Effect of Rare Earth Compounds as Fillers on Friction and Wear Behaviors of PTFE-Based Composites

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Received 26 June 1998; accepted 5 August 1998

ABSTRACT: The friction and wear behaviors of polytetrafluoroethylene (PTFE)-based composites filled with rare earth compounds CeO₂, CeF₃, and La₂O₃ in volume content from 5 to 30% sliding against GCr15 bearing steel under dry friction conditions were evaluated by using an MHK-500 ring-block wear tester. The wear debris and the worn surfaces of the PTFE composites were investigated by using a scanning electron microscope (SEM), whereas the transfer films of the PTFE composites formed on the surface of GCr15 bearing steel were examined by using an optical microscope. Experimental results show that the antiwear properties of the PTFE composites can be improved greatly by filling PTFE with different contents of CeO₂, CeF₃, and La₂O₃, and that the wear of the PTFE composites can be decreased by one to two orders of magnitude compared with that of pure PTFE. The wear of the CeF_3 -filled PTFE composite decreases with the increase of the content of CeF_3 from 5 to 30 vol %, but its friction coefficient increases with the increase of the content of CeF_3 in PTFE. When the content of CeO_2 in PTFE is 15 vol %, the friction and wear properties of CeO_2 -filled PTFE composite are the best. But when the content of La₂O₃ in PTFE is between 15 and 20 vol %, the PTFE composite filled with La₂O₃ exhibits excellent friction and wearreducing properties. Microscope investigations show that rare earth compounds CeO₂, CeF₃, and La₂O₃ in PTFE not only inhibit the large-scale destruction of the banded structure of PTFE but also enhance the adhesion of the transfer films of the PTFE composites to the surface of GCr15 bearing steel, so they greatly reduce the wear of the PTFE composites. With the increase of the content of CeO_2 , CeF_3 , and La_2O_3 in PTFE, the actions with which the rare earth compounds inhibit the large-scale destruction of the banded structure of PTFE are enhanced; so the wear of the PTFE composites decreases. Meanwhile, because CeO₂- and La₂O₃-filled PTFE composites can easily form uniform transfer films on the surface of GCr15 bearing steel, the friction and wear-reducing properties of CeO₂- and La₂O₃-filled PTFE composites are better than those of CeF₃-filled PTFE composites under dry friction conditions. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 72: 361-369, 1999

Key words: PTFE composites; rare earth compounds; friction and wear

INTRODUCTION

It is known that low friction coefficient, high thermal stability, and excellent chemical resistance make polytetrafluoroethylene (PTFE) an attractive engineering polymeric material for sliding applications. However, its high rate of wear and excessive viscoelastic deformation under load greatly limit the application of PTFE in practice. It has been found that almost any type of fillers or fibers can reduce greatly the wear of the PTFE

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Journal of Applied Polymer Science, Vol. 72, 361–369 (1999)

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composites.^{1,2} The friction and wear properties as well as the mechanisms of the PTFE composites filled with various kinds of fillers already have been studied by many coworkers.^{2–9}

Rare earth metal and rare earth compounds have been used widely in many engineering fields (such as optics, electronics, metallurgy, chemical engineering, special materials, etc.) because of their typical characteristics. In recent years, the applications and studies of rare earth compounds in tribology also have attracted the attention of tribologists throughout the world. At present, the friction and wear properties of rare earth compounds, such as CeO₂, La₂O₃, Y₂O₃, CeF₃, LaF₃, NdF_3 , SmF_3 and so forth, used as high-temperature lubricants, additives in lubricating oil, grease (paste), and bonding coatings have been studied.¹⁰⁻¹⁶ However, until now, much less information has been available about the effects of rare earth compounds as fillers on the friction and wear behaviors of polymers (such as PTFE).

The purpose of this work is to study the effect of rare earth compounds on the friction and wear behaviors of PTFE composites under dry friction conditions and gain some insights into the friction and wear mechanisms of rare earth compoundsfilled PTFE composites. It is expected that this study may be helpful to the applications of the PTFE composites filled with rare earth compounds in practice.

EXPERIMENTAL

The materials used for preparing the PTFE composites include PTFE powder with a grit size of about 30 μ m and rare earth compounds CeO₂, CeF_3 , and La_2O_3 with a grit size of about 76 μ m. The proportion of CeO_2 , CeF_3 , and La_2O_3 as fillers in PTFE was 5, 10, 15, 20, and 30 vol %, respectively. First, CeO_2 , CeF_3 , and La_2O_3 at a certain percent were mixed completely with the PTFE powder, respectively. Second, 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. The surfaces of these PTFE composite blocks were polished with number 800 grade SiC abrasive paper to a surface roughness of R_a $= 0.2 - 0.4 \ \mu m.$

The friction and wear tests were carried out on an MHK-500 ring-block wear tester (Timken wear tester, Jinan Testing Machinery Factory, P. R. China) with a steel ring, which is 49.2-mm diameter and 13.0-mm length, rotating on a PTFE composite block, which is $12.3 \times 12.3 \times 18.9$ mm in size. The steel ring, made of GCr15 bearing steel (C, 0.950–1.050%; Mn, 0.200–0.400%; Si, 0.150–0.350%; Cr, 1.300–1.650%; P, <0.027%; S, <0.020%; Fe, the remainder) was polished with number 900 grade SiC abrasive paper to a surface roughness of $R_a = 0.15 \ \mu$ m.

The friction and wear tests were performed at room temperature in ambient atmosphere (the relative humidity is between 35 and 40%) with a sliding speed from 1.0 to 2.5 m s^{-1} and loads from 100 to 400N in dry friction conditions. Each friction and wear test was performed for 30 min. Before each test started, the surfaces of the PTFE composite blocks and the GCr15 bearing steel ring were cleaned by rubbing with a soft cloth dipped in acetone and then air-dried. 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, whereas 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. In this work, three to five samples were tested at each condition; the friction coefficient and the wear were the average values of these tests for each condition.

The wear debris and the worn surfaces of CeO_2 -, CeF_3 -, and La_2O_3 -filled PTFE composites were investigated by using a JEM-1200EX/S scanning electron microscope (SEM), whereas the transfer films of rare earth compounds filled the PTFE composites formed on the surface of GCr15 bearing steel ring were examined by using an optical microscope.

RESULTS AND DISCUSSION

Effect of Content of Rare Earth Compounds on Friction and Wear of PTFE Composites

The relationship between the friction coefficients of the PTFE composites and the content of CeO₂, CeF₃, and La₂O₃ in PTFE under dry friction condition is shown in Figure 1. The results in Figure 1 show that the friction properties of CeO₂- and La₂O₃-filled PTFE composites are better than that of CeF₃-filled PTFE composite under dry friction condition. The friction coefficient of CeO₂filled PTFE composite first decreases with the



Figure 1 Relationship between the friction coefficients of the PTFE composites and the content of CeO_2 , CeF_3 , and La_2O_3 in PTFE under the dry friction condition (sliding speed, 1.5 m s⁻¹; load, 100*N*).

increase of the content of CeO_2 and then increases as the content of CeO_2 increases. When the content of CeO_2 in PTFE is 15 vol %, the friction coefficient of the PTFE composite is the lowest. However, the friction coefficient of the CeF_3 -filled PTFE composite increases with the increase of the content of CeF_3 . For the La_2O_3 -filled PTFE composite, when the content of La_2O_3 in PTFE is lower than 10 vol %, the friction coefficient of the PTFE composite increases with the increase of the content of La_2O_3 . Then the friction coefficient decreases with the increase of the content of La_2O_3 , and when the content of La_2O_3 in PTFE is 20 vol %, the friction coefficient of the PTFE composite is the lowest. But when the content of La_2O_3 in PTFE is higher than 20 vol %, the friction coefficient of the PTFE composite increases sharply with the increase of the content of La₂O₃.

The relationship between the wear of the PTFE composites and the content of CeO₂, CeF₃, and La₂O₃ in PTFE under dry friction condition is shown in Figure 2. Comparison of the wear results in Figure 2 with that of pure PTFE (385.4 mg)^{17,18} shows that filling PTFE with a different content of CeO₂, CeF₃, and La₂O₃ can reduce the wear of the PTFE composites by one to two orders of magnitude; therefore, CeO₂, CeF₃, and La₂O₃ can improve greatly the antiwear properties of the PTFE composites under dry friction conditions. The results in Figure 2 also show that the antiwear properties of CeO₂- and La₂O₃-filled PTFE composites are better than that of the

CeF₃-filled PTFE composite; this indicates that the wear-reducing actions of CeO₂ and La₂O₃ are more effective than that of the CeF₃. Meanwhile, the wear of CeF3-filled PTFE composite decreases with the increase of the content of CeF₃. However, the wear of CeO₂- and La₂O₃-filled PTFE composites first decreases with the increase of the content of CeO₂ and La₂O₃ and then increases as the content of CeO_2 and La_2O_3 increases. When the content of CeO_2 and La_2O_3 in PTFE is 15 vol %, the wear of CeO₂- and La₂O₃-filled PTFE composites is the lowest. But when the content of CeO_2 , CeF_3 , and La_2O_3 in PTFE is 30 vol %, the wear of CeO_2 -, CeF_3 -, and La_2O_3 -filled PTFE composites is almost the same under the given conditions in this experiment.

Therefore, it can be concluded from the results of Figures 1 and 2 that the friction and wearreducing properties of CeO₂- and La₂O₃-filled PTFE composites are better than that of the CeF₃-filled PTFE composite. When the content of CeO₂ in PTFE is 15 vol %, the friction and wear properties of CeO₂-filled PTFE composite are the best. Meanwhile, when the content of La₂O₃ in PTFE is between 15 and 20 vol %, La₂O₃-filled PTFE composite also exhibits excellent friction and wear properties. However, the friction coefficient of CeF₃-filled PTFE composite increases with the increase of the content of CeF₃, whereas its wear decreases with the increase of the content of CeF₃ from 5 to 30%.



Figure 2 Relationship between the wear of the PTFE composites and the content of CeO_2 , CeF_3 , and La_2O_3 in PTFE under the dry friction condition (sliding speed, 1.5 m s⁻¹; load, 100*N*).



Figure 3 Variations of friction coefficients with load for CeO₂-, CeF₃-, and La₂O₃-filled PTFE composites under dry friction conditions (sliding speed, 1.5 m s^{-1}).

Effect of Load and Velocity on Friction and Wear of PTFE Composites

The variations of friction coefficients and wear with load for the PTFE composites filled with CeO_2 , CeF_3 , and La_2O_3 in 15 vol % under dry friction conditions are shown in Figures 3 and 4, respectively. The results in Figure 3 show that the friction coefficient of the PTFE + 15 vol % CeF_3 composite decreases with the increase of load from 100 to 400N under dry friction conditions. When the load is lower than 200N, the friction coefficients of the PTFE + 15 vol % CeO₂ and the PTFE + 15 vol % La₂O₃ composites increase with the increase of load. But when the load is higher than 200N, the friction coefficient of the PTFE + 15 vol % CeO₂ composite decreases with the increase of load, whereas the load has little effect on the friction coefficient of the PTFE + 15 vol % La₂O₃ composite. Meanwhile, it can be seen from Figure 3 that the friction properties of the PTFE + 15 vol % CeO₂ and PTFE + 15 vol %



Figure 4 Variation of wear with load for CeO_2 -, CeF_3 -, and La_2O_3 -filled PTFE composites under dry friction conditions (sliding speed, 1.5 m s⁻¹).





Figure 5 Variation of friction coefficient with sliding speed for the PTFE + $15 \text{ vol } \% \text{ CeO}_2$ composite under dry friction conditions (load, 300N).

 La_2O_3 composites are better than that of the PTFE + 15 vol % CeF₃ composite under different loads in dry friction conditions. However, the friction property of the PTFE + 15 vol % La₂O₃ composite is the best under lower loads (<300N), whereas the friction property of the PTFE + 15 vol % CeO₂ composite is the best under higher loads (>300N).

The results in Figure 4 show that the wear of the PTFE composites filled with CeO₂, CeF₃, and La₂O₃ in 15 vol % increases with the increase of load under dry friction conditions. When the load is lower than 300*N*, the antiwear properties of the PTFE + 15 vol % CeO₂ and PTFE + 15 vol % La₂O₃ composites are better than that of the PTFE + 15 vol % CeF₃ composite. But when the load is higher than 300*N*, the antiwear property of the PTFE + 15 vol % CeO₂ composite is the best, whereas that of the PTFE + 15 vol % La₂O₃ composite is the worst. Therefore, the PTFE + 15 vol % CeO₂ composite exhibits excellent friction and wear properties under higher loads (>300*N*) in dry friction conditions.

The variations of friction coefficient and wear rate with sliding speed for the PTFE + 15 vol % CeO_2 composite in dry friction conditions 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 + 15 vol % CeO_2 composite increase with the increase of sliding speed. It is believed that, under dry friction conditions, the temperature at frictional surfaces increases with the increase of sliding speed;





Figure 6 Variation of wear rate with sliding speed for the PTFE + 15 vol % CeO₂ composite under dry friction conditions (load, 300*N*).

the temperature increase at frictional surfaces would lead to the reduction of mechanical strength and load-supporting capacity of the PTFE composite and so, in turn, would lead to the increase of the friction coefficient and wear rate of the PTFE composite. Therefore, the PTFE + 15 vol % CeO₂ composite is not suitable for application at higher sliding speed in dry friction conditions.

SEM Investigation of Wear Debris

The electron micrographs of the wear debris of CeO₂-, CeF₃-, and La₂O₃-filled PTFE composites under dry friction conditions are shown in Figure 7. It can be seen from Figure 7 that the wear debris of pure PTFE and the CeO_2 -, CeF_3 -, and La₂O₃-filled PTFE composites are all in the form of flakelike pieces, but the wear debris of CeO_2 -, CeF₃-, and La₂O₃-filled PTFE composites are much smaller than that of pure PTFE. This indicates that the rare earth compounds CeO_2 , CeF_3 , and La₂O₃ prevent the banded structure of PTFE from large-scale destruction, change the formation mechanism of the wear debris, and so make the wear debris transform from the large flakelike pieces of pure PTFE to the small flakelike pieces of the filled PTFE composites. Therefore, they greatly reduce the wear of the PTFE composites.^{3,4}

Meanwhile, the results in Figure 7 show that the wear debris of the PTFE + 30 vol % CeO₂,

PTFE + 30 vol % CeF₃, and PTFE + 30 vol % La₂O₃ composites are much smaller than those of the PTFE + 5 vol % CeO₂, PTFE + 5 vol % CeF₃, and PTFE + 5 vol % La₂O₃ composites. It is believed that, with the increase of the addition quantity of CeO₂, CeF₃, and La₂O₃ in PTFE, the actions with which the rare earth compounds inhibit the large-scale destruction of the banded structure of PTFE are enhanced; this would lead to the reduction of the size of the wear debris and so, in turn, lead to the decrease of the wear of the PTFE composites. These analysis results are consistent with the results of wear tests.

SEM Investigation of Worn Surfaces

It was found in the experiments that the width of the wear scar on the worn surface of pure PTFE is about 12 mm under the dry friction condition, but the width and the depth of the wear scars on the worn surfaces of CeO_2 -, CeF_3 -, and La_2O_3 -filled PTFE composites are much smaller than that of pure PTFE. Figure 8 gives the electron micrographs of the worn surfaces of CeO₂-, CeF₃-, and La₂O₃-filled PTFE composites sliding against GCr15 bearing steel under dry friction conditions. It can be seen from Figure 8 that there are still some smaller wear scars in the large wear scar of pure PTFE, but the worn surfaces of CeO₂-, CeF_3 -, and La_2O_3 -filled PTFE composites are smoother than that of pure PTFE. This indicates that filling CeO_2 , CeF_3 , and La_2O_3 to PTFE can reduce greatly the wear of the PTFE composites and therefore make the antiwear properties of the PTFE composites be improved greatly.

Meanwhile, it can be seen from Figure 8 that adhesive wear occurs to the PTFE + 15 vol % CeF_3 and PTFE + 15 vol % La_2O_3 composites under higher loads (>200N) in dry friction conditions. Therefore, it can be deduced that the friction and wear properties of the PTFE + 15 vol % CeF_3 and PTFE + 15 vol % La_2O_3 composites are poorer than those of the PTFE + 15 vol % CeO_2 composite under higher loads (>200N). These analysis results also are consistent with the results of the friction and wear tests under dry friction conditions.

Optical Microscope Examination of Transfer Films

The optical micrographs of the transfer films formed on the surface of GCr15 bearing steel for CeO₂-, CeF₃-, and La₂O₃-filled PTFE composites under dry friction conditions are shown in Figure



Figure 7 Electron micrographs of the wear debris of CeO_2 -, CeF_3 -, and La_2O_3 -filled PTFE composites under dry friction conditions (sliding speed, 1.5 m s⁻¹; load, 100*N*). (a) PTFE, (b) PTFE + 5 vol % CeO₂, (c) PTFE + 5 vol % CeF₃, (d) PTFE + 5 vol % La₂O₃, (e) PTFE + 30 vol % CeO₂, (f) PTFE + 30 vol % CeF₃, and (g) PTFE + 30 vol % La₂O₃.



Figure 8 Electron micrographs of the worn surfaces of CeO_2 -, CeF_3 -, and La_2O_3 -filled PTFE composites under dry friction conditions (sliding speed, 1.5 m s⁻¹). (a) PTFE, 100N; (b) PTFE + 15 vol % CeO_2 , 300N; (c) PTFE + 15 vol % CeF_3 , 300N; and (d) PTFE + 15 vol % La_2O_3 , 300N.

9. It can be seen from Figure 9 that there are some obvious transfer films formed on the surface of GCr15 bearing steel for these composites, 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 under dry friction conditions, it can be deduced that the rare earth compounds CeO_2 , CeF_3 , and La_2O_3 enhance the adhesion of the transfer films to the surface of GCr15 bearing steel and thus 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.^{5,6}

However, the results in Figure 9 show that the transfer films of CeO_2 - and La_2O_3 -filled PTFE composites formed on the surface of GCr15 bearing steel are thicker than those of CeF_3 -filled PTFE composite. This indicates that the adhesion between the transfer film of a CeF_3 -filled PTFE composite and the surface of GCr15 bearing steel is weak, so it is difficult to form uniform transfer films on the surface of GCr15 bearing steel for the

CeF₃-filled PTFE composite. However, CeO₂- and La₂O₃-filled PTFE composites can easily form uniform transfer films on the surface of GCr15 bearing steel, and then the friction between the PTFE composites and GCr15 bearing steel can be transformed to the friction between the PTFE composites and its transfer films formed on the surface of GCr15 bearing steel. Therefore, the friction and wear properties of CeO₂- and La₂O₃-filled PTFE composites are better than those of CeF₃-filled PTFE composite under the given conditions in this work. These analysis results also are consistent with the results of the friction and wear tests as shown in Figures 1 and 2.

CONCLUSIONS

1. The antiwear properties of the PTFE composites can be improved greatly by filling PTFE with a different content of CeO₂, CeF₃, and La₂O₃, and the wear of the PTFE composites can be decreased by one to two



Figure 9 Optical micrographs of the transfer films formed on the surface of GCr15 bearing steel for CeO₂-, CeF₃-, and La₂O₃-filled PTFE composites under the dry friction condition (×128) (sliding speed, 1.5 m s⁻¹; load, 100*N*). (a) PTFE, (b) PTFE + 15 vol % CeO₂, (c) PTFE + 15 vol % CeF₃, and (d) PTFE + 15 vol % La₂O₃.

orders of magnitude compared with that of pure PTFE under the dry friction condition. However, the friction and wear-reducing properties of CeO_2 - and La_2O_3 -filled PTFE composites are better than those of CeF_3 -filled PTFE composites.

- 2. The friction coefficient and the wear of the CeO_2 -filled PTFE composite first decrease with the increase of the content of CeO_2 in PTFE and then increase as the content of CeO_2 in PTFE is 15 vol %, the friction and wear properties of CeO_2 -filled PTFE composite are the best.
- 3. When the content of La_2O_3 in PTFE is between 15 and 20%, the La_2O_3 -filled PTFE composite exhibits excellent friction and wear-reducing properties. For the CeF₃filled PTFE composite, its friction coefficient increases with the increase of the content of CeF₃ in PTFE, but its wear decreases with the increase of the content of CeF₃. However, when the content of CeO₂,

CeF₃, and La₂O₃ in PTFE is 30 vol %, the wear of CeO₂-, CeF₃-, and La₂O₃-filled PTFE composites is almost the same under the given conditions in this work.

- 4. The wear of the PTFE composites filled with CeO_2 , CeF_3 , and La_2O_3 increases with the increase of load. Meanwhile, the friction coefficient and the wear rate of the PTFE + 15 vol % CeO_2 composite also increase with the increase of sliding speed, so the PTFE + 15 vol % CeO_2 composite is not suitable for application at higher sliding speed under higher load in dry friction conditions.
- 5. Rare earth compounds CeO_2 , CeF_3 , and La_2O_3 prevent the banded structure of PTFE from large-scale destruction and change the formation mechanism of the wear debris, so they greatly reduce the wear of the PTFE composites. With the increase of the additional quantity of CeO_2 , CeF_3 , and La_2O_3 in PTFE, the actions with which the rare earth compounds inhibit

the large-scale destruction of the banded structure of PTFE are enhanced, so the antiwear properties of the PTFE composite can be improved greatly.

6. Rare earth compounds CeO_2 , CeF_3 , and La_2O_3 enhance the adhesion of transfer films to the surface of GCr15 bearing steel and thus promote the transfer of the PTFE composites onto the surface of GCr15 bearing steel, so they greatly reduce the wear of the PTFE composites. Because the formation ability of transfer films of CeO_2 - and La_2O_3 -filled PTFE composites on the counterfaces is better than that of the CeF₃-filled PTFE composite, the friction and wear properties of CeO_2 - and La_2O_3 -filled PTFE composite, the friction and wear properties of CeO_2 - and La_2O_3 -filled PTFE composites are better than those of CeF_3 -filled PTFE composites under dry friction conditions.

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