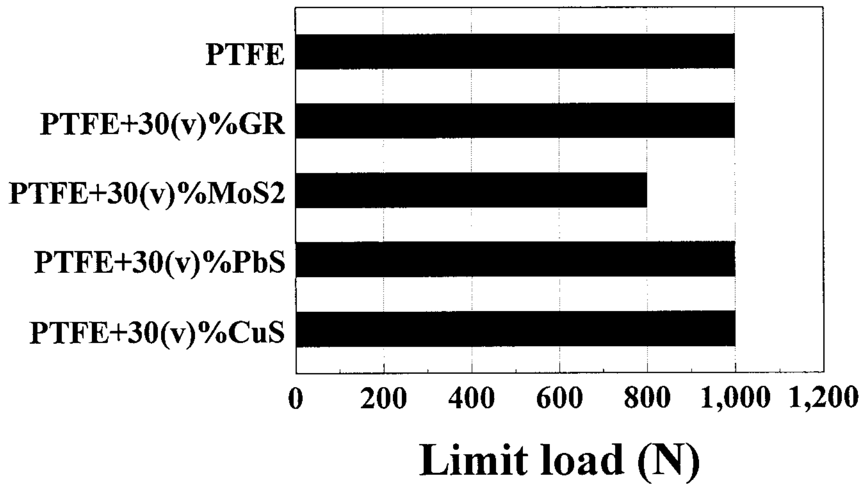

Figure 8 Limit loads of metal sulfides or graphite-filled PTFE composites sliding against GCr15-bearing steel under lubrication of liquid paraffin (sliding speed, 2.5 m/s).

conditions (as shown in Figure 8). Figure 9 gives electron micrographs of the worn surfaces of the PTFE composites filled with metal sulfides or graphite sliding against GCr15-bearing steel under lubrication of liquid paraffin. It can be seen from Figure 9 that there are some obvious wear scars on the worn surface of pure PTFE in liquid paraffin lubrication, but there are some obvious cracks on the worn surfaces of the PTFE composites filled with MoS_2, PbS, CuS, or graphite under the given conditions.

It is known that filling MoS_2, PbS, CuS, or graphite to PTFE can produce some microdefects in the PTFE composites. Therefore, it is believed that the interaction between liquid paraffin and the PTFE composites, especially the absorption of liquid paraffin into the microdefects of the PTFE composites under load, can create some cracks on the worn surfaces of the PTFE composites. The creation and the development of these cracks under load reduce the mechanical strength and load-supporting capacity of the PTFE composites.[10,11] This would lead to the deterioration of the friction and wear properties of the PTFE composites under higher loads ($>600N$) in liquid paraffin lubrication. The above analysis results are consistent with the results of friction and wear tests.

Optical Microscope Investigation of Transfer Films

Optical micrographs of the transfer films formed on the surface of GCr15-bearing steel for the PTFE composites filled with metal sulfides or graphite under both dry and liquid paraffin lubricated conditions are shown in Figures 10 and 11, respectively. The results in Figure 10 show that there are obvious transfer films formed on the surface of GCr15-bearing steel for the PTFE composites filled with metal sulfides or graphite in dry friction conditions, but no obvious transfer films formed on the surface of GCr15-bearing steel for pure PTFE. Correlating the above investigations with the results of friction and wear tests in dry friction conditions, it can be deduced that metal sulfides MoS_2, PbS, and CuS, as well as graphite, enhance the adhesion of the transfer films to the surface of GCr15-bearing steel and promote the transfer of the PTFE composites onto the surface of GCr15-bearing steel, so they greatly reduce the wear of the PTFE composites.[4,12] Meanwhile, it can also be seen from Figure 10 that, as for MoS_2-, PbS-, or CuS-filled PTFE composites under dry friction conditions, the transfer film formed on the surface of GCr15-bearing steel for MoS_2-filled PTFE composite is more uniform than those of PbS- or CuS-filled PTFE composites. This indicates that MoS_2-filled PTFE composite can easily form a uniform transfer film on the surface of GCr15-bearing steel so that then the friction between the PTFE composite and GCr15-bearing steel can be transformed to friction between the PTFE composite and the transfer films formed on the counterface. Therefore, the friction property of MoS_2-filled PTFE composite is better than those of PbS- or CuS-

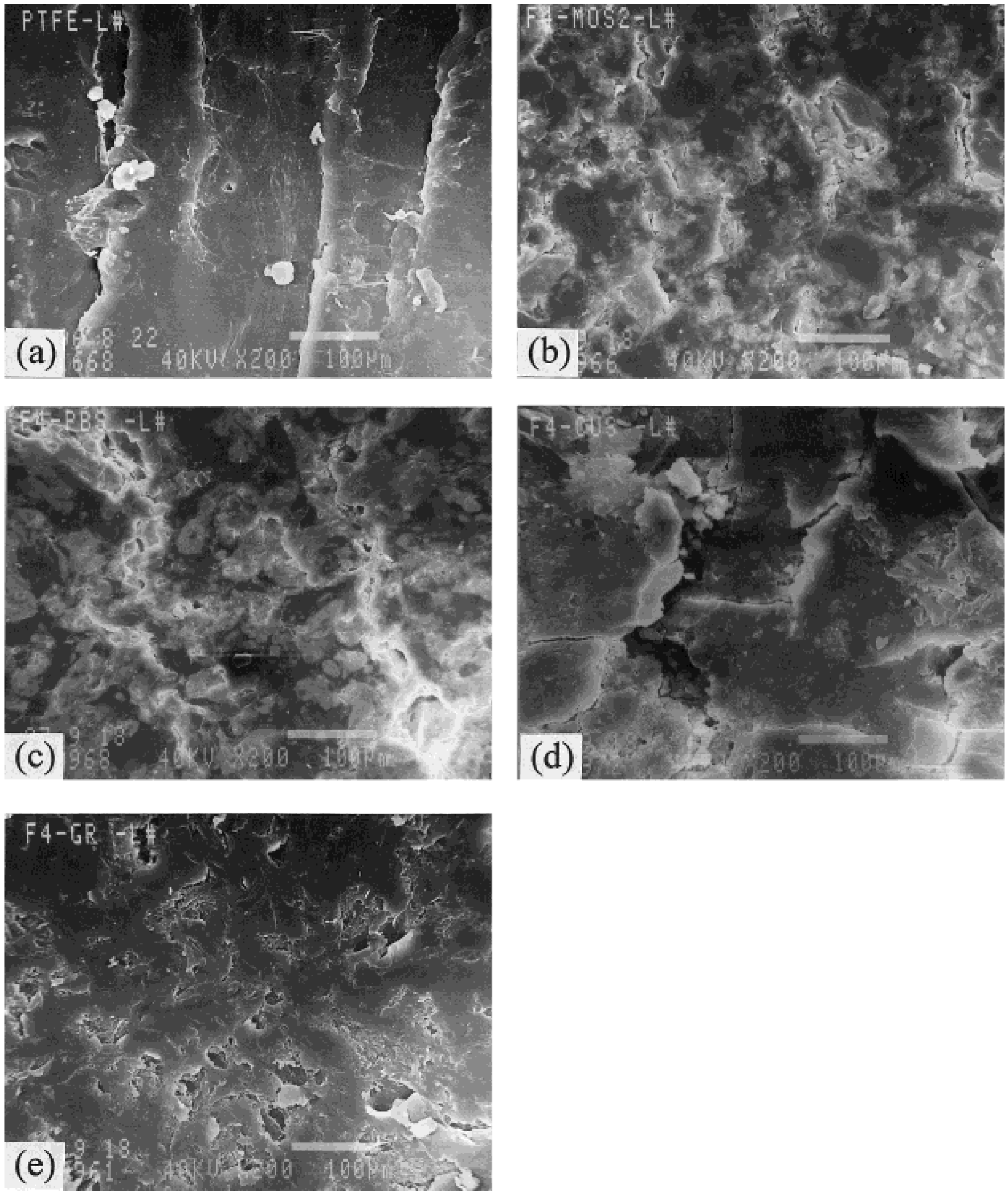

Figure 9 Electron micrographs of worn surfaces of the PTFE composites filled with metal sulfides or graphite sliding against GCr15-bearing steel under lubrication of liquid paraffin (sliding speed, 2.5 m/s): (a) pure PTFE, 1000*N;* (b) PTFE + 30(v)% MoS_2, 800*N;* (c) PTFE + 30(v)% PbS, 1000*N;* (d) PTFE + 30(v)% CuS, 1000*N;* and (e) PTFE + 30(v)% GR, 1000*N*.

filled PTFE composites under dry friction conditions.

The results in Figure 11 show that there are no obvious transfer films formed on the surface of GCr15-bearing steel for the PTFE composites filled with metal sulfides or graphite under lubrication of liquid paraffin. Comparison of the results in Figure 11 to those in Figure 10 indicates that the transfer of the PTFE composites onto the surface of GCr15-bearing steel can be greatly reduced by lubrication with liquid paraffin, but the transfer still takes place.[13,14] It is believed that the formation of lubricating oil films on the rubbing surfaces changes the contact form of the friction pair, so the friction and wear, as well as the transfer of the PTFE composites, can be greatly reduced. All of the above analyses are consistent with the results of the friction and wear tests.

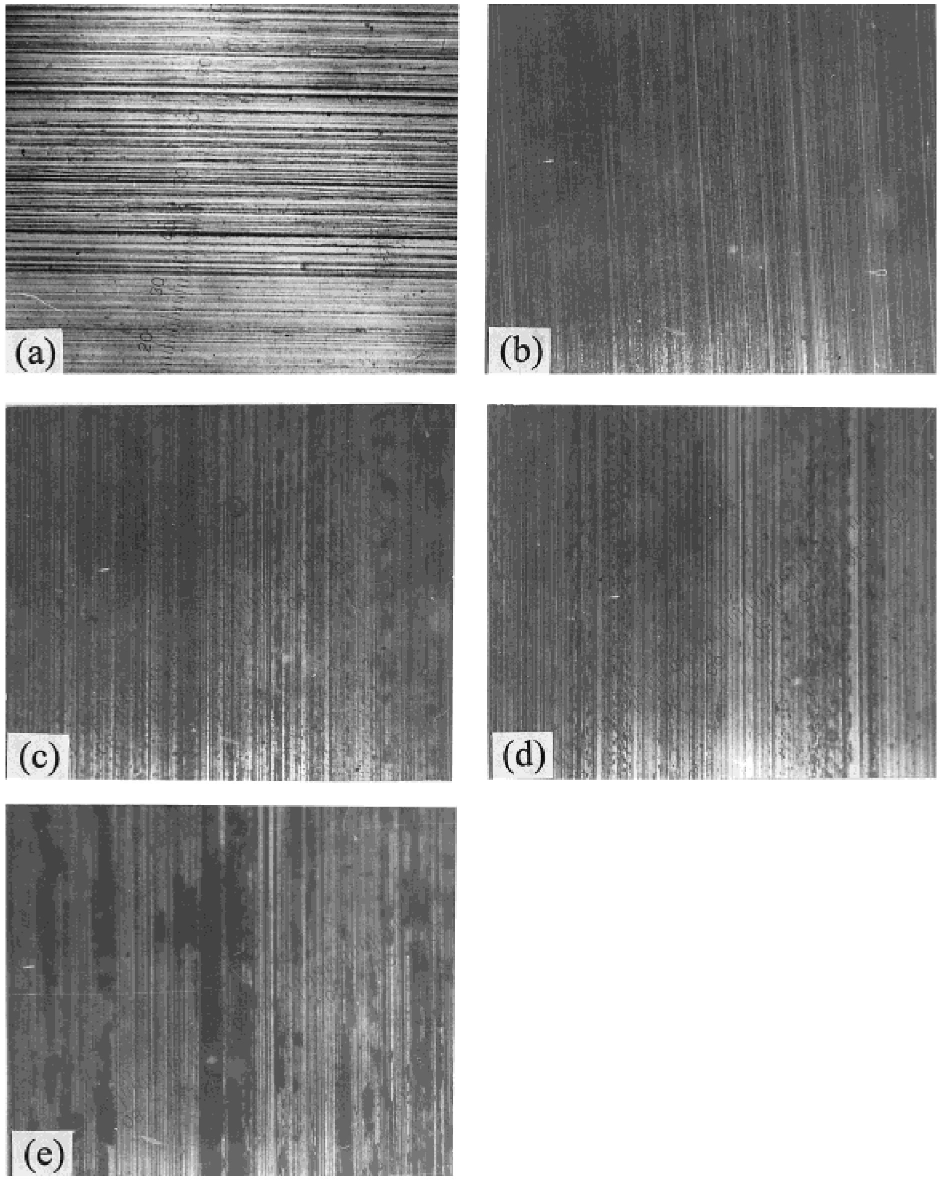

Figure 10 Optical micrographs of transfer films formed on the surface of GCr15-bearing steel for the PTFE composites filled with metal sulfides or graphite under the dry friction condition (128×) (sliding speed, 1.5 m/s; load, 100*N*): (a) PTFE; (b) PTFE + 30(*v*)% MoS_2; (c) PTFE + 30(*v*)% PbS; (d) PTFE + 30(*v*)% CuS; and (e) PTFE + 30(*v*)% GR.

CONCLUSIONS

1. Graphite reduces the friction coefficient of the PTFE composite, but metal sulfides MoS_2, PbS, and CuS increase the friction coefficients of the PTFE composites. As for MoS_2-, PbS-, or CuS-filled PTFE composites under dry friction conditions, the friction property of MoS_2-filled PTFE composite is the best, while that of CuS filled PTFE composite is the worst.
2. The antiwear properties of the PTFE composites can be greatly improved by filling PTFE with MoS_2, PbS, CuS, or graphite, and wear of the PTFE composites can be decreased by two orders of magnitude compared to that of pure PTFE under dry friction conditions. However, the wear-reduc-

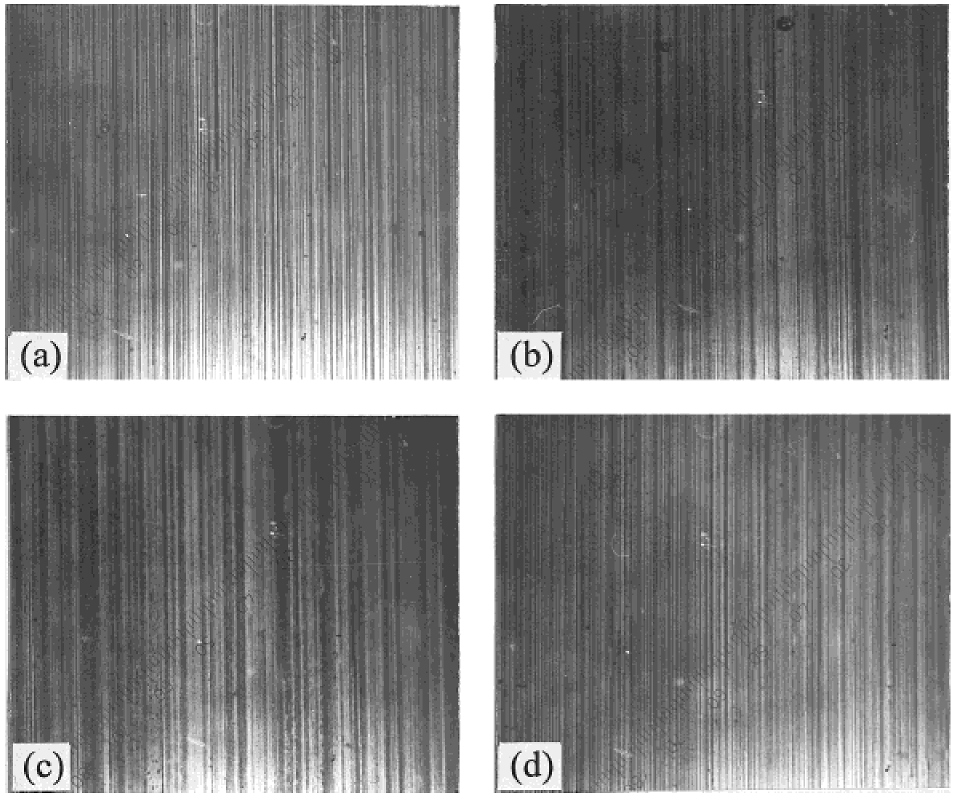

Figure 11 Optical micrographs of transfer films formed on the surface of GCr15-bearing steel for the PTFE composites filled with metal sulfides or graphite under lubrication of liquid paraffin (128×) (sliding speed, 2.5 m/s): (a) PTFE + 30(*v*)% MoS_2, 800*N;* (b) PTFE + 30(*v*)% PbS, 1000*N;* (c) PTFE + 30(*v*)% CuS, 1000*N;* and (d) PTFE + 30(*v*)% GR, 1000*N*.

ing action of PbS is the most effective, and that of MoS_2 is the worst.

3. The friction and wear-reducing properties of the metal sulfides or graphite-filled PTFE composites can be greatly improved by lubrication with liquid paraffin, and the friction coefficients of the PTFE composites can be decreased by one order of magnitude compared to those under dry friction conditions. Under lubrication of liquid paraffin, the wear of the PTFE composites increases with the increase of load, but the friction coefficients of the PTFE composites first decrease with the increase of load and then increase with the increase of load.
4. Under lubrication of liquid paraffin, the wear-reducing properties of the PTFE + 30(*v*)% PbS and PTFE + 30(*v*)% CuS composites are better than those of the other PTFE composites, while the friction property of pure PTFE is better than those of MoS_2-, PbS-, CuS-, and graphite-filled PTFE composites.
5. MoS_2, PbS, CuS, and graphite enhance the adhesion of the transfer films to the surface of GCr15-bearing steel and promote the transfer of the PTFE composites onto the surface of GCr15-bearing steel so that they greatly reduce the wear of the PTFE composites. However, the transfer of the PTFE composites onto the surface of GCr15-bearing steel can be greatly reduced by lubrication with liquid paraffin, but the transfer still takes place.
6. The interaction between liquid paraffin and the PTFE composites, especially the absorption of liquid paraffin into the microdefects of the PTFE composites under load, can create some cracks on the worn surfaces of the PTFE composites. The creation and the development of these cracks under load reduce the load-supporting capacity of the PTFE composites. This would lead to an increase of the friction and wear of the PTFE composites under higher loads in liquid paraffin lubrication.

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