Enhanced thermal conductivity and wear resistance of polytetrafluoroethylene composites through boron nitride and zinc oxide hybrid fillers

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ABSTRACT: In this study, the thermal conductivity and wear resistance of the polytetrafluoroethylene (PTFE)/boron nitride (BN), PTFE/zinc oxide (ZnO), PTFE/tetra-needle-shaped zinc oxide whiskers (T-ZnO), and PTFE/hybrid filler composites were investigated. Moreover, hot-press molding was used to prepare the composites, and scanning electron microscopy was used to observe the morphology of the fillers and the friction interface of the composites. The results show that continuous thermally conductive paths could be formed in the PTFE/hybrid fillers (T-ZnO and BN) composites so that the thermal conductivity of the PTFE was improved through addition of the hybrid fillers. Meanwhile, the synergistic effects of the hybrid fillers were useful for reducing the wear rate of the composites. In addition, for the pure PTFE, abrasive and adhesive wear was found. Compared to the worn surface of the pure PTFE, the worn surface of the PTFE composites filled with ZnO, T-ZnO, BN, and hybrid fillers presented much smoother surfaces, and slighter ploughing occurred. Therefore, the hybrid fillers improved not only the thermal conductivity but also the wear resistance of the PTFE composites. The data obtained in this study contributed to the construction of a technical foundation for the preparation of composites with a high thermal conductivity and wear resistance. © 2015 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2015**, *132*, 42302.

INTRODUCTION

Polytetrafluoroethylene (PTFE) is widely used in industry as antifrictional wear-resistant materials because of its unique properties, in particular its self-lubricating capacity, high chemical inertness, and high-temperature capability.[1,2] However, PTFE exhibits severe creep deformation and poor wear and abrasion resistance; this leads to early failure and leakage during use.[3,4] To solve these problems, many investigations have been done. Earlier studies showed that the filling of some inorganic particles and fibers, such as graphite, copper, molybdenum disulfide, glass and carbon fibers, zinc oxide (ZnO), carbon black, and aluminum oxide, can significantly improve the wear rate of PTFE composites and cause the abrasive properties to reduce the life of precise friction units.[5–10]

It is well known that the material properties of polymers are strongly temperature-dependent. According to previous studies, it was found that the wear mechanism of polymers was changed at different temperatures. Abrasive wear was observed at low temperatures, whereas at high temperatures, adhesive wear dominates.[11–13]

The tribological performances of polymers depend significantly on the temperature at the friction contact, especially for polymers with low heat conductivity. Therefore, to improve the thermal diffusion at the friction interface, it is necessary to enhance the thermal conductivity of the polymers. Generally, to improve the thermal conductivity of the polymers, many kinds of thermally conductive fillers have been introduced into the polymer matrix; these include aluminum nitride,[14–17] silicon nitride (Si_3N_4),[18,19] alumina,[20] and boron nitride (BN).[21–23] The thermal conductivity of the polymer composites mainly depends on the thermally conductive chains or networks formed by fillers.

Yu *et al.*[24] found that aluminum nitride particles surrounding the polystyrene matrix particles as a special dispersion state of the filler in the composites was very helpful for improving the thermal conductivity of the composites. Moreover, Zhou *et al.*[25] prepared a novel thermally conductive composite from a mixture of Si_3N_4 particles and an ultra-high-molecular-weight polyethylene/linear low-density polyethylene blend. With hot-press molding, the Si_3N_4 particles could form a continuously connected dispersion state that acted as the dominant thermally conductive pathway through the polymer matrix. According to previous investigations,[24,26] the particle mixture can lead to a networked dispersion of filler in the matrix, that is, filler

particles surrounding matrix particles. It is possible to improve the thermal conductivity of a composite at a low filler content with this kind of networked dispersion of filler rather than a melted mixture.[27] Therefore, to obtain a maximum thermal conductivity at relatively low filler content and to minimize the adverse effect of the filler on the other properties of the composites, powder mixing was used often in this study.

In this study, to enhance the thermal conductivity of the PTFE composites, BN, ZnO, and ZnO whiskers were selected as the thermally conductive fillers. Meanwhile, to improve the wear resistance of the PTFE composites, hybrid fillers (ZnO and BN) were used. Moreover, hot-press molding was also use to prepare the composites. Scanning electron microscopy (SEM) was used to observe the dispersed morphology of the fillers and the friction interface of the composites. The thermal conductivity, wear resistance of the PTFE composites, and crystallinity of the PTFE were also investigated. The data obtained in this study contributes to the construction of a technical foundation for the preparation of composites with a high thermal conductivity and wear resistance.

EXPERIMENTAL

Materials

PTFE powder (FR104-4), with an average particle size of approximately 50 μm, which purchased from Shanghai 3F New Materials Co., Ltd., was used as the matrix resin for the composites. BN, with a particle size of 3–5 μm, was purchased from Shandong Pengcheng Special Ceramics Co., Ltd. Tetra-needle-shaped zinc oxide whiskers (T-ZnO) were obtained from Chengdu National Chiao Tung University Jingyu Co., Ltd. The length of each needle of T-ZnO was about 10–15 μm, and the diameter of each needle at the growth point was about 1–5 μm. ZnO, with a particle size of 2–3 μm, was purchased from Sichuan Apollo Solar Energy Technology Co., Ltd.

Specimen Preparation

BN, ZnO, and T-ZnO were first dried in oven at 80°C for 12 h. After that, PTFE mixed with BN, ZnO, and T-ZnO in a high-speed mixer (GH-10DY, Beijing Plastic Machinery Factory). Then, the mixture was compressed at 25 MPa of pressure for 5 min at room temperature. Finally, the mold was sintered at 380°C for 4 h in a high-temperature oven and carefully cooled down to room temperature under a pressure of 50 MPa.

Morphological Observation

SEM was used to examine the dispersed morphology of the fillers and the frictional interface of the composites. The composites were quenched in liquid nitrogen and cryofractured, and then, the sample measurements were coated with gold and observed with a Hitachi S3400 + EDX scanning electron microscope (Japan) at an accelerating voltage of 20 kV.

Thermal Conductivity Measurement

To measure the thermal conductivity of the PTFE composites, a thermal constants analyzer (Hot Disk thermal conductivity detector 1500) produced by Hot Disk Co. (Sweden) was used. The thermal constants analyzer used the transient plane source (TPS) method to measure the thermal conductivity of the materials.[28] The TPS method used the Fourier law of heat conduc-
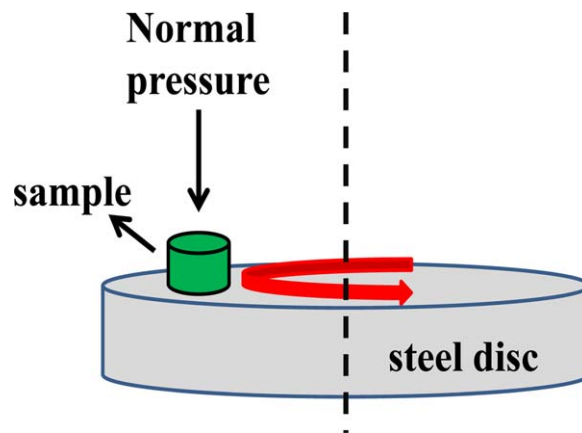

Figure 1. Contact geometry of the friction pairs between the PTFE composites and the steel disc. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

tion as its fundamental principle for measuring. The uncertainties of the TPS methods were about 5%.[29]

Wear Testing

The wear behavior of the PTFE composites was examined on a pin-on-disc tester. The contact geometry of the friction pairs is shown in Figure 1. A steel disc (AISI1045 steel) with a 40 mm diameter and 10 mm thickness was applied as the counter friction part against the PTFE composites. The sliding velocity was 0.04 m/s, and the normal contact load was 120 N. The contact pressure was 6 MPa during the pin-on-disc wear test. The roughnesses of the steel disk and specimens were 1000 and 500, respectively. The wear tests run for 4 h in the ambient atmosphere with relative humidities of 50–60%. The worn surfaces of the PTFE composites were observed by SEM. The wear loss mass of the PTFE composite pin was measured by the wear mass loss, which was weighed on an electronic balance with an accuracy of 0.01 mg. The wear rate of the PTFE composites was calculated according to the following formula:

$$K = \Delta m / FL\rho$$

where K is the wear rate of the PTFE composites (mm^3/N m), Δm is the wear loss mass (g), ρ is the density of the PTFE composites (g/mm^3), and F and L are the normal load (N) and sliding distance (m) of wear testing, respectively.

Wide-Angle X-ray Diffraction (WAXD)

The crystalline structure of the composites was investigated with WAXD (PaNalytical X'Pert PRO diffractometer with Ni-filtered Cu Kα radiation, PaNalytical, The Netherlands). The continuous scanning angle range used in this study was from 10 to 60° at 40 kV and 40 mA.

RESULTS AND DISCUSSION

Thermal Conductivity of the Composites

Figure 2 shows the effect of different kinds of fillers on the thermal conductivity of the PTFE composites. It was clear that the thermal conductivity of the composites increased with incensement of the filler content. At 5 wt % filler content, the composites prepared with the three kinds of fillers showed a significantly different thermal conductivity. Compared to the
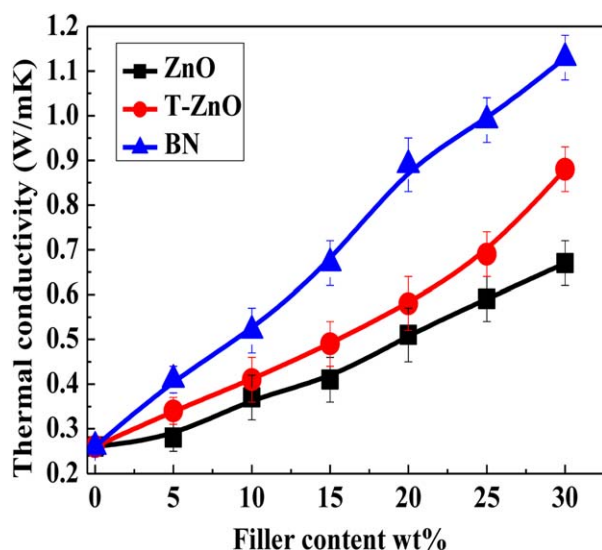
Figure 2. Effects of different kinds of fillers on the thermal conductivity of the PTFE composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figure 3. Effects of the hybrid fillers on the thermal conductivity of the PTFE composites. The total filler content was 30 wt %. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

PTFE/ZnO and PTFE/T-ZnO composites, the PTFE/BN composites showed the highest thermal conductivity, Moreover, when the fillers content continued to increase, the fillers contacted each other, and this resulted in the formation of the thermally conductive paths between the fillers and fillers. Therefore, the thermal conductivity of the composites increased significantly when the loading of the fillers was high. According to Figure 2, compared to the ZnO particles, the T-ZnO particles was more helpful for enhancing the thermal conductivity of the PTFE; this indicated that the morphology of the ZnO particles had a strong influence on the thermally conductive networks in the PTFE matrix. Moreover, compared to the PTFE/T-ZnO and PTFE/ZnO, the PTFE/BN composites showed the highest thermal conductivity. That was to say, to improve the thermal conductivity of PTFE, the addition of BN into the PTFE matrix was effective. In this study, BN and T-ZnO particles were used as hybrid fillers with an attempt to maximize the abundance of conductive paths. As shown in Figure 3, the hybrid filler (total filler content = 30 wt %) filled PTFE displayed a higher thermal conductivity than that filled with the thermally conductive filler alone at the same filler content; in particular, the thermal conductivity of the composites reached 1.38 W/mK when the T-ZnO content was 3 wt % in the hybrid fillers. Furthermore, the thermal conductivity of the composites also reached 1.22 W/mK when the ZnO content was 5 wt % in the hybrid fillers. Because of the relatively low thermal conductivity of ZnO filler in comparison with the BN filler, the obtained high thermal conductivity of the composites containing the hybrid filler successfully demonstrated the usefulness of the concept of a synergistic effect of hybrid filler in enhancing the thermal conductivity of the composites for a given filler loading.

Prediction of the Thermal Conductivity of the PTFE Composites

The Agari model considers the effect of the polymer structure and dispersion state of the filler on the thermal conductivity of
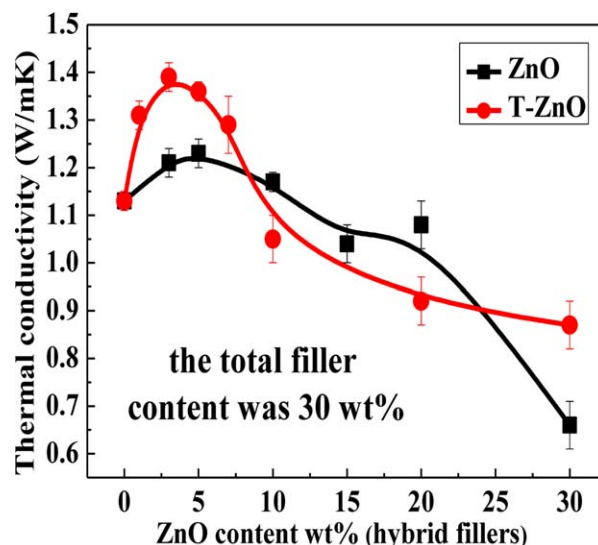
the composites through introduction factors (C_1 and C_2) as follows.[30]

$$\log k_c = V_f C_2 \log k_f + (1 - V_f) \log(C_1 k_m) \quad (1)$$

where V_f is the volume fraction of the filler, k_c is the thermal conductivity of the composite, k_f is the thermal conductivity of the filler, k_m is the thermal conductivity of matrix, C_1 is a factor related to the polymer structure, and C_2 is a factor related to the formation of the thermally conductive network of the filler. The thermal conductivity of the composites can be expressed well by the Agari model, as shown in Figure 4. $C1_1$ and $C2_2$ obtained by the data fitting are shown in Table I. According to this table, for the PTFE/ZnO and PTFE/T-ZnO composites, C_1 did not change obviously, whereas C_2 changed greatly in comparison with C_1. Compared to C_1 (PTFE/ZnO) and C_1 (PTFE/T-ZnO), C_1 (PTFE/BN) changed obviously; this indicated that
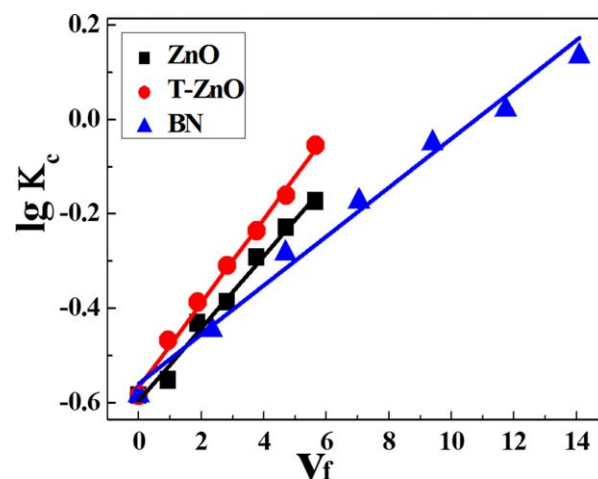

Figure 4. Thermal conductivity of the PTFE composites expressed with the Agari model. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table I. C_1 and C_2 Values Obtained with Agari's Model for the PTFE Composites, and the R was the Related Coefficient

Sample	C_1	C_2	R
PTFE/ZnO	3.793	0.0474	0.987
PTFE/T-ZnO	3.796	0.0663	0.991
PTFE/BN	3.982	0.0312	0.989

the effect of BN on the PTFE structure was different from ZnO and T-ZnO. Moreover, C_2 (PTFE/ZnO) was less than C_2 (PTFE/T-ZnO); this indicated that PTFE/T-ZnO was more helpful in forming the thermally conductive chains in the polymer matrix than the PTFE/ZnO composites. In addition, compared to C_2 of PTFE/ZnO and C_2 of PTFE/T-ZnO, C_2 of PTFE/BN was the smallest; this indicated that it was difficult for BN to form a thermally conductive network between the BN and BN fillers. However, according to Agari *et al.*,[30] the thermal conductivity of the composites was decided by the crystallinity of the polymer and network between fillers. Therefore, it was necessary to investigate the crystallinity of the PTFE in the composites in a later study.

Morphologies of the Fillers and PTFE Composites

Figure 5 shows the morphologies of the fillers and the thermally conductive PTFE composites. According to Figure 5(a–c), the morphology of T-ZnO was a tetra-needle-shaped whisker, that of ZnO was a sphere, and that of BN was a sheet structure, which liked graphite. Figure 5(d) shows the T-ZnO and BN hybrid fillers. To obtain a high thermal conductivity in the PTFE composites, hot-press molding was used for the preparation of the PTFE composites, and thermally conductive paths were observed through SEM. According to Figure 5(e), obvious thermally conductive paths were observed in the PTFE/hybrid filler (T-ZnO + BN) composites. Similarly, the clear and continuous thermally conductive paths were also observed [Figure 5(f)] for the PTFE/BN composites. According to Figure 5(e′), for the PTFE/hybrid filler composites, in addition to the thermally conductive paths formed between BN and BN particles, we also observed that T-ZnO and BN could contact each other to form thermally conductive paths. However, we only observed thermally conductive paths formed between BN and BN particles [Figure 5(f′)] for the PTFE/BN composites; this indicated that the more thermally conductive paths could be formed in the PTFE/hybrid filler composites so that the thermal
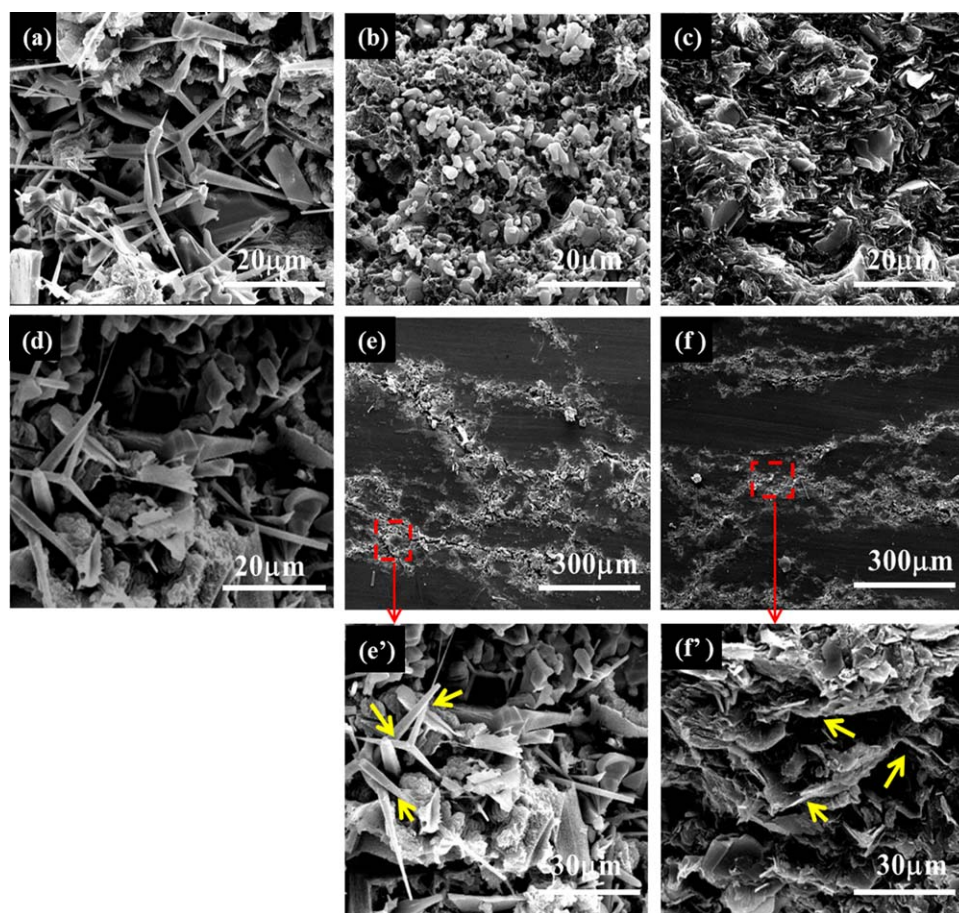

Figure 5. Morphologies of the fillers and the thermally conductive paths of the PTFE composites: (a) morphology of T-ZnO, (b) morphology of ZnO, (c) morphology of BN, (d) morphology of the hybrid fillers (T-ZnO/BN = 3 : 27), (e) thermally conductive paths of the PTFE/hybrid filler composite, (f) thermally conductive paths of the PTFE/BN composite, (e′) local enlarged image of the PTFE/hybrid filler composite, and (f′) local enlarged image of the PTFE/BN composite. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
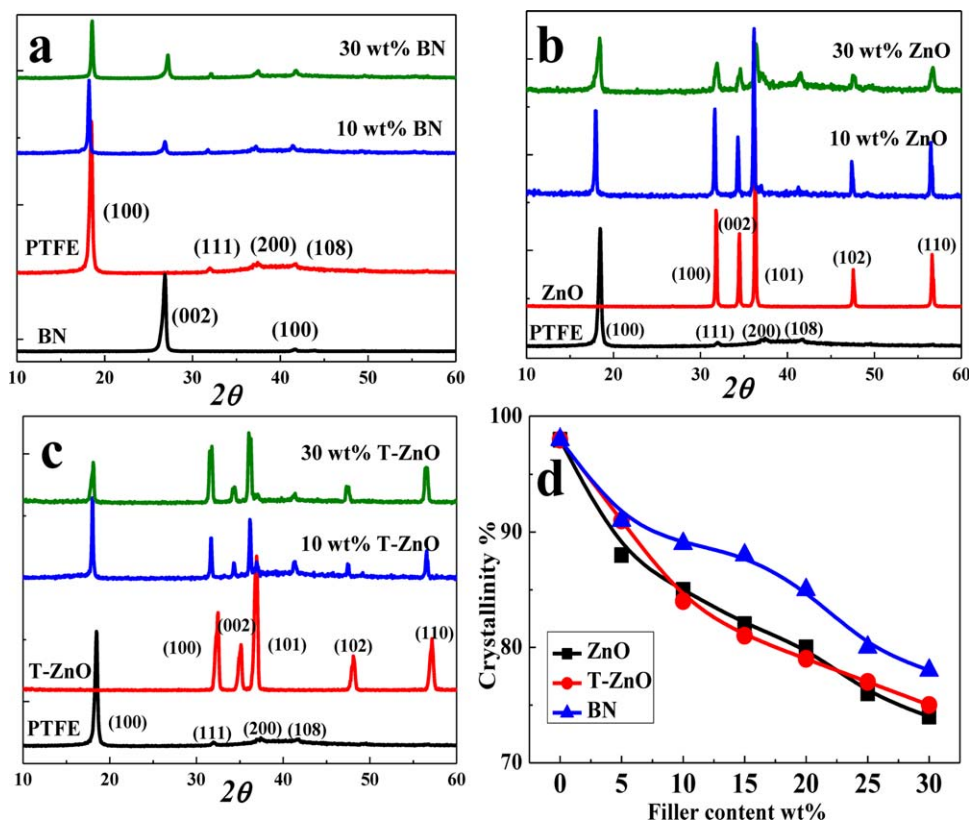
Figure 6. WAXD profiles showing the crystalline structures and crystallinity of the PTFE composites: (a) PTFE/BN composites, (b) PTFE/ZnO composites, (c) PTFE/T-ZnO composites, and (d) crystallinity of PTFE in its composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

conductivity of PTFE could be improved through the addition of the hybrid fillers.

WAXD

To understand the crystallization behavior of the PTFE in the composites, the crystalline structure was characterized with WAXD, and the results are shown in Figure 6. The results of the pure PTFE, T-ZnO, ZnO, and BN are also shown for comparison. As shown in Figure 6(a), the pure PTFE had four intense diffraction peaks at $2\theta = 18.4$, 32.1, 37.3, and 41.4°; these corresponded corresponding to the (100), (111), (200), and (108) crystal planes, respectively. For BN, two peaks appeared at $2\theta = 26.8$ and 41.8° and corresponded to the (002) and (100) crystal planes. In addition to the stacked peaks of PTFE and BN, a new peak was not found when BN was added to PTFE; this indicated that the new crystal was not formed. Similarly, the new peak was not also found in the PTFE/T-ZnO and PTFE/ZnO composites.

Lima[31] investigated the crystalline structure of the polypropylene (PP), and the crystallinity of the PP was calculated through the WAXD profiles of PP as follows:

$$X_c = A_c/(A_c + A_a) \times 100\%$$

where X_c is the crystallinity and A_c and A_a are the areas of the peaks in the crystalline and amorphous regions, respectively. In this study, the crystallinity of the PTFE in the composites was also calculated through WAXD profiles. The result is shown in Figure 6(d), and it was found that the crystallinity of PTFE

decreased with increasing filler content. Meanwhile, compared to the crystallinity of the PTFE in the PTFE/BN composites, the crystallinity of the PTFE in the PTFE/T-ZnO and PTFE/ZnO composites decreased obviously. In addition, according to Figure 6(d), the crystallinity of the PTFE in the PTFE/T-ZnO and
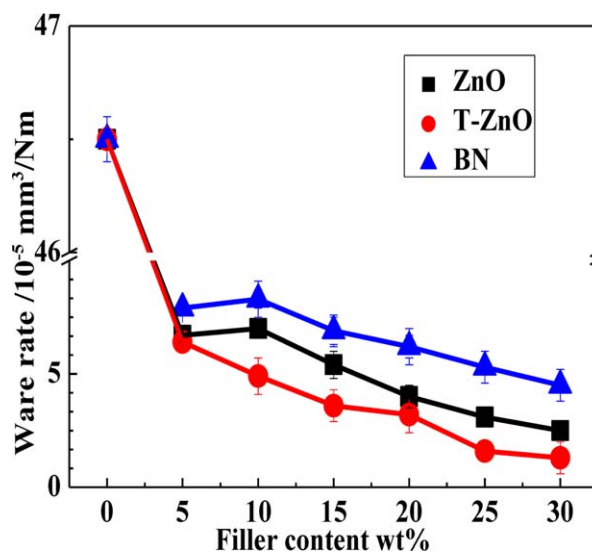

Figure 7. Wear rates of the PTFE composites filled with different fillers. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
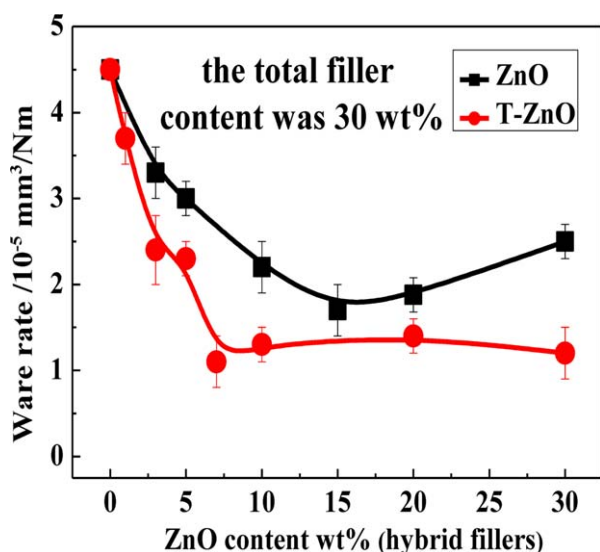
Figure 8. Wear rates of the PTFE composites filled with hybrid fillers. The total filler content was 30 wt %. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

PTFE/ZnO composites changed slightly at the same filler content; this indicated that the morphology of ZnO had little effect on the crystallinity of PTFE when the filler content was the same.

Combined with the results of the thermal conductivity of the PTFE composites, the enhanced thermal conductivity of the PTFE/BN composites was ascribed to not only the high thermal conductivity of the BN filler but also the higher crystallinity of PTFE in comparison with that in the PTFE/T-ZnO and PTFE/ZnO composites.

Wear Rate and Wear Mechanism

The wear rate of the PTFE composites filled with different fillers is shown in Figure 7. It was noted that the filling of BN, ZnO,

and T-ZnO decreased the wear rate of the PTFE composites. According to Figure 7, compared to the PTFE/BN and PTFE/ZnO composites, the wear rate of the PTFE/T-ZnO composites was the lowest; this indicated that T-ZnO was more helpful for improving the wear resistance of the PTFE. The composites filled with 30 wt % T-ZnO exhibited the lowest wear rate with a value of 1.2×10^{-5} mm^3/Nm. In particular, the wear rate of the composites filled with 30 wt % T-ZnO decreased about 39 times in comparison with that of the pure PTFE. Therefore, to improve the wear resistance of the PTFE composites, hybrid fillers were considered to add to PTFE. The wear rate of the PTFE composites filled with hybrid fillers (total filler content = 30 wt %) is shown in Figure 8. The composites that were filled with 7 wt % T-ZnO and 23 wt % BN hybrid fillers exhibited the lowest wear rate, with a value of 1.1×10^{-5} mm^3/Nm. Therefore, the obtained low wear rate of the PTFE composites containing hybrid fillers also successfully demonstrated the useful synergistic effect of hybrid filler in enhancing the wear rate of the composites for given fillers.

The morphology of the worn surface of the pure PTFE and its composites filled with different fillers was investigated by SEM, as shown in Figure 9. For pure PTFE, there are a lot of ploughs, adhered flakes, and fibers on the worn surface of the pure PTFE [Figure 9(a)]; this implied that abrasive and adhesive wear occurred. As shown in Figure 9(b), the cracks and the pulled BN particles were observed on the worn surface of the PTFE composite that incorporated 30 wt % BN. However, the worn surfaces of the PTFE composite incorporating 30 wt % ZnO and T-ZnO [Figure 9(c,d)] were smoother than that of the pure PTFE, on which ZnO particles, T-ZnO particles, and adhered flakes were also observed. In particular, rigid particles (BN, ZnO, and T-ZnO) were exposed on the worn surface of the PTFE composites during the process of friction and wear; these could bear the main part of the stress applied on the worn surfaces of polymer materials and then inhibit the mass loss
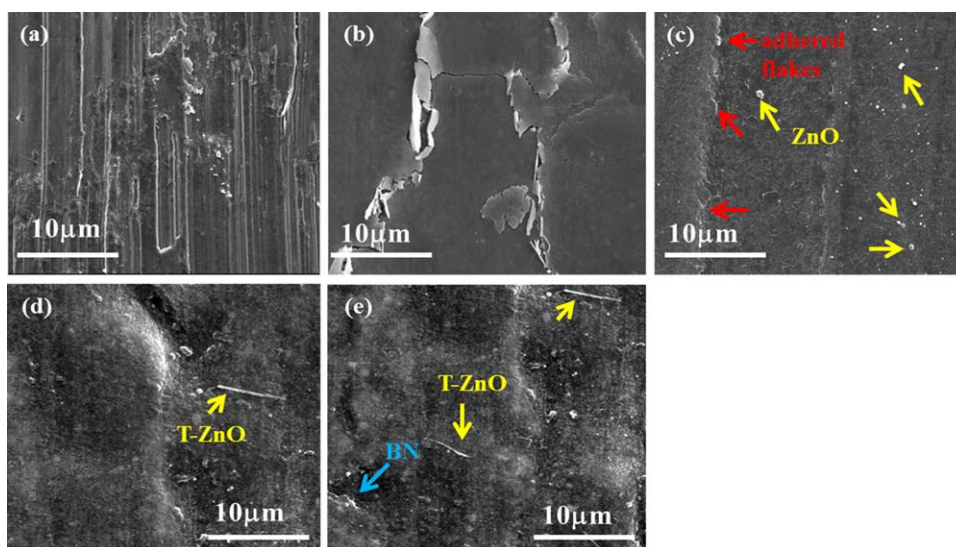

Figure 9. Morphologies of the worn surfaces of pure PTFE and its composites filled with different fillers: (a) pure PTFE, (b) PTFE/BN composites, (c) PTFE/ZnO composites, (d) PTFE/T-ZnO composites, and (e) thermally conductive paths of the PTFE/hybrid filler composite (T-ZnO/BN = 3 : 27). The filler content was always 30 wt %. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

and improve the wear resistance of the PTFE composites.[31,32] Compared to the worn surface of the pure PTFE, the worn surface of the composites filled with 30 wt % (ZnO, T-ZnO, BN, and hybrid fillers [Figure 9(e)] presented a much smoother surface, and slighter ploughing occurred. During the wear process, particles (BN, ZnO, and T-ZnO) were gradually exposed on the worn surface of the PTFE.

CONCLUSIONS

In this study, the thermal conductivity and wear resistance of the PTFE/BN, PTFE/ZnO, PTFE/T-ZnO, and PTFE/hybrid filler composites were investigated. Clear and continuous thermally conductive paths were observed through SEM images for the PTFE/hybrid fillers (T-ZnO + BN) composites. The hybrid filler (total filler content = 30 wt %) filled PTFE displayed a higher thermal conductivity than those filled with only the thermally conductive filler at the same filler content; in the particular, the thermal conductivity of the composites reached 1.38 W/mK when the T-ZnO content was 3 wt % in the hybrid fillers. Meanwhile, the composites filled with 7 wt % T-ZnO and 23 wt % BN hybrid fillers exhibited the lowest wear rate with a value of 1.1×10^{-5} mm^3/Nm. Therefore, the synergistic effect of the hybrid fillers were useful for enhancing the wear rate of the PTFE composites. Moreover, for the pure PTFE, abrasive and adhesive wear was found. The worn surfaces of the PTFE composites filled with ZnO, T-ZnO, BN, and hybrid fillers were much smoother than those of the pure PTFE, and slighter ploughing occurred.

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