

Tribological Action of Metallic Fillers in Poly(tetrafluoroethylene) Composites

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Received 25 May 2000; accepted 21 February 2001

ABSTRACT: The tribological behavior of poly(tetrafluoroethylene) composites filled with various metallic particles was investigated under the extreme friction conditions of a heavy load and reciprocation with a small amplitude. Two obviously distinct friction stages corresponding to the contact conditions were found. The phenomena of the accumulation of fillers on the contact surface and extrusion wear of the poly(tetrafluoroethylene) matrix were also observed. Based on the experimental results, the actions of metallic fillers on the transfer film formation were analyzed. It was noted that the transfer and accumulation of fillers could actually be considered to be an assimilating process of the friction pairs. Accordingly, the physical model of the transfer film formation process was described and some suggestions on the mechanism of load-supporting actions of hard fillers were also proposed. © 2002 John Wiley & Sons, Inc. *J Appl Polym Sci* 83: 1832–1840, 2002

Key words: poly(tetrafluoroethylene); composite; transfer film; metallic filler; transfer process; load-supporting action

INTRODUCTION

Polymer composites can offer a combination of properties for a diversity of applications unobtainable with metals, minerals, ceramics, or polymers alone. They have emerged as a new-generation material with tremendous potential as an alternative to polymers in specific applications. In the tribological field, poly(tetrafluoroethylene) (PTFE) exhibits a low friction coefficient but a high rate of wear and cold-flowing phenomena under load. Various fillers have been added to the polymer to achieve the desired properties of

longer wear life. For the development of novel polymer composites, it is necessary to understand the mechanisms of the tribological actions of fillers.

Briscoe and coworkers pointed out that fillers do not appear to significantly modify the chemical or physical structure of the polymer but interact strongly with the substrate during the transfer process.¹ The mechanism responsible for enhanced adhesion between the transfer film and its counterpart surface has been found by many researchers to be either physical or chemical in nature.^{1–7} It is widely accepted that the transfer film is held by a combination of chemical or physical bonding between the filler and the substrate and physical or mechanical bonding between the polymer and the filler.¹

Khrushchov demonstrated in abrasive wear tests involving multiphase metal systems that the wear resistance of the composite system dis-

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Contract grant sponsor: National Natural Science Foundation of China; contract grant number: 29871033.
Contract grant sponsor: High-Tech R & D Bureau, Chinese Academy of Sciences.

Journal of Applied Polymer Science, Vol. 83, 1832–1840 (2002)
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DOI 10.1002/app.2309

plays a linear rule of mixtures dependence on the wear resistances and bulk volume fractions of its components.⁸ Axen and Jacobson showed that such linear rule of mixtures behavior of wear resistance would be predicted in "equal wear" cases where the system components must share the same rates of wear depth per unit of time.⁹ The surface enrichment of filler during sliding was observed experimentally by some researchers; this was especially true for the hard particulate fillers in viscoplastic polymer matrixes.^{1,4,10} The results also support the view of the linear rule of mixtures of wear resistance.¹¹ Recently, a model for the steady-state wear behavior of polymer composite materials, including the effects of preferential load support by and surface accumulation of wear-resistant filler particles, was further developed experimentally and theoretically by Han and Blanchet.^{11,12}

Another mechanism of the wear-reducing actions of the fillers in polymer composites is the preferential load support of fillers, which was proposed by Lancaster and further developed theoretically by Tanaka and Kawakami.¹³ The preferential load support action of fillers was considered to be due to the existence of excess tangential stress on the filler surface along the loading direction. In this case, the wear-reducing actions of the fibers were then thought to be more effective than those of particle fillers or lamellar solid lubricants.

Although a lot of attention was paid to the tribological actions of various fillers during friction, there are still some unanswered questions. Tribological researchers emphasize the influence of friction, which is the shear force, but usually neglect the load effect, which is the vertical force. The purposes of this work were to study the characteristics of various metallic fillers influencing the tribological behavior and transfer of polymer composite under a type of extreme friction condition (high load and reciprocating motion) and to support or supply more evidence for the proposed viewpoints, such as the transfer process and the preferential load support of hard fillers.

EXPERIMENTAL

Wear-Testing Machine and Experimental Conditions

A ball on disk reciprocating wear tester was employed in this work. All friction and wear tests

were carried out in air under a normal load of 200 N, an amplitude (stroke) of 1.5 mm, a frequency of 20 Hz, a room temperature of about 23°C, and 64% relative humidity. The average reciprocating sliding speed corresponding to both the amplitude and frequency was 0.06 m/s. The test duration for each specimen was 5 min (18-m sliding distance). The counterpart steel ball with a diameter of 10 mm and roughness of about 0.005 μm was made of GCr15 bearing steel (SAE52100 Steel). Before the friction and wear test, the balls and disks were cleaned by rubbing them with soft cotton dipped in acetone. Each composite specimen was directly used in the friction and wear test without further mechanical processing to minimize the influence of other operations. The roughness of the composite disk was measured to be about 0.05 μm before a test. The measurements of the friction coefficient were made continuously during the wear process, and the wear volumes were obtained by measuring the profiles and worn surface areas of wear scars.

The friction conditions used in this work can be considered to be a typical condition of a heavy load and low reciprocating velocity, which was assumed to be a simulation of asperity contact under high stress.

Specimens

Seven kinds of PTFE-based composites filled with metallic particles of Cr, Fe, Ni, Cu, Pb, Zn, and Sn were prepared into 8-mm thickness, 24-mm diameter plates by compression molding at 370°C in a hydrogen atmosphere. The consolidating pressure of the composite was 40 MPa with a continuance time of 2 min. The volume content of each filler in the composite was fixed at 30%. The grit size of each metallic filler particle (purity above 98.0%) was controlled at about 70 μm by sifting.

RESULTS

Friction and Wear Characteristics

According to our previous work and that of other researchers,^{3,4,7,14} under the normal sliding conditions of a steel ring on a composite disk or composite pin on a steel disk, all metallic fillers used in this work should obviously decrease the wear of PTFE but increase its friction coefficient. Strangely, however, under the friction conditions of a heavy load and reciprocation with a small

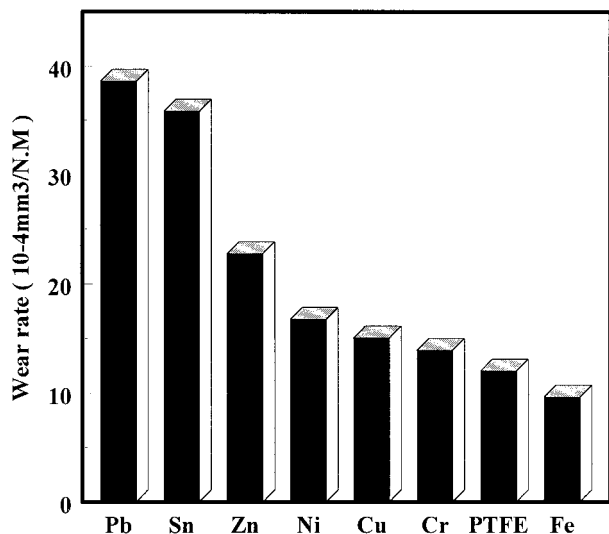


Figure 1 The wear rates of PTFE and PTFE composites filled with various metallic fillers (30 vol %).

amplitude, there was no obvious wear-reducing action of the fillers observed. Figure 1 shows the wear rates of PTFE composites filled with various metallic fillers. An approximate rule seemed to exist: the softer the metallic filler, the higher the wear rate of the composite. The wear rate of the Fe-PTFE composite did not obey the rule. This was supposed to be determined by the strong adhesion between the tribopairs of the same composition. The above results indicated that the tribological actions of metallic fillers were strongly restricted to the friction and wear conditions.¹ No wear of the counterpart steel ball under the protection of a transfer film, except for sliding against the Cr-PTFE composite, was found in this work.

The typical friction curves of pure PTFE and its composites (e.g., Cu-PTFE composite) are shown in Figure 2. Note that the friction coefficient of pure PTFE was relatively stable at about 0.15. For the composites filled with metallic fillers, two obviously distinct friction stages, the first steady stage and the second steady stage, were observed on the friction curves. In the first steady stage, which in some cases was very short (about 10 s), the friction coefficients of the PTFE composites were usually as low as 0.15, similar to pure PTFE. However, in the second stage the friction coefficients of the composites increased to higher values of about 0.25–0.32. This change should not have been completely attributable to the contact stress changing from point to plane with the wear of the materials, because the same

phenomenon for pure PTFE was not observed. One important reason was that the friction gradually changed with sliding from polymer against steel to composite against steel or composite against transferred composite on the counterface if a transfer film existed.

Surface Analysis of Tribopairs in First Steady Stage

It was interesting to note that in the first stage, as a common phenomenon for each specimen, there were almost no transferred metallic elements of fillers formed on the counterpart surface; only transferred PTFE was detected by Auger electron spectroscopy (AES) analysis. The same phenomena were found for the initial worn surfaces of the composites. This indicated that at the commencement of friction the PTFE transferred to the counterpart surface preferentially. Figure 3 shows the AES spectra of the counterpart surface sliding against the Sn-PTFE composite in different friction stages. In the first stage there were no Sn peaks on the AES spectrum (Fig. 3, spectrum a).

Surface Analysis of Tribopairs in Second Steady Stage

In the second stage the metallic fillers were exposed on the worn surfaces of the composite matrix. The AES spectra of the worn specimens simultaneously showed the peaks of filler metal, C, F, and O, where C and F were attributed to PTFE and O and some C were due to adsorbed elements on the specimen surface from the environment.

The AES analysis of the surfaces of the counterpart steel balls showed that there were three

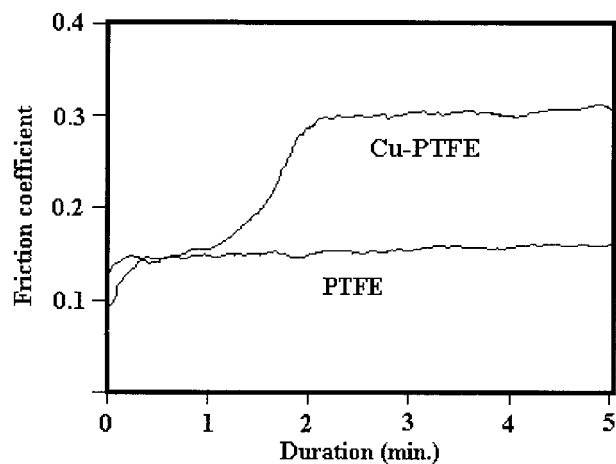


Figure 2 The typical friction curves of PTFE and Cu-PTFE composites.

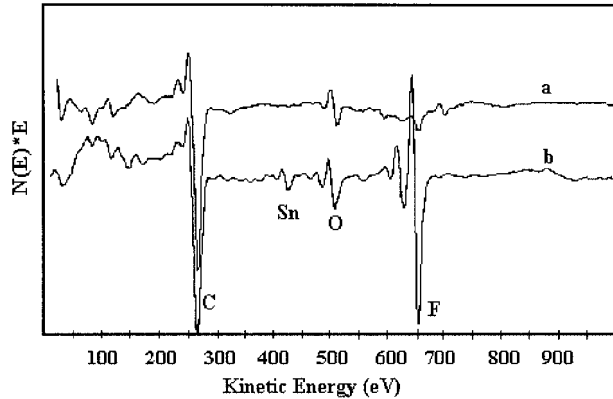


Figure 3 AES spectra of Sn-PTFE transfer film at the first (spectrum a) and second (spectrum b) steady stage.

kinds of phenomena occurring in the second steady stage. The first was that for most metallic filler filled PTFE composites the transfer of composite to counterface could occur and the transferred film on the counterface was always composed of filler metal and PTFE, such as the Sn-, Fe-, Pb-, Cu-, and Ni-PTFE composites in our study. In this case the friction was changed from polymer against counterpart steel in the first steady stage to the polymer composite against the transferred composite on the counterpart surface. It was interesting to note that the composition or metallic filler content on the worn surface and on the transferred film were not the same as that in the bulk composite.^{1,4,10-12} The accumulation of metallic filler on the contact surface was also found in this work.

The second case was found for the counterface sliding against the Zn-PTFE composite. Figure 4 shows the AES spectrum of the counterface of the steel ball in the second steady stage. Interestingly, there were no AES peaks of C and F, which denote the existence of PTFE. This indicated that the PTFE could not transfer or be held mechanically on the surface of the transfer film of Zn. In other words, the transfer film was composed entirely of Zn, the metallic filler.

The third case was found for the counterface sliding against the Cr-PTFE composite. Neither an F peak nor a Cr peak was observed in this case. This indicated that no transfer occurred during the friction process. As is known, unfilled PTFE can easily transfer to a steel surface, as shown in spectrum a in Figure 3. Thus, the absence of transfer film on the counterface sliding against the Cr-PTFE composite may be mainly attributed

to the fact that Cr cannot transfer to the steel surface because its hardness is much higher than the counterface or its properties are incompatible with the counterface. Another reason could be that Cr particles accumulate gradually on the worn surface of the composite and the accumulated Cr particles on the composite surface may prevent the formation of PTFE transfer film by an erasing action.

Accumulation of Metallic Fillers

Under the friction conditions of a heavy load and reciprocation with a small amplitude the hard metallic fillers are easily accumulated on the worn surfaces of PTFE composites and then in the wear debris and transferred film. This is because the metallic fillers cannot be removed with the polymer at the same rate. As an example, Figure 5 shows the SEM micrographs [Fig. 5(A)] and Ni X-ray dot maps [Fig. 5(B)] of the wear debris, wear trace, and transfer film of the Ni-PTFE composite. It clearly shows that the Ni fillers accumulated on the wear debris, wear trace, and transfer film during the friction. Direct observation with the naked eye also indicated that in the first friction stage the wear debris was white (the color of PTFE) while in the second stage the wear debris changed to the color of the metallic filler.

The whole transfer film on a counterface sliding against the Zn-PTFE composite was composed of Zn. Figure 6(A) shows the SEM micrograph of its transfer film. If the hard metallic filler could not transfer to the counterpart surface, then no PTFE transfer film could be formed steadily and the counterface could be worn by the accumulated fillers. Figure 6(B) shows the SEM micrograph of the counterface scratched by the hard Cr fillers.

Regardless of whether a transfer film could be formed or not, the accumulation of the hard me-

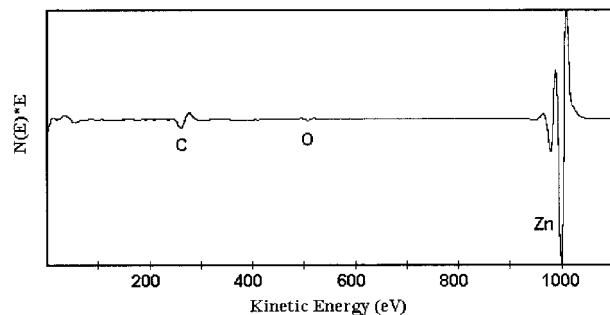


Figure 4 The AES spectrum of Zn-PTFE transfer films in the second steady stage.

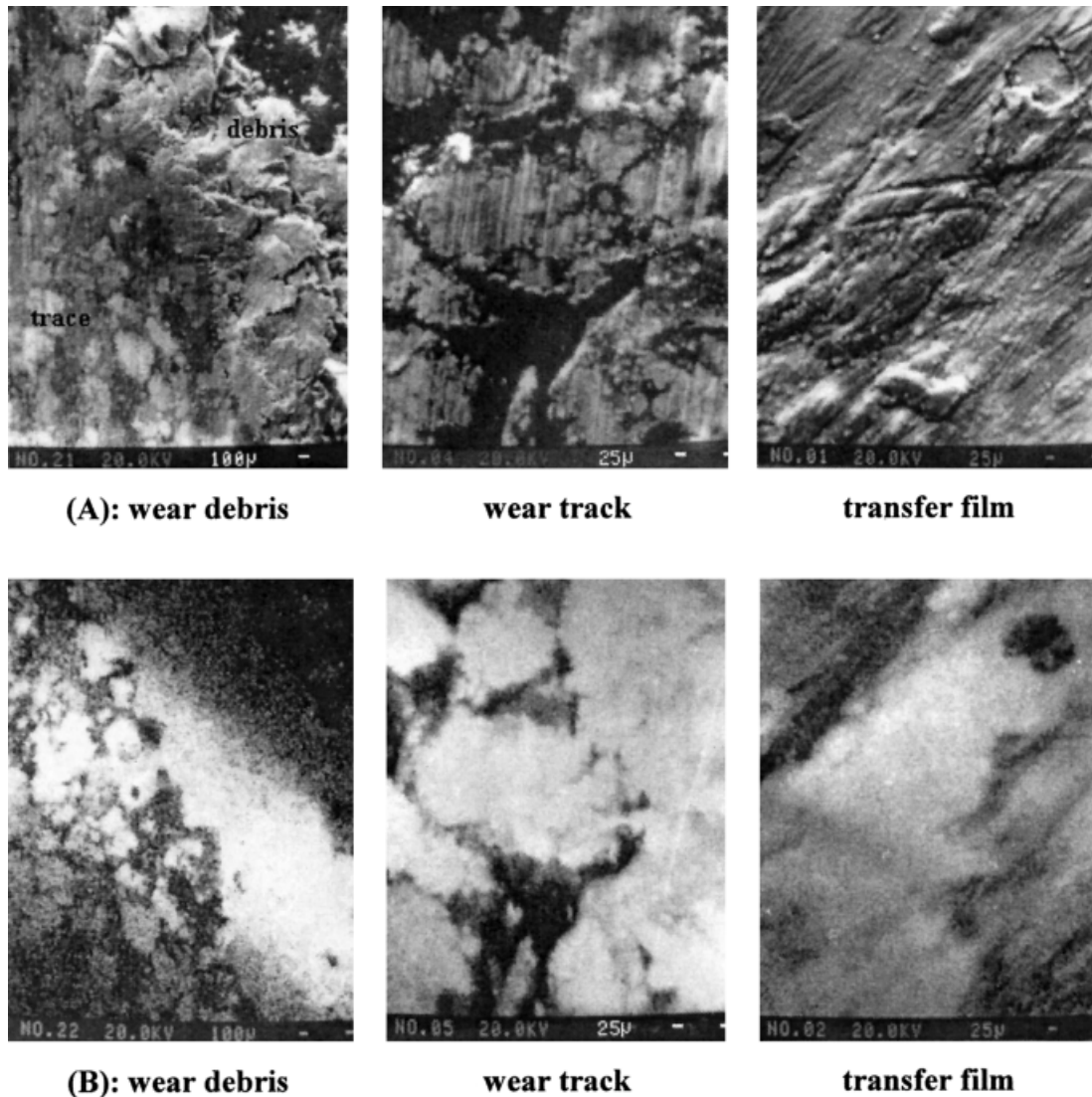


Figure 5 The (A) SEM micrographs and (B) Ni dot maps of the wear debris (original magnification $\times 50$), wear trace (original magnification $\times 100$), and transfer film (original magnification $\times 100$) of the Ni-PTFE composite; the white dots represent the distribution of Ni.

tallic fillers on the worn surface of the composite always occurred under the friction conditions of a heavy load and reciprocation with a small amplitude. This indicated that the removal rates of hard particles and PTFE were different under these conditions. In other words, the composition of the wear debris differed from that of the bulk composite.

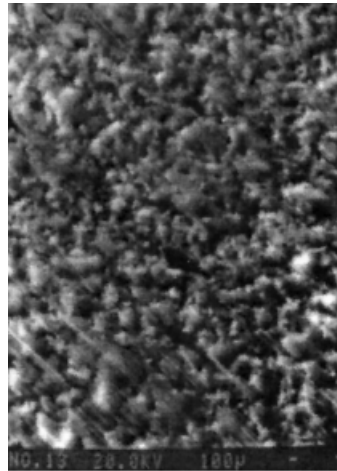
DISCUSSION

The friction conditions; wear mechanisms; load-supporting action of fillers; transfer process; and

interaction among the filler, polymer, and counterpart surface were all factors influencing the wear behavior of the composites. In this study the different fillers had quite different wear-reducing roles in the PTFE composites (Fig. 1).

Friction State and Wear Mechanism

The ball on disk contact conditions in this study are an extreme friction state in which the normal stress is much higher than the compressive yield strength of the composite. Even if there is no action of the friction force, an obvious spherical



(a)



(b)

Figure 6 SEM micrographs of (A) the transfer film (original magnification $\times 50$) of Zn-PTFE composite and (B) the worn surface (original magnification $\times 100$) of the counterpart sliding against the Cr-PTFE composite.

depressed scar still exists. The process of a steel ball indenting the counterpart composite disk is a process in which the compressive stress is continuously reduced as the contact area gradually increases with the increase of the indentation depth. The reciprocating of the steel ball restricts the tendency of the compressive stress to decrease, although the wear depth becomes deeper and deeper with the wear process. This is because the actual contact area is only at the bottom and sides (perpendicular to the friction direction) of the steel ball when the ball is reciprocating. Thus, the conclusion is that the dynamic loading stress is higher than the static one.

It was found in our previous work that the wear of PTFE under a heavy load and small amplitude reciprocation was mainly due to the extrusion caused by the counterpart steel ball and the friction force acting on the softened polymer matrix at higher temperature.¹⁵ The wear debris was in the form of complete unbroken wavelike ribbons for the ductile PTFE-based composites or flakes for PTFE-based composites with low ductility (high filler content). In this work we found that at the commencement of friction there was unbroken wear debris while in the second steady stage of friction the wear debris changed to flakes. This also indicated that the relatively rigid metallic fillers did accumulate on the worn surface of the composites.

Transfer Process

It is believed that the characteristics of filler particles influence the transfer behavior and wear behavior of polymer-based composites. The model proposed by Blanchet and Han for the transient running-in and steady-state wear behavior of polymer composites, including the effects of preferential load support by and surface accumulation of wear-resistant filler particles, is reasonable and may be used to explain the transfer behavior of polymer composites.^{11,12} Based on their wear model, the transfer behavior of metal-filled polymer composites is discussed in this study.

As a general rule, the outer surfaces of polymer composites are always coated by the polymer layers. Therefore, it is understandable that the friction coefficient of polymer composites is always lower at the commencement of friction. The physical model for the transfer film formation process of PTFE composites under a heavy load and small-amplitude reciprocation is given in Figure 7. In the first steady stage a very thin transfer film of pure PTFE formed initially on the counterpart surface because the PTFE on the surface

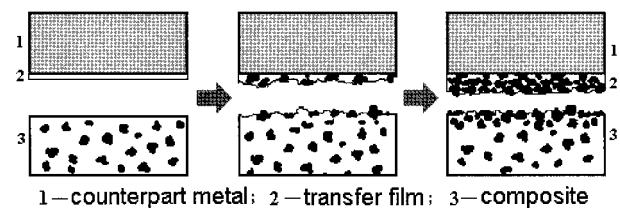


Figure 7 The physical model of the transfer process of metal-PTFE composites.

of the composite preferentially contacted with the counterpart surface. In this case the state of contacting couples could be considered as PTFE sliding against PTFE (transfer film) with a resultant low friction coefficient. With repeated sliding the PTFE on the surface of composite was removed from the contacting region, and the hard metallic fillers were exposed to the transfer film of PTFE or the counterpart surface. When the transferred PTFE film was partially scraped off, the exposed fillers came into direct contact with the counterpart steel surface, forming a composite transfer film with higher shear strength. According to the viewpoints of cohesive strength and surface energy,¹⁶ the transfer of metallic fillers should be much easier than that of PTFE because the surface energy of PTFE is much lower than that of metals. The metallic transferred film possesses higher hardness and shear strength than the polymer. Thus, it can be concluded that the action of metallic filler on the transfer film formation should be divided into two aspects: the hard filler exposed on the wear surface of the composite can scrape the polymer transfer film off the counterpart surface, and the transferred film composed of metallic filler can mechanically hold the polymer and form a composite transfer film with higher mechanical strength than the polymer. It was interesting that no PTFE was observed on the rough transfer film of Zn [Fig. 6(A)]. This could be because the debris of PTFE was extruded in the form of an unbroken ribbon and the adhesion force between Zn and PTFE was too weak to hold the PTFE.

When the process of the transfer film formation on the counterpart surface and the process of the metallic fillers accumulating on the worn surface of the composites were observed, it was found that for the tribopairs with different properties (hardness, composition, etc.) the transfer and the accumulation could actually be considered as a mutual assimilating process of the pairs. Subsequently, for the steel ball counterpart, with the formation of the composite transfer film the nature of the counterpart surface (hardness, composition, etc.) gradually approached that of the composite (the transfer film can decrease the hardness and change the composition of the counterpart); for the composite, the accumulation of metallic fillers changed the surface hardness and surface composition of the composite to approximate the counterpart steel ball (the accumulation of fillers increased the hardness and changed the composition of the composite sur-

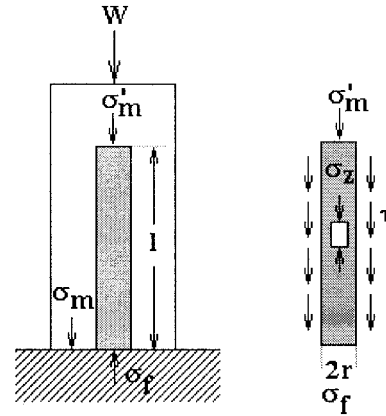


Figure 8 The theoretical analysis of the load-supporting action of the fiber-filled polymer proposed by Tanaka and Kawakami¹³; a contact model of the polymer-based composite incorporating the fiber and buildup of compressive stress (σ_z) in the fiber due to excess tangential surface stress (τ).

face). A balancing process was reached under the action of friction. It should be pointed out that the balance state, for example, for the surfaces of the Cr-PTFE/steel ball pair, was never reached. However, the tendency of mutual assimilation of the pair still existed, which was characterized by the dynamically contacting interfaces of pairs with different properties, similar to the physical phenomenon of diffusion between interfaces.

Suggestions on Preferential Load-Supporting Action of Fillers

The load-supporting action of fillers proposed by Lancaster was theoretically explained by Tanaka and Kawakami by introducing the concept of an excess tangential surface stress (τ) on the filler along the direction of loading.¹³ The theoretical analysis is cited in Figure 8, where the stress σ_f is the contact stress of the filler and is expressed as

$$\sigma_f = \frac{2\pi r l \tau}{\pi r^2} + \sigma'_m = \frac{2l}{r} \tau + \sigma'_m \quad (1)$$

In our work, it was found that the load-supporting action of the hard fillers was reasonable, which differed from the theoretical analysis of Tanaka and Kawakami.¹³ The key problem was the concept of the τ on the fillers. Mechanical analysis indicated that the τ was the relative motion (or tendency of relative motion) between the filler and the matrix under the load, and the motion

was understood to be the deformation of the contact surface of the specimen. When the compression displacement rate of the filler was faster than that of the matrix, the direction of τ was downward as shown in Figure 8 by Tanaka and Kawakami.¹³ However, when the displacement rate of the filler was slower than that of the matrix, the direction of τ changed to upward. If there is no relative motion between the filler and matrix, then there will be no tangential stress. Because PTFE possesses the characteristic of "cold flow" under load and lower modulus compared with the fillers, the compression of the matrix should occur preferentially to the filler, which is also one reason for the exposure and accumulation of fillers on the contact surface during friction. Thus, the direction of the τ should be upward, and the inverse conclusion will be obtained.

The mechanism of the load-supporting action of the hard filler was reanalyzed in this work. Figure 9 shows a schematic of the mechanical model. For the normal situation, the surface area (S_1) of the lower end of the filler in contact with the counterpart surface was usually much smaller than the area of the cross section (S_2) of the filler in the polymer matrix (Fig. 9). According to the principle of stress transmission of a solid, when the filler and matrix are relatively immobile, no tangential stress exists and the force equilibrium acting on the filler ($\Sigma F = 0$) should be satisfied, where $\sigma_f \times S_1$ is equal to the $\sigma'_m \times S_2$. The equation can be expressed as

$$\sigma_f = \frac{S_2}{S_1} \sigma'_m \quad (2)$$

When the test duration (time) is not very long, the influence of the cold flow behavior of the polymer on the analysis of the mechanical model of the load-supporting action of fillers can be ignored. Two basic situations that are involved should be discussed.

In the low compressive stress situation there is a low load and the same wear rates. When the average initial load stress (σ_{m0} , which is the ratio of the load to the whole contact surface area) is equal to or less than the compressive yield strengths of the polymer ($\sigma_{m0} \leq \sigma_{my}$) and the wear depths per unit of time of the fillers and matrix are equal, the deforming rates of the fillers and matrix under friction should also be equal. In this situation the distribution of stress is uniform, which is called the "equal pressure" state,⁹ and no load-supporting action of fillers exists (i.e., $\sigma_f =$

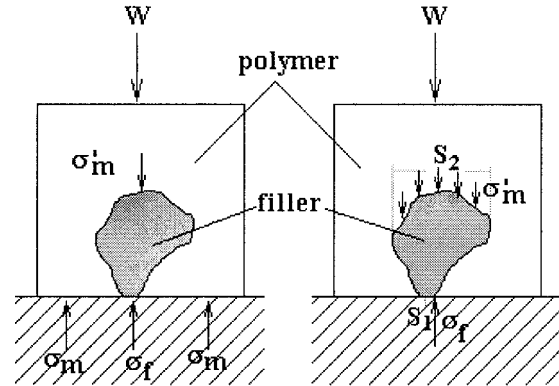


Figure 9 The mechanical analysis of the load-supporting action of filler in the polymer matrix.

$\sigma_m = \sigma_{m0}$). These phenomena are seldom observed because the wear rate of a hard filler is often lower than that of a matrix. However, when the wear rate of fillers is higher than that of the matrix, the fillers will possess the load-supporting action. In this case the mechanism of preferential load-supporting action of fillers is explained as in the following section.

In the high compressive stress situation there is a high load and different wear rates. When the σ_{m0} is higher than the compressive yield strengths of the polymer ($\sigma_{m0} > \sigma_{my}$) or the fillers have greater wear resistance than the matrix, the matrix will be removed more rapidly, leaving a sliding surface that is enriched with the fillers of greater wear resistance. The distribution of compressive stress is not uniform, and the hard fillers will possess the preferential load-supporting action. In this situation the σ_m is approximately equal to the compressive yield stress of the polymer matrix. Although σ'_m may be smaller than the σ_m , σ_f should be much higher than σ_m because S_2 is much bigger than S_1 . The relation among them can be expressed as

$$\sigma_{m0} = \delta_f \frac{S_2}{S_1} \sigma'_m + \delta_m \sigma_m \quad (3)$$

where δ_f and δ_m are the fractions of fillers and polymer matrix exposed on the contact surface, respectively, and $\delta_f + \delta_m = 1$. For the fillers randomly embedded in the polymer matrix, the S_1 and S_2 may be increased with the wear. Especially for the accumulation of fillers in the axial direction of loading, the S_2 will increase significantly because the loading stress is decreased effectively by dispersing the stress from one particle on the contact surface to the particles connected with it.

The above mechanical analysis is mainly aimed at the influence of compressive stress without discussing the influence of the wear rates of the fillers and matrix in detail. It should be pointed out that the compressive deformations, wear depths per unit time of fillers, and matrix are the most important factors influencing the preferential load-supporting action of fillers. In particular, when the wear rate of a filler under a pressure of σ_f is still lower than that of a polymer matrix under the pressure of σ_m , the hard filler particles may "flow" along the loading direction until the same rates of wear depth per unit time can be reached and the applied normal load is also balanced. To reach this dynamic equilibrium, hard filler particles may accumulate on the wear surface of polymer composites to increase δ_f (filler fraction) and decrease δ_m (polymer fraction). In addition, as a result of flow, the hard particles may be connected with each other at near the subsurface of the polymer composites. The S_2 area of connected particles may be obviously increased. In this case, much higher stress on the filler fraction (σ_f) may be allowed to achieve the equal wear mentioned by Axen and Jacobson.⁹

Because there may have been many wear mechanisms, such as abrasive wear, adhesive wear, and fatigue wear, that existed simultaneously under the conditions of this study, no obvious antiwear action of hard metallic fillers was observed in this work. This phenomenon strongly supported the viewpoint proposed by Briscoe and coworkers that the tribological action of filler in the composite was limited by the experimental conditions.¹

CONCLUSIONS

The tribological actions of metallic fillers in PTFE-based composites were strongly restricted to the friction and wear conditions. Under a heavy load and reciprocating motion with small amplitude, two obviously distinct friction stages were observed, which may have corresponded to the contact status of the substrate with the counterpart. The accumulation of fillers on the contact surface and the extruding wear of PTFE were found in this work.

The actions of metallic fillers on the transfer film formation should be divided into two aspects: the hard filler exposed on the wear surface of the composite can scrape the polymer transfer film on the counterpart surface; and the transfer film composed of metallic filler can mechanically hold

the polymer forming the composite transfer film, because its mechanical strength is higher than that of the polymer matrix. The transfer and the accumulation of fillers can actually be considered to be a mutual assimilating process of the pairs. As the result of the assimilation, with the formation of the composite transfer film, the nature of the counterpart steel ball surface gradually approached that of the composite; for the composite, the accumulation of metallic fillers changed the properties of the composite surface, such as the surface hardness and composition, to approximate that of the steel ball counterface with the transfer film. The transfer film formation process was well described with a physical model.

The mechanism of the preferential load-supporting action of the hard filler was also reanalyzed. Some suggestions on the load-supporting actions of hard fillers, which were different with the viewpoints proposed by Tanaka and Kawakami,¹³ were briefly discussed. An important reason for the load-supporting actions of fillers was the ratio of S_2/S_1 .

The authors gratefully acknowledged the financial support from the National Natural Science Foundation of China and the High-Tech R & D Bureau of the Chinese Academy of Sciences.

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