

Research Article

Self-Lubricating Polytetrafluoroethylene/Polyimide Blends Reinforced with Zinc Oxide Nanoparticles

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ZnO nanoparticle reinforced polytetrafluoroethylene/polyimide (PTFE/PI) nanocomposites were prepared and their corresponding tribological and mechanical properties were studied in this work. The influences of ZnO loading, sliding load, and velocity on the tribological properties of ZnO/PTFE/PI nanocomposites were systematically investigated. Results reveal that nanocomposites reinforced with 3 wt% ZnO exhibit the optimal tribological and mechanical properties. Specifically, the wear loss decreased by 20% after incorporating 3 wt% ZnO compared to unfilled PTFE/PI. Meanwhile, the impact strength, tensile strength, and elongation-at-break of 3 wt% ZnO/PTFE/PI nanocomposite are enhanced by 85, 5, and 10% compared to pure PTFE/PI blend. Microstructure investigation reveals that ZnO nanoparticles facilitate the formation of continuous, uniform, and smooth transfer film and thus reduce the adhesive wear of PTFE/PI.

1. Introduction

Lubrication is critical to the operational safety and reliability of industrial manufacturing and processing. Lubrication technology has been widely used in industrial applications, including roller bearings, journal bearings, and gears. Efficient lubrication is valuable to dissipate frictional heat, extend fatigue life, and reduce friction and wear [1]. Existing lubrication systems rely on the use of synthetic lubrication oil or mineral oil, which cannot be used in strictly regulated fields such as pharmaceutical, food, and health care industries due to the potential product contamination [2]. Solid lubricants are considered the best option to control friction and wear if the usage of liquid lubricants is not allowed. However, solid lubricants should meet certain requirements in practical applications, such as

mechanical strength, stiffness, fatigue life, thermal expansion, and damping [3].

Polymers are extensively used as solid lubricants in dynamic mechanical parts due to their unique properties such as high strength, light weight and excellent wear, and solvent resistance [4]. Polytetrafluoroethylene (PTFE), also named “teflon,” is well known for its extremely low friction coefficient and excellent chemical resistance [5, 6]. However, the major drawbacks of PTFE, poor wear resistance and severe creep deformation, restrict its wide use in practical applications. Therefore, fibrous fillers (glass fiber, carbon fiber, and whisker) [7–9] and spherical nanoparticles [10] are added in PTFE to improve the wear resistance. PTFE has also been demonstrated as effective filler in other polymers to improve the tribological property of the polymer blends [11, 12].

Polyimide (PI), a class of high performance engineering plastics, is well known for its excellent mechanical properties and stability at high temperature, as well as superior dielectric properties and good chemical resistance, which have found wide applications in aerospace, automobile, and microelectronics industry [13]. However, the intrinsic large friction coefficient and high wear rate of pure PI limit its use in dynamic motion systems [11, 14]. Tremendous efforts have been devoted to reduce the friction coefficient and wear rate of PI by means of incorporating fibers [4, 15], nanometer particles [16, 17], solid lubricant [18], and so forth.

Zinc oxide (ZnO), with outstanding mechanical properties [19], has been widely used as reinforcing filler in various polymers, including PTFE [20], polymethylmethacrylate (PMMA) [21], ultrahigh molecular weight polyethylene (UHMWPE) [22], polyetheretherketone (PEEK) [23], and polyurethane (PU) [24]. Li et al. [20] reported that the wear volume loss of 15 vol.% ZnO/PTFE is only 1% compared to pure PTFE. Chang et al. [22] found that 10 wt% ZnO nanoparticles filled in UHMWPE reach the optimal tribological and mechanical properties of the composites with 30% and 190% enhancement in wear resistance and compressive strength, respectively, as compared to the pure UHMWPE.

To the best of our knowledge, the effect of ZnO nanoparticles on the tribological and mechanical properties of PI based nanocomposites has rarely been studied. In this work, PTFE/PI blend polymer was reinforced by different loadings of ZnO nanoparticles. The optimal loading was explored in association with greatest tribological and mechanical properties. The microstructures of the worn surface, transfer film, and impact-fractured surface were also examined to understand the reinforcing effect of ZnO in the nanocomposites.

2. Experimental

2.1. Materials. Polyimide powder (YS-20, 1–10 μm) was purchased from Shanghai Research Institute of Synthetic Resins. PTFE was commercially obtained from DuPont (7A-J, 25 μm commercial product). The ZnO nanoparticles (average diameter ≤ 60 nm) are purchased from Nanjing Haitai nano materials Co. Ltd. Before use, all the materials were dried at 110°C for at least 6 hours in oven.

2.2. Preparation of PTFE/PI Composites. In this work, the mass fraction of PTFE in polymer blend is fixed at 15 wt%. ZnO nanoparticles were added into the PTFE/PI blend with different mass ratios: 1, 2, 3, 5, 8, and 12 wt%, respectively. The mixture is weighted accordingly and blended mechanically. Then, the powder mixture was compressed under the pressure of 20.0 MPa and heated to 365°C in a mold with heating rate of 8°C/min. The compressed composite was held at 365°C for 45 min and then cooled down to ambient temperature in the mold while keeping the pressure unchanged. For friction and wear tests, the block was cut into a ring-shaped sample with 26.0 mm outer diameter, 22.0 mm inner diameter, and 2.5–3.0 mm in shoulder height, as seen in Figure 1(c).

2.3. Tribological Tests and Characterization. The friction and wear tests were conducted with a ring-on-ring friction configuration, Figure 1(a). The counter-face material is AISI1045 steel with hardness of HRC 52, Figure 1(b). Sliding was performed under ambient dry friction conditions over a period of 1 hour at sliding velocity of 0.69 (550 rpm) or 1.4 m/s (1115 rpm) and load of 100 N or 200 N. The ambient temperature is controlled at 25°C and relative humidity at $50 \pm 5\%$. Before test, the surfaces of each specimen and counterpart ring were polished to 800-grit finish with surface roughness R_a of 0.2–0.4 μm and then cleaned with alcohol. The friction force was measured using a torque shaft fixed with strain gauges, and the friction coefficient was calculated from the friction force. At the end of each test, the wear volume loss was calculated from the weight loss of each specimen. Three duplicate tests were carried out to minimize the data scattering, and the average of the data was reported.

The morphology of worn surface and impact-fractured surfaces of the composites was characterized by scanning electron microscope (SEM, QUANTA-200). The transfer films on the steel ring were examined by optical microscope.

2.4. Mechanical Tests. The tensile tests were carried out on a Universal Tester (Model CMT4254) at room temperature. The deformation rate was 5 mm/min. The impact tests were performed on an impact test machine (Model XJJ-5). Impact and tensile tests were conducted according to Chinese National Standard GB/T16420-1996 and GB/T16421-1996, respectively. For tensile test sample preparation, each composition was molded into a narrow-waisted dumbbell-shaped specimen, and the size of the narrow part is $30 \times 5 \times 3$ mm. For impact test, unnotched specimens with dimensions of $40 \times 3 \times 2$ mm (with the distance of support of 20 mm) were fractured by the impact mass at a speed of 2.9 m/s and impact input energy of 1.0 J. All the reported values were average of five effective measures.

3. Results and Discussion

3.1. Tribological Properties. Figure 2 shows the wear volume loss and friction coefficient of ZnO/PTFE/PI nanocomposites as a function of ZnO loading at 100 N load and sliding speed of 1.4 m/s. With increasing ZnO loading, both wear volume loss and friction coefficient decrease and reach the minimum value at 3 wt% and continuously goes up afterwards. Specifically, the wear volume loss of the nanocomposites reinforced with 3 wt% ZnO is 20% less than that of the PTFE/PI blend polymer. This loading effect is consistent with most of the literature reports that an appropriate loading of nanomaterials leads to optimized performance [25, 26]. In this work, ZnO can be well dispersed and completely covered by surrounding polymers at relatively lower loadings, for example, 3 wt%. With further increasing ZnO loading, polymer chains are not sufficient enough to cover the extremely large surface area exposed by ZnO nanoparticles and thus nanoparticles tend to agglomerate and negatively affect the interfacial bonding

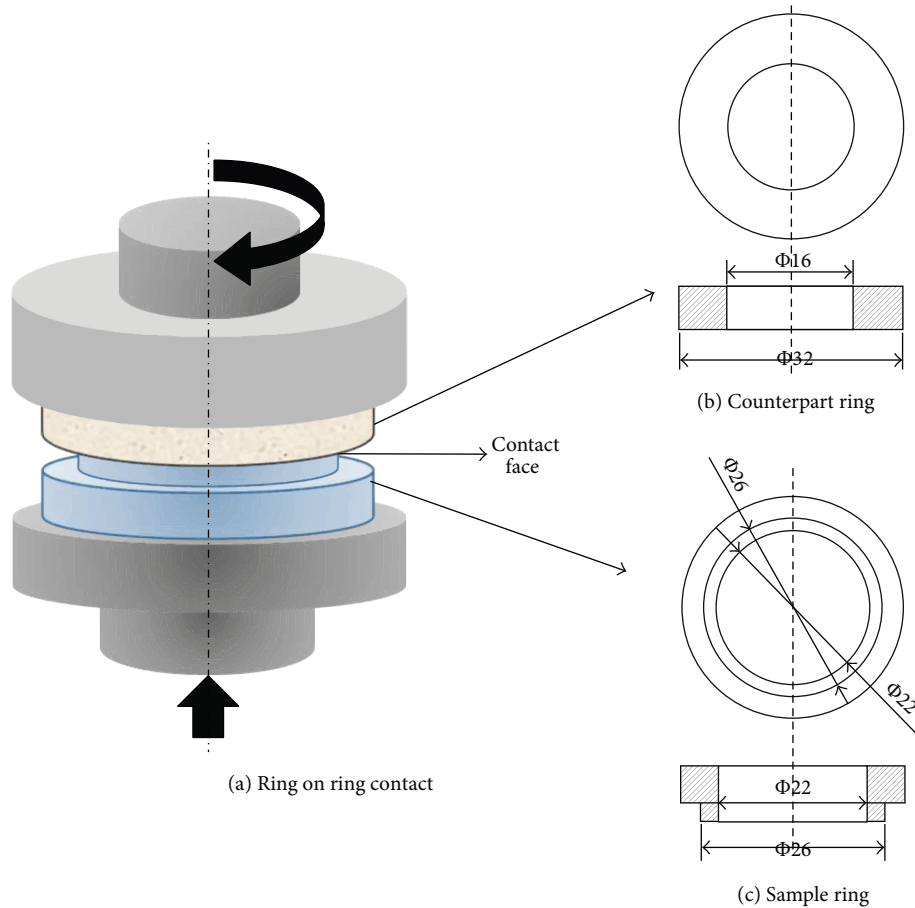


FIGURE 1: The schematic diagram of tribological testing configuration.

with polymer matrix. The weak interfacial bonding becomes weak joint, which can be easily peeled off and thus worse tribological property was observed. Similar phenomenon was also reported in carbon nanofiber/PTFE/PI nanocomposites [27].

The effects of sliding speed on wear volume loss and friction coefficient of various specimens at a load of 200 N are shown in Figures 3 and 4, respectively. It can be seen that the variation of both wear volume loss and friction coefficient experiences the same trend under sliding speeds of 0.69 and 1.4 m/s. The wear volume loss of ZnO/PTFE/PI nanocomposites decreases to the minimum value and then increases afterwards. The lowest wear loss is achieved with 3 wt% ZnO loading at higher sliding speed of 1.4 m/s, while larger ZnO loading of 8 wt% is required to reach the minimum value at relatively lower speed of 0.69 m/s. The ZnO nanoparticles serve as rolling balls between the friction interfaces, which would definitely reduce the interfacial friction and improve the tribological properties [23]. At higher sliding speed, larger shear force and friction energy facilitate the “pulling-off” of ZnO nanoparticles from polymer matrix and accumulation at the interface. Therefore, optimal amount of “rolling” ZnO nanoparticles would be accumulated at the interface from the nanocomposites with relatively lower ZnO loadings [27].

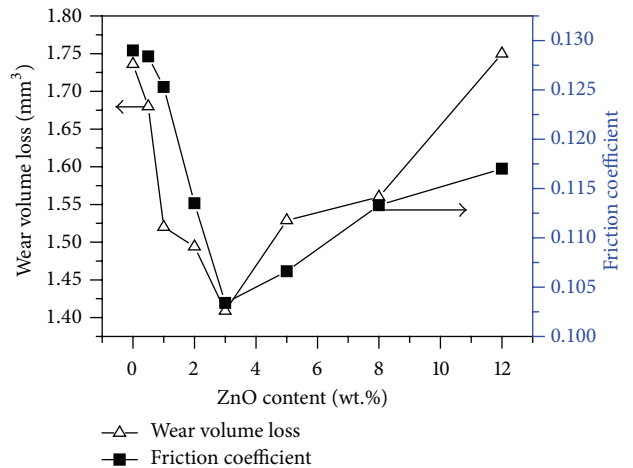


FIGURE 2: Effect of ZnO loading on wear volume loss and friction coefficient. Load: 100 N; sliding speed: 1.4 m/s.

Generally, the friction coefficient decreases with increasing ZnO loading, Figure 4. Moreover, significantly lower friction coefficient is observed at higher sliding speed. At 1.4 m/s, more friction heat is generated at the sliding surface,

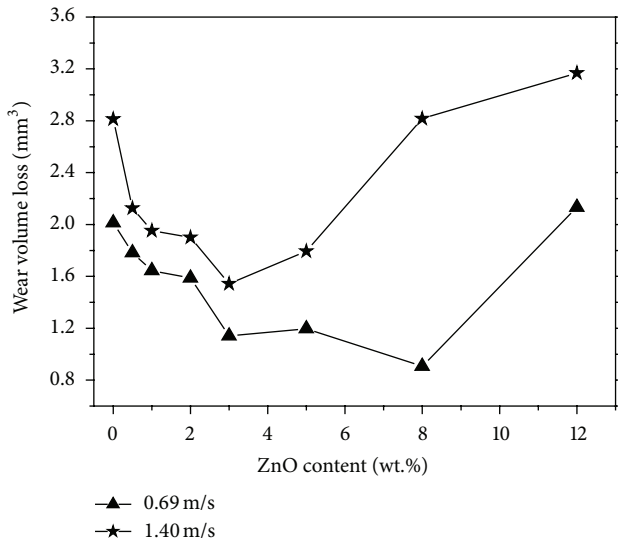


FIGURE 3: Wear volume loss with ZnO loading at 1.4 and 0.69 m/s. Load: 200 N.

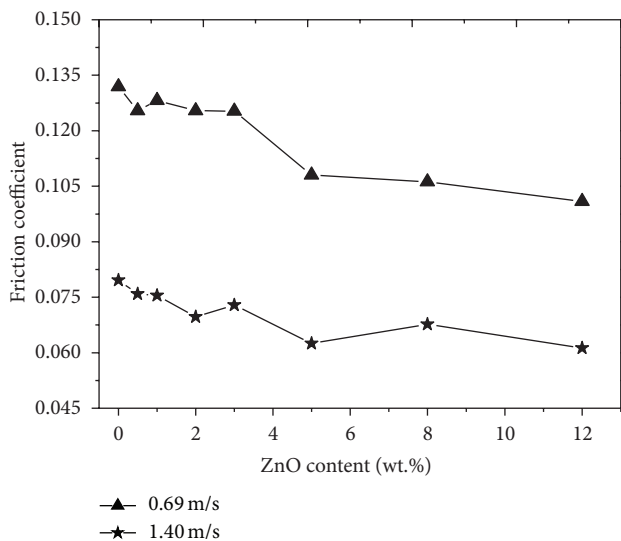


FIGURE 4: Friction coefficient with ZnO loading at 1.4 and 0.69 m/s. Load: 200 N.

which raises up the contact surface temperature and causes the “softening” of polymer chains. By further accumulation of friction heat, micromelting occurs that switches plastic polymer chain into elastoplastic which facilitates the reduction of friction coefficient [28].

To explore the role of ZnO on friction and wear behaviors of ZnO/PTFE/PI nanocomposites, the worn surfaces and transfer films of PTFE/PI, 3 wt% ZnO/PTFE/PI, and 12 wt% ZnO/PTFE/PI composites were comparatively investigated by SEM and optical microscope, Figures 5 and 6.

In Figure 5(a), the worn surface of PTFE/PI shows obvious nicks, shallow furrows, and plastic deformation, which indicates the dominant adhesive wear behavior [11]. After

incorporating 3 wt% ZnO, antiplough and anticut capacities of PTFE/PI composites can be significantly improved, Figure 5(b). Further increasing ZnO loading to 12 wt% peeled debris and deep furrows can be observed on the worn surface, Figure 5(c). These observations are in good agreement with the tribological results obtained in Figures 2–4.

Figure 6 displays the optical micrographs of transfer films formed on the counterpart steel ring after friction test. The transfer film of PTFE/PI appears to be rough and discontinuous, Figure 6(a), which could be easily scaled off from the wear track and negatively affects the wear resistance during sliding. Apparently, the transfer film for 3 wt% ZnO is continuous, uniform, and smooth, which is helpful to maintain a stable friction and thus the best tribological properties are obtained, Figure 6(b). When ZnO loading increases to 12 wt%, the transfer film becomes nonuniform, which reveals that an excess amount of ZnO would hinder the formation of a smooth transfer film, in Figure 6(c). As a result, the wear volume loss was increased considerably at higher filler content. These results reveal that a suitable loading of ZnO in the PTFE/PI polymer blend will favor the formation of smooth and continuous transfer films and contribute to the enhanced tribological properties.

Figure 7 illustrates the roles of ZnO nanoparticles during friction at different loadings. “Rolling ball” effect dominates the interfacial friction at relatively lower ZnO loading due to the good dispersion and particle-polymer interaction, Figure 7(a). Excess amount of ZnO in the nanocomposites leads to severe agglomeration, which affects the integrity of the nanocomposites in bulk and damages the transfer films at the friction interface, Figure 7(b). Therefore, peeled debris has been observed on the worn surface (Figure 5(c)) and also rough and noncontinuous transfer film on counterpart ring (Figure 6(c)).

3.2. Mechanical Properties. The mechanical properties of pure PTFE/PI and ZnO/PTFE/PI composites are shown in Table 1. The impact strength, tensile strength, and elongation-at-break of PTFE/PI composites increase initially with the increase of ZnO loading and reach the maximum value at 3 wt% ZnO and then decrease with further increasing filler content. Compared with PTFE/PI, the impact strength, tensile strength, and elongation-at-break of the 3 wt% ZnO/PTFE/PI composites are increased by 85, 5, and 10%, respectively, compared to PTFE/PI blend. The results indicate that the mechanical properties of PTFE/PI composites are well consistent with the tribological properties. Similar correlations between tribological and mechanical properties were reported as well by Zhang et al. in PEEK composites [29].

Due to its high specific surface area of ZnO, a strong interfacial interaction between ZnO and PTFE/PI matrix would be expected. ZnO is distributed uniformly in the matrix and covered with polymer chains completely when the filler content is relatively low, for example, 3 wt% in this work. It is well known that polymer containing a preexisting crack is a result of external stress and a small craze is often

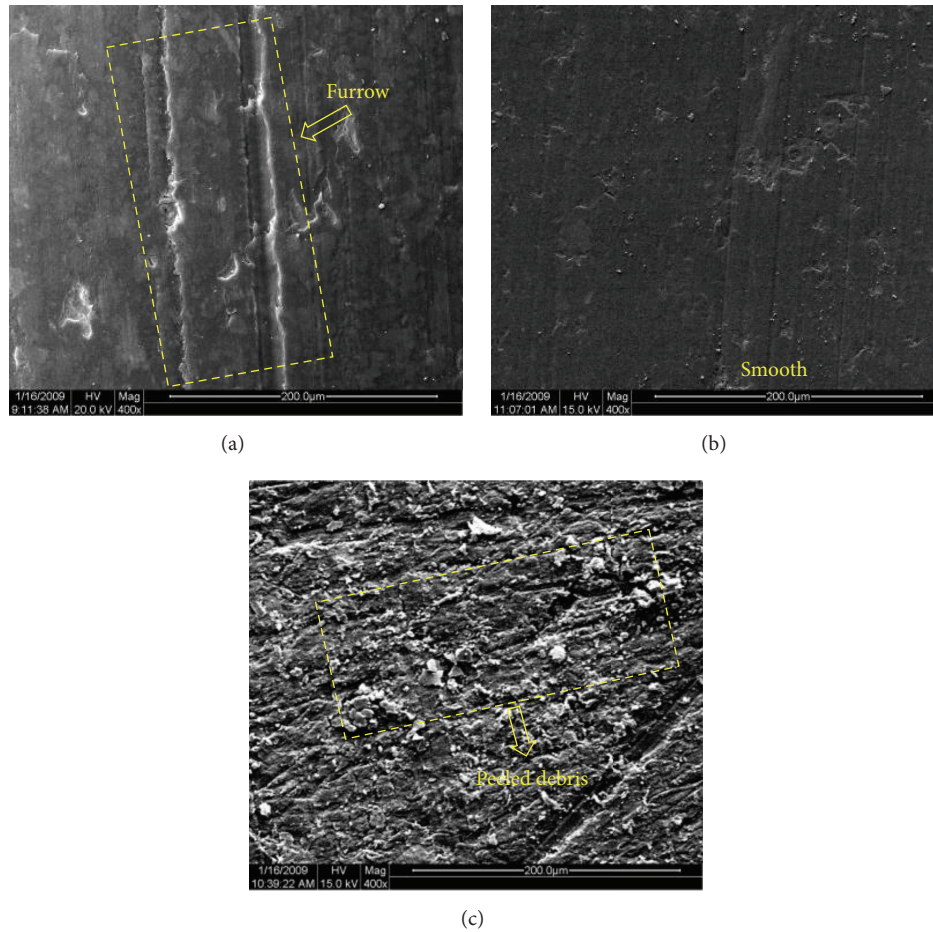


FIGURE 5: SEM micrographs of worn surfaces ($\times 400$, 100 N, 1.4 m/s). (a) PTFE/PI, (b) 3 wt% ZnO/PTFE/PI, and (c) 12 wt% ZnO/PTFE/PI.

TABLE 1: Mechanical properties of ZnO reinforced PTFE/PI nanocomposites.

Sample	Unnotched impact strength, kJ/m^2	Tensile strength, MPa	Elongation-at-break, %
15% PTFE/PI	27.1 ± 0.3	81.7 ± 0.5	27.5 ± 1.3
1% ZnO/15% PTFE/PI	35.6 ± 0.6	82.8 ± 0.6	28.2 ± 0.5
3% ZnO/15% PTFE/PI	50.3 ± 0.7	85.3 ± 0.7	30.1 ± 1.0
8% ZnO/15% PTFE/PI	29.3 ± 0.7	74.0 ± 0.8	20.1 ± 1.0

formed at the tip of the crack. ZnO could serve as binder at the craze region to delay the craze growth rate. In this regard, extra energy would be required to debond ZnO from polymer matrix before crack further develops, which contributes to a significant improvement in tensile strength [30]. However, the interfacial bonding between PTFE/PI matrix and ZnO would be damaged by severely agglomerated ZnO in excess amount, which accounted for the poorer tensile strength of ZnO/PTFE/PI composites at larger ZnO loading [23].

SEM micrographs of the impact-fractured surfaces of pure PTFE/PI and 3 wt% and 8 wt% ZnO/PTFE/PI are shown in Figures 8(a)–8(c). Obviously, the fracture surface appears relatively smooth for pure PTFE/PI but rough for 3 wt% and 8 wt% ZnO/PTFE/PI. The enhancement of impact strength

can be explained by cavitation mechanism of microsized rigid particles [31], where three stages are included: stress concentration, debonding, and shear yielding. ZnO nanoparticles act as stress concentrators, which lead to debonding at the interface between ZnO and polymer matrix. Nanoparticles on the fracture surface have been considered effective evidence to support the debonding claim [32]. ZnO nanoparticles marked by arrows in Figures 8(b) and 8(c) clearly indicate the debonding and subsequent plastic void growth of the polymer. As shown in Figure 8(b), the voids caused by debonding can be clearly seen on the fracture surface, which absorbs large quantity of energy upon fracture, reduces stress transfer to crazing zone, and therefore enhances mechanical properties [33]. However, the increase of the voids will reduce the bond strength and the impact strength of the

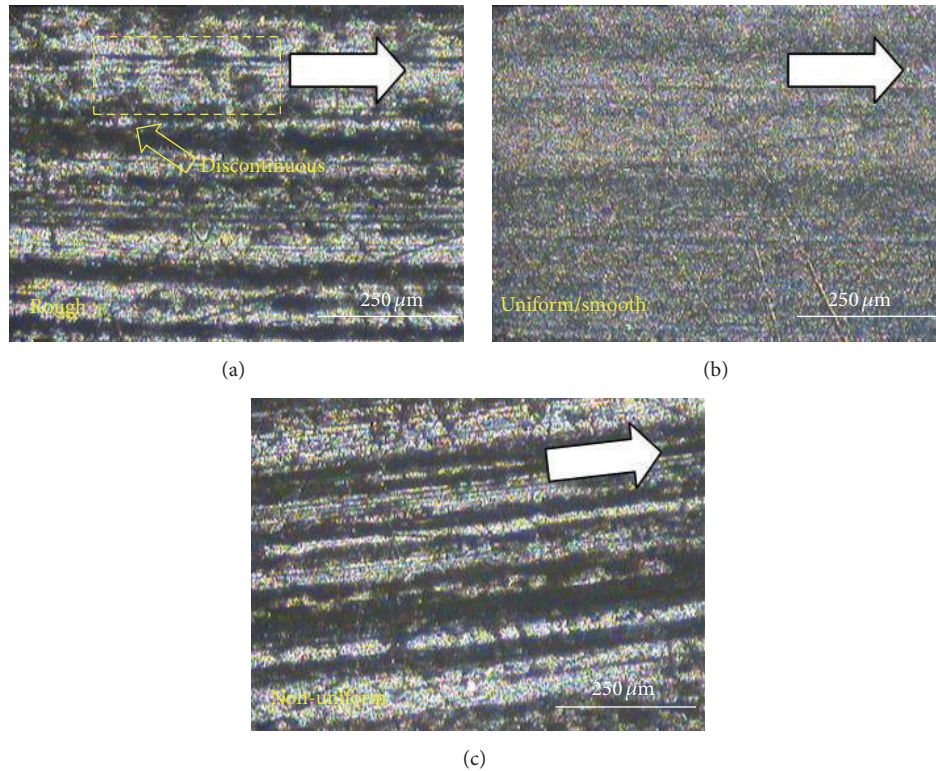


FIGURE 6: Optical micrographs of transfer films ($\times 200$, 100 N, 1.4 m/s). (a) PTFE/PI, (b) 3 wt% ZnO/PTFE/PI, and (c) 12 wt% ZnO/PTFE/PI. Arrow indicates the sliding direction.

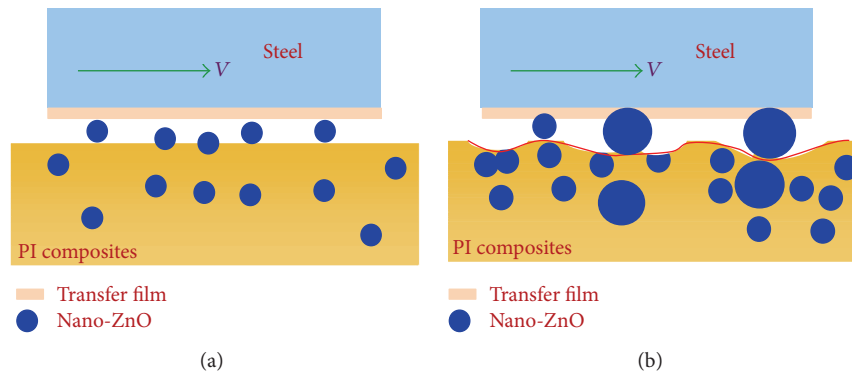


FIGURE 7: Schematic illustration of the role of ZnO at (a) low and (b) high loadings.

materials with the increase of ZnO loading, as shown in Figure 8(c).

4. Conclusions

To sum up, the tribological and mechanical properties ZnO/PTFE/PI composites have been investigated in this work. In comparison with unfilled PTFE/PI polymer blend, the antiwear property of 3 wt% ZnO/PTFE/PI composite is increased by 20%, which could be attributed to the formation of coherent, uniform transfer film that reduces the adhesive wear of PTFE/PI composites. Meanwhile, the impact strength, tensile strength, and elongation-at-break

of the 3 wt% ZnO/PTFE/PI nanocomposite are observed to increase by 85, 5, and 10%, respectively, due to the excellent interfacial interaction between ZnO and PTFE/PI matrix. The achieved superior tribological and mechanical properties of the ZnO/PTFE/PI nanocomposite allow its promising tribological and mechanical applications in bearing, compressor piston rings, impeller, and so forth.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

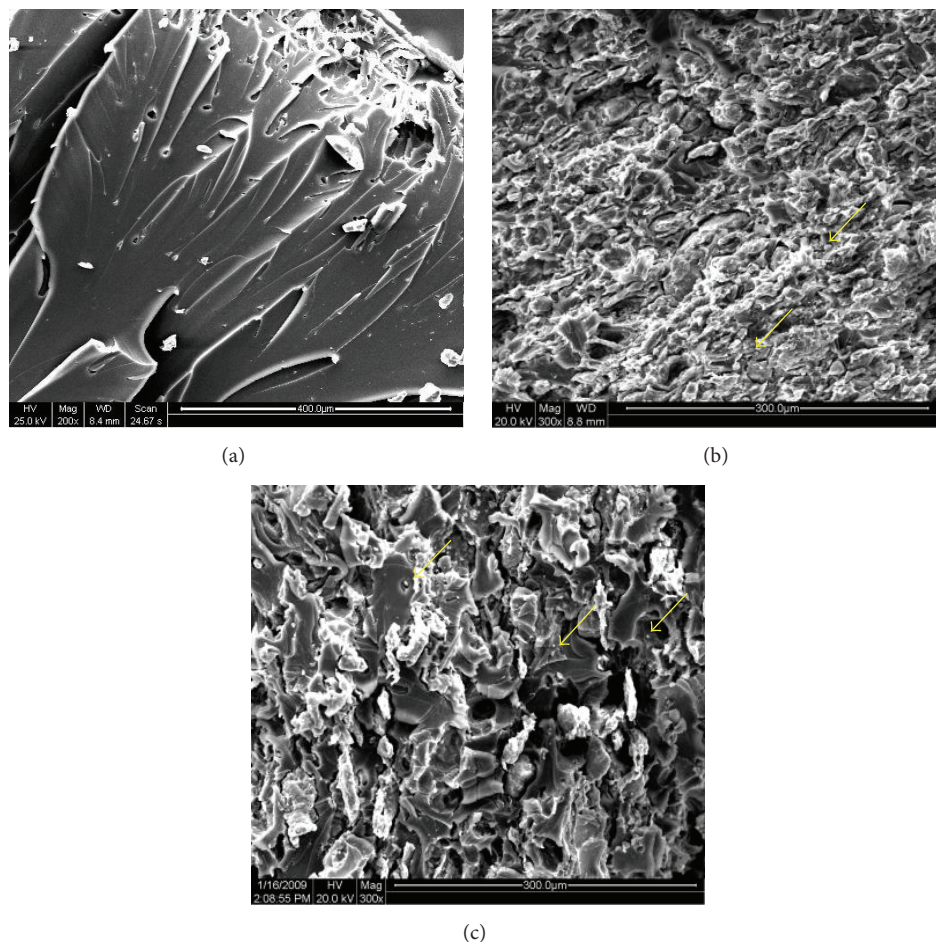


FIGURE 8: SEM impact-fracture surfaces. (a) PTFE/PI, (b) 3 wt% ZnO/PTFE/PI, and (c) 8 wt% ZnO/PTFE/PI. Deboned ZnO nanoparticles are marked by arrows.

Acknowledgments

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