

Study on Wear and Friction Behavior of Graphite Flake-Filled PTFE Composites

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ABSTRACT: The wear rate and coefficient of friction for graphite flake (GF)-filled polytetrafluoroethylene (PTFE) composites were evaluated on a pin-on-disk wear tester under dry conditions. Scanning electron microscopy showed significant reduction in the abrasive wear of the composites. The wear rates of 5 and 10 wt % GF composites were reduced by more than 22 and 245 times, respectively, at sliding speed of 1 m/s. With increasing sliding distance from 1 to 8 km, the wear rate of pure PTFE decreased by 1.4 times whereas that of composites, it decreased up to three times. The significant decrease in wear rate and coefficient of friction might be attributed to the formation of a thin and tenacious transfer film on the counter-surface. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2012

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INTRODUCTION

Polytetrafluoroethylene (PTFE) is a semi-crystalline polymer which exhibits a crystalline melting point of 342°C, low coefficient of friction, good toughness, excellent chemical resistance, and thermal stability. Owing to these properties, it is widely used for bearings and sliding applications. However, it exhibits high wear rate ($\sim 740 \times 10^{-6} \text{ mm}^3/\text{Nm}$) under dry sliding conditions compared to other polymers.^{1–3} Its wear rate has been reduced about two-four orders of magnitudes with the addition of Al_2O_3 ,⁴ ZnO ,⁵ MoS_2 , SiC , SiO_2 ,⁶ ZrO_2 ,⁷ Al ,⁸ TiO_2 ,⁹ Cu , Pb , Ni ,¹⁰ CNE ,¹¹ carbon fiber,¹² glass fiber,¹³ PEEK,¹⁴ and bronze¹⁵ in the PTFE matrix. The decrease in wear rate depends on several factors such as shape, size, type, and crystal structure of the fillers, degree of dispersion of fillers in the matrices, and operating conditions.^{16–19} Nevertheless, the nature of interface between the sample and counter-surface also plays an important role in reducing the wear rate and coefficient of friction.⁸

Sawyer et al.⁴ investigated that the wear rate of PTFE composites decreased with increasing nano- Al_2O_3 content, but the coefficient of frictions of composites increased. In contrast, according to Li et al.,⁵ the coefficient of friction for PTFE/ ZnO was decreased compared to that of PTFE. Moreover, the addition of inorganic hard particles increases the density of PTFE composites. Furthermore, hard micron sized particles tend to abrade the counter-surface, hence prevents the formation of

good quality transfer films and sometimes lead to third body wear of the composites. To avoid third body wear of the components, self-lubricated filler like graphite powder ($7 \mu\text{m}$) was filled into the PTFE matrix and a decrease in wear rate up to 3.8 times was found.⁸ Such low improvement in wear resistance (i.e., inverse of wear rate) is not sufficient for the application. There is need to improve more than two orders of magnitude wear resistance without increasing density of the composites.

In view of this, in this study, we report a significant decrease in wear rate (i.e., up to 245 times) for the 5–10 wt % graphite flake (GF)-filled PTFE composites. These composites were prepared using a simple method, that is, by dispersing commercial GF powder in the PTFE matrix via solvent suspension method followed by cold pressing and then sintered below melting temperature of PTFE.

EXPERIMENTAL

Materials

Commercial polytetrafluoroethylene (PTFE) purchased from a local supplier was used as matrix. Its average particle size was $10 \mu\text{m}$. GF purchased from Sigma-Aldrich Co., USA was used as filler without treatment. As shown in Figure 1, the GF have widths ranging from few μm to tens of μm and thicknesses less than $1 \mu\text{m}$, that is, its aspect ratio is comparatively high. Tetrahydrofuran (THF) purchased from a commercial source was used as solvent medium for blending PTFE and GF.

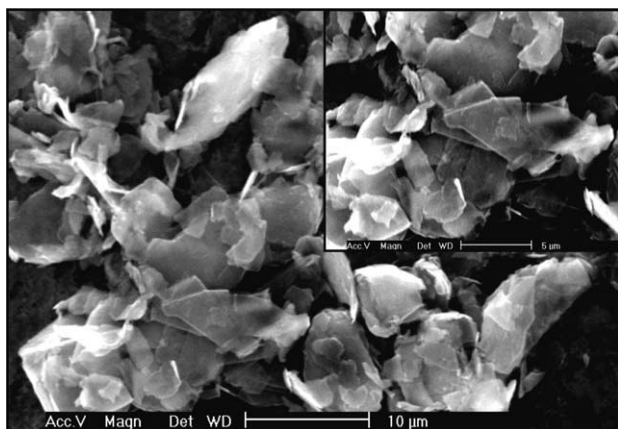


Figure 1. SEM image of GF powder. Inset shows image at higher magnification (scale bar; 5 μm).

Preparation of PTFE/GF Composites

Both PTFE and GF powders were dried in a vacuum oven at 120°C for 2 h. Then, an appropriate weight of both PTFE and GF powders were mixed together in a THF solvent using ultrasonic bath and magnetic stirrer. After proper mixing, the solvent was evaporated and then, dried powder was filled into a steel die. The composites containing 0–10 wt % GF were prepared using compression molding machine at room temperature under 60 MPa. Then, the samples were sintered in the vacuum oven at 280°C for 18 h. The diameter and the height of the samples were 13 and 25 mm, respectively. Figure 2(a–c) showed the optical microcopy images of the composites containing 2, 5, and 10 wt % GF in the PTFE matrix, respectively. It can be seen that the GF are almost uniformly dispersed in the PTFE matrix. However, as the content of the GF in the PTFE matrix increased from 2 to 10 wt %, the GF aggregates are formed as shown in Figure 2(b,c). This is due to the decrease in the inter-particle distances between the GF particles with increasing GF content in the matrix.

Characterization of Samples

The specific wear rate and the coefficient of friction of the samples were determined on a pin-on-disk wear tester at sliding speed of 1.0 m/s and normal load of 25 N. The counter surface was made of stainless steel. The tests were conducted for the sliding distances varying from 1 to 8 km at 30°C. The surface of the counter-surface was abraded with a 600 grit emery paper before testing. The surfaces of counter-surface and the pin were cleaned thoroughly with acetone dipped cotton. The specific wear rate of the samples was calculated using the equation:

$$\text{Specific wear rate } (k) = \Delta m / (\rho FL) \text{ (mm}^3/\text{Nm)} \quad (1)$$

where, Δm is the mass loss (g), ρ is the density of sample (g/cc), F is the normal load (Newton), and L is the sliding distance (m).

The coefficient of friction was calculated from the ratio of the force applied to the normal force on the sample. Scanning electron microscopy (SEM) (JOEL: JSM 6360A) was used to examine the worn surface of the composite pins and wear debris.

The samples were coated with platinum to avoid their charging during analysis.

RESULTS AND DISCUSSION

Wear and Friction Tests

Figure 3 shows the specific wear rate as a function of GF content in the PTFE matrix. It can be seen that the specific wear rate of

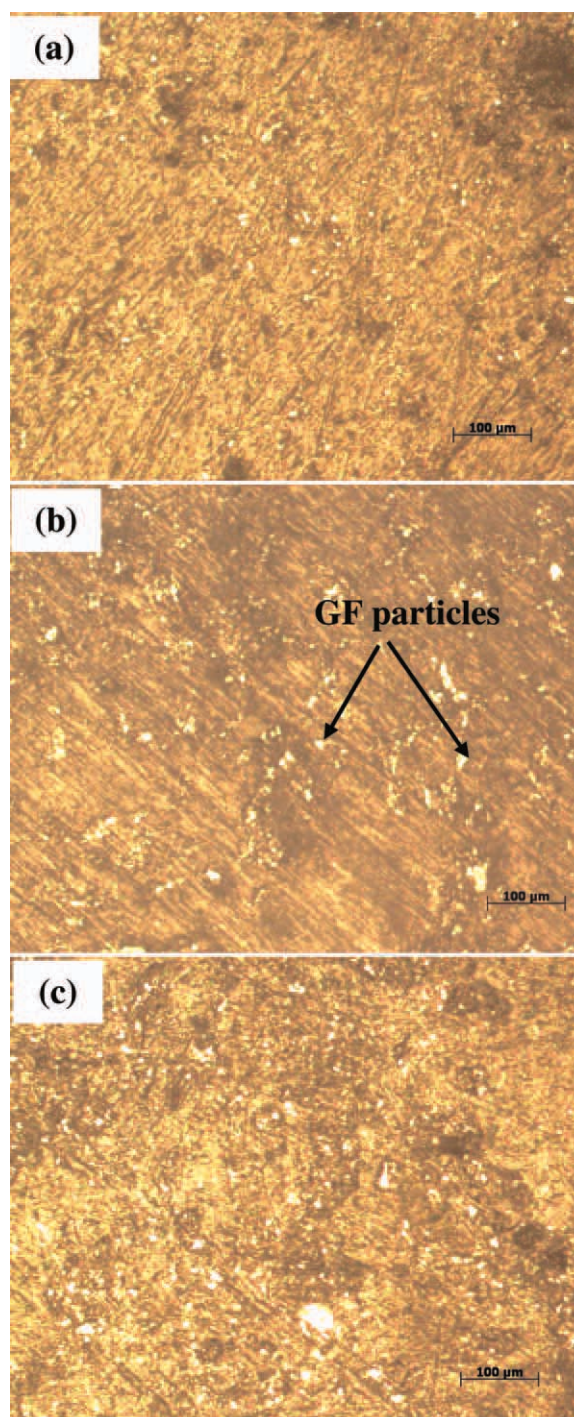


Figure 2. Optical microscopy images of PTFE/GF composites containing (a) 2 wt % GF, (b) 5 wt % GF, and (c) 10 wt % GF. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

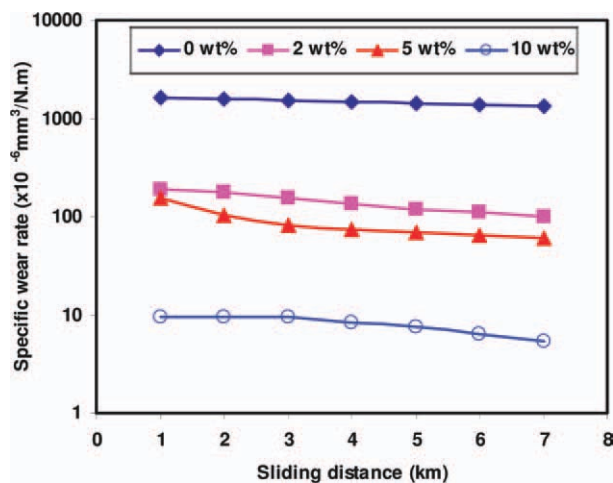


Figure 3. Specific wear rate of PTFE/GF composites as a function of sliding distances (load: 25 N, speed: 1 m/s). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

pure PTFE decreases slightly with increasing sliding distance, that is, it decreases from $1.64 \times 10^{-3} \text{ mm}^3/\text{Nm}$ (after 1 km) to $1.17 \times 10^{-3} \text{ mm}^3/\text{Nm}$ (after 8 km). In case of composites, for a given wt% GF, the specific wear rate decreases sharply with increasing sliding distance. After the sliding distances of 1 and 8 km, 5 wt % composite exhibits wear rates of 152.7 and $52.6 \times 10^{-6} \text{ mm}^3/\text{Nm}$, respectively, whereas 10 wt % composite exhibits 9.5 and $4.7 \times 10^{-6} \text{ mm}^3/\text{Nm}$, respectively (Figure 4). Compared to pure PTFE, the maximum improvement in wear resistance (inverse of wear rate) for 2, 5, and 10 wt % composites is about 12.6 \times , 22 \times , and 245 \times , respectively. The wear rate of 10 wt % PTFE/GF composite is close to that of 20 vol % (\sim 20 wt %) carbon nanotube (CNT)-filled PTFE composite.²⁰ In contrast, the wear resistance of 15 vol % (\sim 15 wt %) graphite filled PTFE composite was increased by 3.8×10^8 and that of 10 wt % nano- Al_2O_3 filled PTFE composite by about $50 \times$.⁴ Similarly, Li et al. reported $\sim 3 \times$ increase in wear resistance for 10 wt % micron sized graphite filled PTFE composite.¹⁶ Compared to the literature values, the

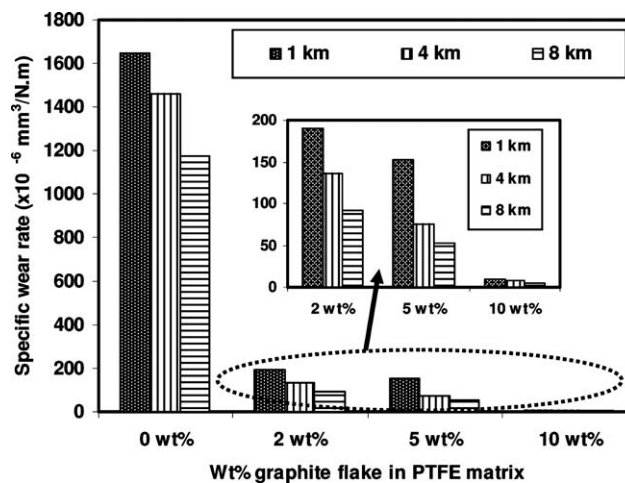


Figure 4. Specific wear rate of PTFE/GF composites as a function of GF content after the sliding distances of 1, 4, and 8 km (load: 25 N, speed: 1 m/s).

significant reduction in the wear rate of PTFE/GF composites may be attributed to the higher aspect ratio of GF as confirmed from Figure 1 and its better dispersion in the matrix, which probably results in the formation of a thin and adherent transfer film on the counter-surface (Figure 5). It can be clearly seen from Figure 5(a) that relatively large area of the wear track is free from PTFE layers. Moreover, wear debris of PTFE has larger sizes which are rested on the counter-surface but away from the wear track indicating poor adhesion with the counter-surface. Figure 5(b) shows that large area of the wear track is covered with a thin transfer film and no loose debris away from the wear track indicating good transfer film on the wear track. Probably, presence of GF helps in bonding the transfer film with the counter-surface. Secondly, higher surface energy of graphite than that of PTFE also helps in making good bonding with the counter-surface.²¹

Figure 6 shows the coefficient of friction of PTFE and its composites as a function of sliding distance. The coefficient of friction of pure PTFE decreases with increasing sliding distance, that is, it varies from 0.28 at 1 km to 0.24 at 8 km. The high

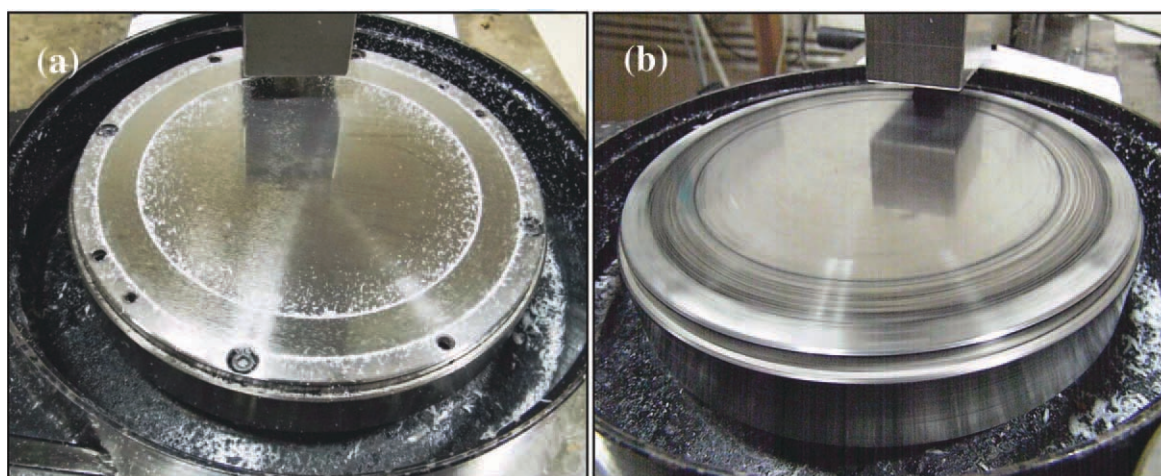


Figure 5. Digital camera photo of wear tracks of (a) pure PTFE and (b) 2 wt % GF-filled PTFE composites after a sliding distance of 8 km. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

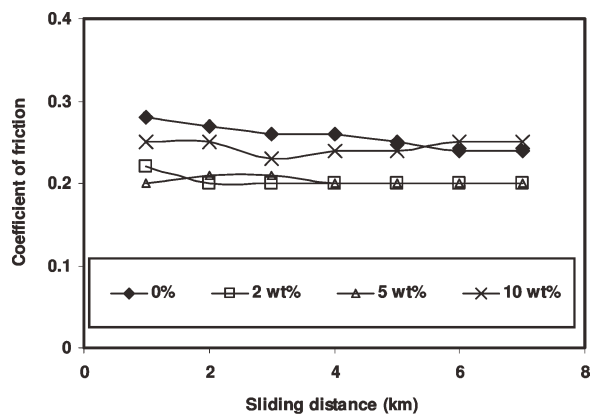


Figure 6. Coefficient of friction of PTFE/GF composites as a function of sliding distance.

coefficient of friction may be due to the abrasive wear between the polymer pin and the counter-surface. The abrasive wear resulted in due to the microploughing action leaving deep grooves in the polymer surface as confirmed from the SEM images (Figure 7). In case of composites, the coefficient of friction decreases slightly with increasing GF content. The lowest coefficient of friction was 0.20 for 2 and 5 wt % composite and thereafter, it increases slightly (but lower than the pure PTFE) with further increasing content of GF in the matrix. The slight increase in coefficient of friction may probably be due to the relatively inferior quality of transfer film. It is well known that as the content of GF increases in the matrix, the GF particles have a tendency to the formation of aggregates which hinder the formation of thin and tenacious transfer film. In contrast, an increase in coefficient of friction was reported for PTFE/ Al_2O_3 ⁴ and PTFE/babbit composites.

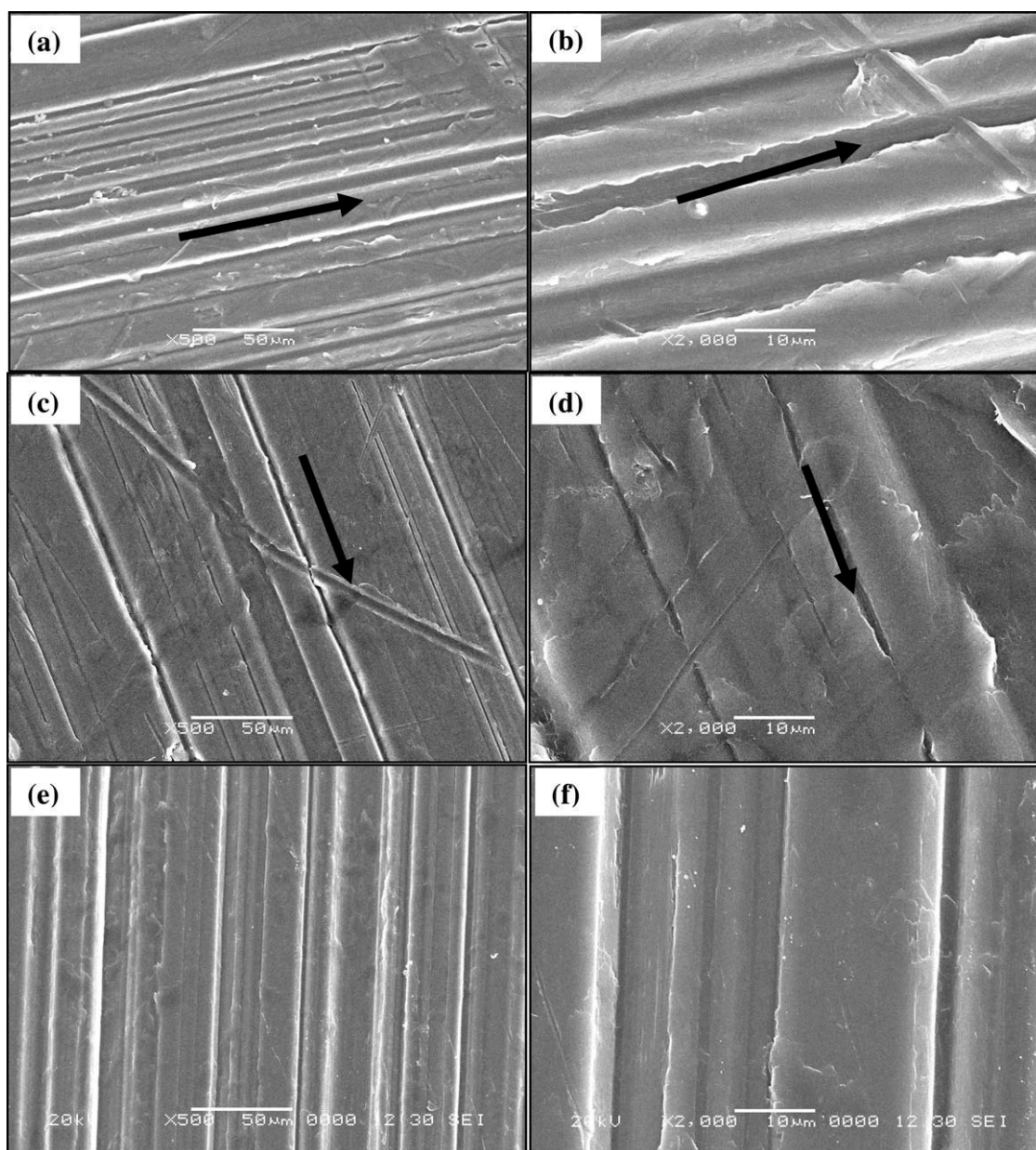


Figure 7. SEM images of (a,b) pure PTFE, (c,d) 2 wt % GF, and (e,f) 5 wt % GF composites at magnification of $\times 500$ (a,c,e) and $\times 2000$ (b,d,f), respectively. Arrow shows the sliding direction.

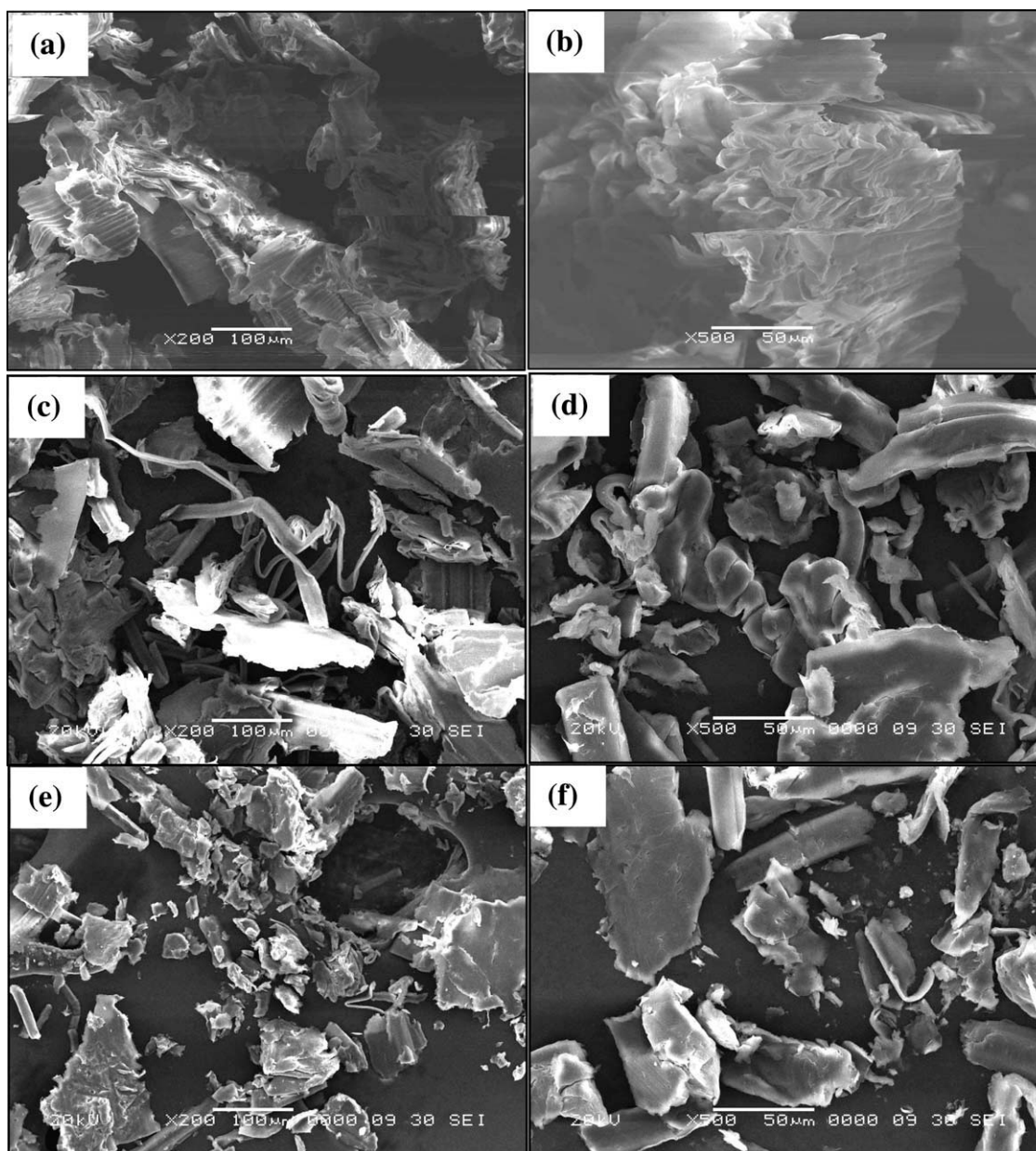


Figure 8. SEM images of debris of (a,b) pure PTFE, (c,d) 2 wt % GF, and (e,f) 5 wt % GF composites at magnification of $\times 200$ (a,c,e) and $\times 500$ (b,d,f), respectively.

SEM Study on Worn Surface and Wear Debris

Figure 7(a–f) show the SEM images of worn surface of pure PTFE and its composite pins. Figure 7(a,b) show the SEM of worn surface of pure PTFE at $\times 500$ and $\times 2000$, respectively. They reveal the formation of grooves on the pin surface. The asperities of the counter-surface act as microblades which results in larger sized plough marks on the pin surface. This indicates that the abrasive wear is the main wear mechanism for the wear of the pure PTFE. Figure 7(c,d) show the SEM images of worn surface of 2 wt % composite which indicate relatively less intense sign of apparently plucked and ploughed marks compared to pure PTFE. Figure 7(e,f) are the SEM images of 5 wt % composite and reveal the formation of smooth scar on the pin surface. This indicates that adhesion is the main dominant wear mechanism for composites.

The decrease in the depth of scratches may probably be attributed to the formation of stable, adhesive and intact transfer film on the counter-surface.^{22–24} The addition of GF filler in the matrix increases the surface energy that results in strong adhesion of polymer layer to the counter-surface. As the adhesive wear increases the wear product (i.e., debris) of the composite gets accumulated in the grooves of the counter-surface and covers the deep asperities. This results in the formation of adherent film on the counter-surface and thus decreases wear rate.

Figure 8(a–f) show the SEM images of wear debris of pure PTFE, 2 and 5 wt % composite. Figure 8(a,b) show the wear debris of pure PTFE indicating that the pure PTFE generates debris containing lumpy slabs with thickness more than $150 \mu\text{m}$

and each slab seems to have several loosely bounded PTFE sheets. Such high thickness of debris may be attributed to the higher sliding speed (i.e., 1 m/s) which in-turn causes higher coefficient of friction and thus, lumpy slabs.²³ These large debris are unable to fill up the grooves and hence, they pileup during repeated sliding and subsequently discarded as debris. Figure 8(c–f) shows that the size of wear debris decreases with increasing content of GF in the matrix. For composites, the size of wear debris is much smaller than that of pure PTFE; they easily fill up the counter-surface grooves and served as spacers and preventing the direct contact between the two mating surfaces. Therefore, there is significant reduction in wear rate and coefficient of friction for the composites. The similar nature of debris was reported for PTFE/CNT composites.²⁰

CONCLUSION

Specific wear rate of pure PTFE decreased marginally whereas that of composites decreased significantly with increasing sliding distance. This is due to the formation of the thin and tenacious transfer film of the composites on the counter surface. The addition of 10 wt % GF in the PTFE matrix decreases specific wear rate by more than 245× under 25 N load. This is really a significant improvement in the wear resistance of the composites. There is marginal decrease in the coefficient of friction with increasing content of GF in the matrix. Compared to pure PTFE, composites showed stable coefficient of friction with increasing sliding distance.

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