Tribological behavior and energy dissipation characteristics of nano-Al₂O₃-reinforced PTFE-PPS composites in sliding system

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Abstract: Nanoparticles are increasingly being used to improve the friction and wear performance of polymers. In this study, we investigated the tribological behavior and energy dissipation characteristics of nano-Al₂O₃-reinforced polytetrafluoroethylene-polyphenylene sulfide (PTFE-PPS) composites in a sliding system. The tribological behaviors of the composites were evaluated under different normal loads (100–300 N) at a high linear velocity (2 m/s) using a block-on-ring tester. Addition of the nano-Al₂O₃ filler improved the antiwear performance of the PTFE-PPS composites, and the friction coefficient increased slightly. The lowest wear rate was obtained when the nano-Al₂O₃ content was 3% (volume fraction). Further, the results indicated a linear correlation between wear and the amount of energy dissipated, even though the wear mechanism changed with the nano-Al₂O₃ content, independent of the normal load applied.

Key words: nano-Al₂O₃; polytetrafluoroethylene-polyphenylene sulfide (PTFE-PPS); tribological behavior; dissipated energy

1 Introduction

Wear is a highly complex phenomenon and involves mechanical and chemical interactions that depend on several factors such as the sliding velocity and load, material components used, environmental temperature, surface roughness, and constituent material of the counterpart. Designers and engineers have attempted to predict the wear properties of different tribological systems. Archard's law is one of the earlier equations used to relate the wear volume and tribological parameters and is given as follows [1]:

$$V_{\text{wear}} = \frac{KFL}{H} \tag{1}$$

where *K* is the dimensionless wear coefficient; *F* is the normal force; *L* is the sliding distance; *H* is the hardness of the softest surface. Similar models have been proposed by other researchers as well [2-4].

Researchers have analyzed tribological systems using several methods. For instance, the energy dissipated has been used to determine the wear volume and elucidate the wear mechanism. MATVEEVSKY [5] was the first to define the energy dissipated generated on per contact area as the friction power intensity, $Q_{\rm f}$. MOHRBACHER et al [6, 7] calculated the energy during fretting tests and proposed the concept of dissipated energy. HUQ and CELIS [8] used the dissipated energy to predict the wear loss owing to friction in TiN and (Ti, Al)N coatings. They reported that there exists a linear relationship between the wear loss and the amount of energy dissipated. Similar results were observed by FOUVRY et al [9, 10] during the fretting wear of metals and hard coatings. JAHANGIRI et al [11] established an accurate predictive model based on energy dissipation to describe the wear failure of a W-25wt%Cu matrix composite. In their later work [12], the authors defined SWV as a new criterion for evaluating material friction. The wear-dissipated energy linear relationship has also been observed in the case of polymer/metal friction pairs. COLAÇO et al [13] stated that the linear wear volumedissipated energy relationship exists in the case of ultra-high-molecular-weight polyethylene/metal contacts under different lubrication environments. Finally, SMERDOVA et al [14] recently proposed that the dissipated energy is a function of the measured mass loss in the case of epoxy/epoxy sliding contacts. In their study, the amount of energy dissipated increased linearly with

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Engineers have studied the tribological performance of polytetrafluoroethylene (PTFE) because of its self-lubricating nature, high melting temperature, and chemical inertness [15], owing to which it is used widely to fabricate self-lubricating polymer composites in the field of mechanical sealing [16]. In order to improve the performance of mechanical components, polymer materials and nanoparticles have been used to enhance the properties of PTFE. LUO et al [17] found that PA6/PTFE blends exhibit different mechanical properties for different PTFE contents. BURRIS and SAWYER et al [18] reported that the wear resistance of PTFE increases monotonically with the addition of Al₂O₃ nanoparticles. BURRIS and SAWYER [19] and LI et al [20] also observed similar improvements in the properties of PTFE at higher nanoparticle contents. Finally, SCHWARTZ and BAHADUR [21] used Al₂O₃ nanoparticles to improve the wear resistance of polyphenylene sulfide (PPS). The wear rate decreased initially and then increased, with the lowest wear rate observed at a loading rate of 2% (volume fraction).

In this work, we aimed to characterize the tribological behaviors of 5% (volume fraction) PTFE-PPS composites filled with Al_2O_3 nanoparticles using the energetic analysis method. The main objective was to investigate the tribological behaviors of the nano- Al_2O_3 -reinforced PTFE-PPS composites and elucidate the relationship between their wear properties and frictional energy dissipation. A simple theoretical model is proposed to investigate the wear of the composites, with the aim of using the method as a tool for designing mechanical components.

2 Experimental

2.1 Materials used

M18F PTFE powder (average size of 38 μ m, apparent density of 0.47 g/mL) was obtained from Daikin Corporation, Japan, while PPS was supplied in powder form (apparent density of 2.12 g/cm³) by Polyplastics Corporation, Japan. γ -phase Al₂O₃ nanoparticles with an average diameter of 20 nm were supplied by XF NANO Corporation, China.

First, a mixture of the PTFE and PPS powders was prepared using a high-speed mechanical mixer; the PTFE and PPS contents were 95% and 5% (volume fraction), respectively. Next, the Al₂O₃ nanoparticles were added to the PTFE-PPS mixture in volume proportions of 0%, 1%, 2%, 3%, and 5%. The dried PTFE-PPS powder and the nanoparticles were mixed again in a high-speed mechanical mixer for 600 s. Then, the mixture was compressed in a special mold under a pressure of 45 MPa. After the cold compression process, the samples were sintered in an electric heating furnace with a temperature control system. The samples were heated from room temperature to 320 °C over 2 h and held at this temperature for 60 min. Next, the sintering temperature was elevated to 360 °C over 1 h and this temperature was maintained for 2 h. Subsequently, the samples were cooled to room temperature. The dimensions of the samples were 12.3 mm×12.3 mm× 19.6 mm.

2.2 Wear test

To evaluate the friction and wear performance of the PTFE-PPS and nano-Al₂O₃-reinforced PTFE-PPS composite, a block-on-ring tester (MRH-3) was used, as shown in Fig. 1. The counterpart used was a GCr15 steel ring with a roughness, R_a , of 0.2–0.4 µm. Three normal loads of 100, 200, and 300 N were applied to the ring at a steady linear sliding speed of 2 m/s, which was affected by circular rotation at 800 r/min, under dry conditions. The pausing periods were not identical for every test. The sliding interval for the sliding test was 1800 m. Thus, the test distances were 1800, 3600, 5400, 7200, and 9000 m. The mass loss of the samples was measured repeatedly after the tests in order to prevent the running-in period from affecting the test results. The samples were weighed using an analytical balance with an accuracy of 0.1 mg. Before and after the tests, each sample was cleaned in acetone and dried in an oven. The volume wear rate, K, was calculated using the following equation:

$$K = \frac{\Delta m}{\rho NL} \tag{2}$$

where Δm is the mass loss; ρ is the density of the composite; N is the contact normal load; L is the total sliding distance.

The temperature of the friction surface is a significant parameter and should be considered during

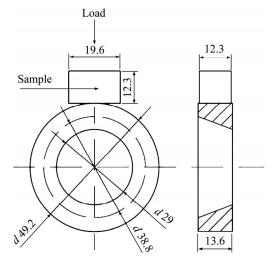


Fig. 1 Schematic of contact diagram for friction couple (unit: mm)

friction measurements. Thus, a thermocouple was placed 1.8 mm below the friction surface of the test sample so that the temperature of the worn surface could be determined with reasonable accuracy. The test sample was dried at 26 °C before the test by vacuum drying in an oven, and the ambient temperature was kept constant at 24 °C during the test. The thermocouple was connected to a MY-700 temperature controller (TESHOW, China) for recording the temperature.

The morphologies of the worn surfaces of the samples were investigated with a scanning electronic microscopy apparatus (SEM; JSM–5600LV, JEOL, Japan). A gold film was sputtered on the worn surfaces before the observations.

3 Results

3.1 Tribological properties of composites

3.1.1 Effect of filler content on tribological properties of composites

Figure 2 shows the typical variations in the coefficient of friction and volume wear rate values of the nano-Al₂O₃-reinforced PTFE-PPS composites with the Al₂O₃ nanoparticle content. The wear tests were carried

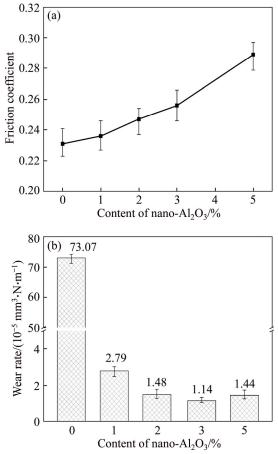


Fig. 2 Variations in coefficient of friction (a) and volume wear rate (b) of nano-Al₂O₃-reinforced PTFE-PPS composites under 200 N at 2 m/s for different filler contents

out under dry sliding conditions at a normal load of 200 N, sliding speed of 2 m/s, and sliding distance of 7200 m. As can be seen in Fig. 2(a), the friction coefficient varied from 0.231 to 0.288 and was strongly dependent on the filler content. The friction coefficient increased slowly with the addition of 1% filler and then increased rapidly with a further increase in the filler content. Further, Fig. 2(b) shows that the wear rate first decreased and then increased with the addition of the nano-Al₂O₃ filler. A significant reduction was observed when the filler content was 1%. The best antiwear characteristics were observed in the case of the composite with 3% filler. The wear resistance of this composite was 64 times higher than that of the filler-free composite. This is primarily because the Al₂O₃ particles improved the mechanical strength of the composite and thus increased its shearing resistance and load-carrying capacity [21]. The results also suggested that the nano-Al2O3 content plays a key role in determining the antiwear performance of PTFE-PPS composites.

3.1.2 Variations in friction and volume wear rate of composites with sliding distance

Figure 3 shows the typical variations in the coefficient of friction and volume wear rate of the composites with the sliding distance. The friction

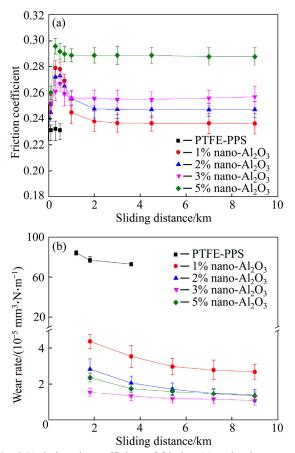


Fig. 3 Variations in coefficient of friction (a) and volume wear rate (b) of nano-Al₂O₃-reinforced PTFE-PPS composites with sliding distance under 200 N at 2 m/s

phenomenon exhibited a run-in state before reaching a stable state during most tests. As can be seen in Fig. 3(a), the friction coefficient increased rapidly initially and then decreased, eventually plateauing at a value much higher than that corresponding to the stationary state. With an increase in the nanoparticle content, the decrease in the friction coefficient was smaller. The volume wear rate of the composites, however, changed continuously with the sliding distance, as shown in Fig. 3(b). Further, as was observed in the case of the friction coefficient, the wear rate decreased with an increase in the sliding distance. A relation between the friction coefficient and wear rate is observed: the change of both value is relatively obviously at the transient friction state and the increased proportion of nano-Al2O3 leads to a decrease of the decreasing trend.

3.1.3 Variations in friction and volume wear rate of difference normal loads

Figure 4 shows the typical variations in the coefficient of friction and volume wear rate of the nano- Al_2O_3 -reinforced PTFE-PPS composites with different filler contents for different normal loads. Figure 4(a) shows that the friction coefficient of each composite decreased monotonously with an increase in the normal load. Notably, the friction coefficient of the

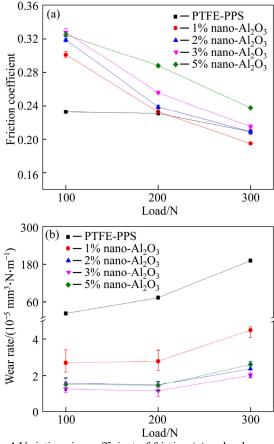


Fig. 4 Variations in coefficient of friction (a) and volume wear rate (b) of nano-Al₂O₃-reinforced PTFE-PPS composites under different normal loads at 2 m/s

1% nano-Al₂O₃/PTFE-PPS composite was the lowest, indicating that lower nanoparticle content was more effective in improving the friction characteristics of the composites at higher loads. The results in Fig. 4(b) indicate that the volume wear rate of the PTFE-PPS composite increased sharply with the increase in the load. In contrast, while the volume wear rates of the nano-Al₂O₃/PTFE-PPS composites also increased with the load and their values were markedly lower than that of the filler-free PTFE-PPS composite for every normal load. These results also lead to the same conclusion, that is, the best antiwear properties were achieved when 3% nano-Al₂O₃ filler was added.

3.2 Characterization of worn surfaces

In order to elucidate the effects of the nano-Al₂O₃ filler on the friction and wear properties of the nano-Al₂O₃-reinforced PTFE-PPS composites, their worn surfaces were imaged using SEM, as shown in Fig. 5. It can be seen from Fig. 5(a) that the worn surfaces have many adhering fibers and film marks, which were caused by the destruction and transfer of the macromolecular chain structure of PTFE. This indicates that the wear mechanism of the PTFE-PPS composite is adhesion. In contrast, the nano-Al₂O₃/PTFE-PPS composites exhibit two wear mechanisms: abrasive wear in the case of composites with nano-Al₂O₃ content lower than 1% (see Fig. 5(b)) and adhesion and ploughing wear in the case of the composites with nano-Al₂O₃ contents between 2% and 5% (see Figs. 5(c) and (d)). It can also be seen from Fig. 5(b) that uniform and continuous plough marks are present on the worn surface of the 1% nano-Al₂O₃/PTFE-PPS composite, meaning that the presence of the Al₂O₃ nanoparticles between the material matrix increased the strength of the composite and prevented the macromolecular chain structure from creeping and gliding, thus reducing the adhesion between the PTFE fibers and films. With an increase in the filler content, the extent of wear changes from severe to mild. Further, it can be seen from Figs. 5(c) and (c) that the worn surface consists of many grains and shows extensive adhesive wear. Based on a previous study [22], this phenomenon may be explained by the fact that Al₂O₃ nanoparticles destroy the crystalline and ordered structure of PTFE, with movement of the debris on the surface because of sliding and rolling.

For comparing the wear mechanisms under different normal loads, SEM image of the worn surface of the 3% nano-Al₂O₃/PTFE-PPS composite after sliding under 100 N is shown in Fig. 6(a). As seen from the figure, a few fibers adhere to the surface, and the surface exhibits slight adhesive wear. Further, as can be seen from Fig. 6(b), when the normal load was increased to 300 N, large matrix of composite adhesion on the worn surface

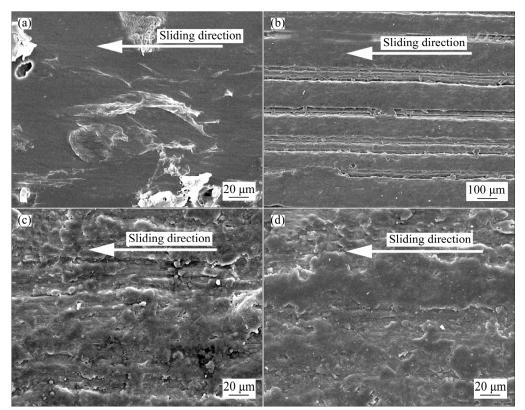


Fig. 5 SEM images of worn surfaces of various composites at sliding speed of 2 m/s and normal load of 200 N: (a) PTFE-PPS; (b) 1% nano-Al₂O₃/PTFE-PPS; (c) 3% nano-Al₂O₃/PTFE-PPS; (d) 5% nano-Al₂O₃/PTFE-PPS

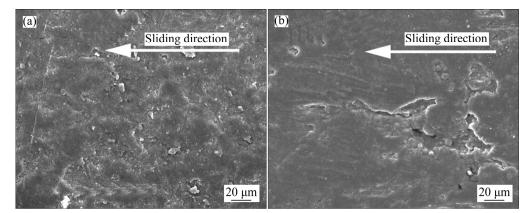


Fig. 6 SEM images of worn surface of 3% nano-Al₂O₃/PTFE-PPS at sliding speed of 2 m/s and normal load: (a) 100 N; (b) 300 N

and the polymer matrix melted because of the high surface temperature, with the extent of wear being very high. Indeed, the friction surface temperature increased markedly under high normal loads, which, in turn, led to material softening, resulting in the wear mechanism becoming more complex. The surface temperatures are listed in Table 1. It can be seen that the temperature for a normal load of 300 N was very high.

4 Discussion

In this study, we attempted to investigate the friction and wear properties of nano-Al₂O₃-reinforced PTFE-PPS composites in a sliding system using an energetic approach, in order to better understand these properties. The dissipated energy, E_d , was calculated using the following equation:

$$E_{\rm d} = F_{\rm T}(x)L = F_{\rm N}\mu(x)L \tag{3}$$

where $F_{\rm T}$ is the tangential friction force; $F_{\rm N}$ is the normal load; μ is the friction coefficient; *L* is the relative displacement during wear. Hence, a differential equation for the dissipated energy, tangential friction force, and relative displacement during wear could be obtained as follows:

$$dE_{d} = F_{N}\mu(x)dx \tag{4}$$

where dE_d is the increment in the dissipated energy

Table 1 Temperature data for PTFE-PPS composites with different nano-Al₂O₃ contents when subjected to dry sliding under different conditions (sliding speed: 2 m/s; sliding distance: 7200 m)

Volume proportion of Al ₂ O ₃ /%	Normal load/N	Stead-state temperature/°C			
1	100	130.5			
1	200	180.8			
1	300	205.3			
2	100	139.8			
2	200	188.9			
2	300	230.5			
3	100	140.2			
3	200	189.1			
3	300	230.6			
5	100	144.1			
5	200	207.6			
5	300	265.5			

corresponding to an increment in the relative displacement during wear and

$$E_{\rm d} = F_{\rm N} \int_{x=0}^{x=L} \mu(x) {\rm d}x$$
 (5)

where E_d is the cumulative dissipated energy.

However, the mathematical relationship between the dissipated energy and the relative displacement during wear is not known. In the present work, the Newton-Cotes method was used to verify the experimental data [23]. Thus, E_d was determined using the following equation:

$$E_{\rm d} = F_{\rm N} \sum \mu_{\rm k} \Delta x_{\rm k} \tag{6}$$

A fundamental factor affecting wear and the amount of energy dissipated in tribological systems is the tangential friction force. Hence, studying the friction force and its variations is essential for being able to relate the wear to the energy dissipated. Figure 7 shows the change in the original tangential friction force versus the distance for the nano-Al₂O₃-reinforced PTFE-PPS composites with different filler contents under the three normal loads. It can be seen that the initial tangential friction force is considerably higher than the tangential friction force in the stationary state and that the variations in the force depend strongly on the nano-Al₂O₃ content under the different normal loads. The variations in the force decrease with the addition of nano-Al₂O₃, as can be seen clearly for a normal load of 300 N.

Based on the tangential friction force data, the amount of energy dissipated for each interval (spacing of 600 m) versus the wear distance is shown in Fig. 8(a).

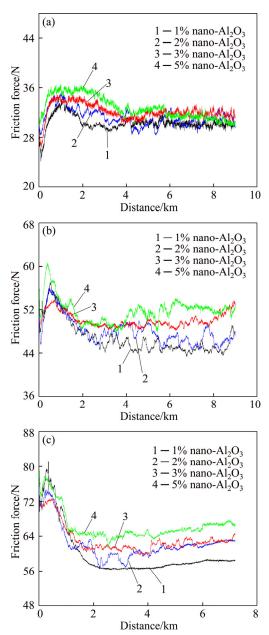


Fig. 7 Changes in original tangential friction force with distance for nano-Al₂O₃-reinforced PTFE-PPS composites with different filler contents under normal loads: (a) 100 N; (b) 200 N; (c) 300 N (sliding speed of 2 m/s)

The energy corresponding to each interval should be added, as indicated by Eq. (6). Thus, it can be seen that there is good correlation between the data shown in Figs. 7(b) and 8(a). As shown in the figure, the cumulative energy that is dissipated at the start of the friction process is more than that in the steady wear state, in keeping with previous studies wherein the reasons the energy is the primary factor causing wear were investigated [9, 10]. Thus, the wear volume loss is greater at the start of the friction process. Similarly, the curve of the total amount of energy dissipated versus the wear distance is shown in Fig. 8(b). All the composites exhibit a fully linear "E-D" curve, in contrast to the "W-D" curve, with the curves having a constant slope. Thus, the slopes indicate that the average tangential friction force remains constant, owing to the excellent lubrication performance of the PTFE-PPS composite. Moreover, the dissipated energy increases owing to an increase in the tangential friction force, which is caused by the addition of the nano-Al₂O₃ filler. Hence, the nano-Al₂O₃ content has a determining effect on the wear as well as the amount of energy dissipated.

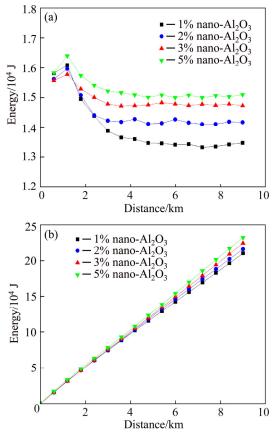


Fig. 8 Dissipated energy (a) and total amount (b) of energy dissipated versus distance for each interval (spacing of 600 m) for each composite under normal load of 200 N and sliding speed of 2 m/s

Figure 9 relates the wear volume loss with the E_d values of the nano-Al₂O₃/PTFE-PPS composites. The slope, ∞_{ν} , represents the volume loss per unit of energy dissipated, which can be considered a criterion for developing a wear model for the composites under any process [11, 12]. In all the cases, it can be seen clearly that the wear volume loss exhibits a linear relationship with the dissipated energy. This implies that the volume loss corresponds to the cost of energy. As stated above, although the friction and wear change during the friction process, a steady slope indicates that the wear mechanism for each composite remains relatively constant. Moreover, the friction phenomenon is very sensitive to changes in the filler content. With an increase in the nano-Al₂O₃ content of the composites, the

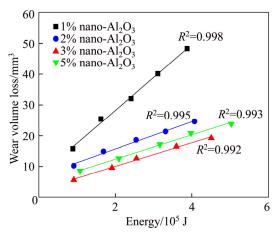


Fig. 9 Wear volume loss-dissipated energy curves for nano-Al₂O₃-reinforced PTFE-PPS composites under normal load of 200 N and sliding speed of 2 m/s

wear mechanism shifts from being primarily adhesion based to being related to ploughing and adhesion abrasion. Thus, as shown in the figure, the plotted results do not fit this trend. When the nano-Al₂O₃ content was doubled from 1% to 2%, ∞_v decreased from 1.09× 10^{-4} mm³/J to 4.58×10^{-5} mm³/J. This means that the wear mechanism of the composite changed significantly. Further, when the filler content was increased further from 2% to 5%, ∞_v coincides with one other, which has a value between 4.58×10^{-5} mm³/J and 3.83×10^{-5} mm³/J. These results show that, although the composites exhibited differences in their morphologies, including the deformation of their worn surfaces and the adhesion of the fibers and films, they underwent similar extents of wear damage by consuming the same amount of energy.

The similar occurs when the tests are carried out under the different normal load. Figure 10 shows the "wear volume loss-energy" curves for the composites under the three normal loads. The corresponding data are listed in Table 2. Based on the fact that the slopes of the

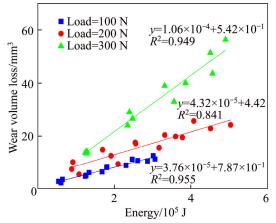


Fig. 10 Wear volume loss-dissipated energy curves of nano-Al₂O₃-reinforced PTFE-PPS composites (2%-5%) under three normal loads (sliding speed of 2 m/s)

Table 2 Wear data and corresponding dissipated energy values for various wear distances for PTFE-PPS composites with different nano-Al₂O₃ filler contents under dry sliding conditions (A: 1800 m; B: 3600 m; C: 5400 m; D: 7200 m; E: 9000 m; linear speed: 2 m/s)

Volume proportion of Al ₂ O ₃	Normal	Wear volume loss/mm ³				Dissipated energy/10 ⁵ J					
	load/N	А	В	С	D	Е	А	В	С	D	Е
1	100	8.18	12.71	16.54	21.86	26.30	0.533	1.01	1.63	2.24	2.84
1	200	15.75	25.49	32.16	40.18	48.36	0.893	1.62	2.41	3.11	3.88
1	300	30.99	56.48	80.65	96.76		1.16	2.25	3.19	4.33	_
2	100	2.92	5.20	8.45	11.26	12.66	0.544	1.23	1.61	2.30	2.86
2	200	10.22	14.89	18.63	21.39	24.69	0.914	1.69	2.54	3.31	4.07
2	300	13.6	29.05	38.99	51.40		1.23	2.39	3.31	4.50	_
3	100	2.34	4.83	6.57	9.01	10.62	0.606	1.24	1.69	2.46	2.91
3	200	5.57	9.53	12.65	16.52	19.26	0.915	1.91	2.56	3.59	4.51
3	300	13.54	24.07	33.04	43.62	_	1.27	2.34	3.55	4.56	
5	100	3.06	6.26	8.17	10.78	11.37	0.647	1.37	1.79	2.61	3.15
5	200	8.53	12.62	17.18	20.87	23.89	1.07	2.09	3.16	3.97	5.03
5	300	14.34	26.62	40.02	56.41		1.28	2.47	3.85	4.90	_

all the curves are constant, it can be inferred that each composite exhibited similar wear under each normal load. In this regard, to compare the different between slopes, the wear under normal loads of 100 N and 200 N is similar. However, for a load of 300 N, the wear is more extensive, indicating that each unit of dissipated energy caused a greater wear volume loss on the axis. Finally, with regard to the wear morphology, the worn surfaces primarily indicated adhesion-related wear under low loads but complex wear under higher loads.

As stated above, the nano-Al₂O₃ filler had a pronounced positive effect on the tribological properties of the composite. Even though the morphology of the worn surfaces changed dramatically with the addition of the nano-Al₂O₃ filler, the proposed method is reasonable for determining the energy and thus measuring the wear loss. In other words, although differences were observed between the composites in terms of the extent of wear, wear morphology, and friction process, it is possible to evaluate the wear using the dissipated energy approach, and vice versa.

5 Conclusions

In this work, the tribological behaviors of PTFE-PPS composites reinforced with nano- Al_2O_3 in different contents were investigated using a dissipated energy approach. Based on the results of tests performed using a block-on-ring tester, the following conclusions can be drawn:

1) The addition of nano-Al₂O₃ improved the antiwear performance of the PTFE-PPS composites, with the friction coefficient increasing slightly. The lowest

wear rate was observed when the nano-Al_2O_3 content was 3%.

2) There exists a linear relationship between the wear volume loss and the amount of energy dissipated for the nano-Al₂O₃-reinforced PTFE-PPS composites under the experimental conditions investigated in this study. This was the case for all three normal loads used.

3) The rate of energy consumption was similar for the PTFE-PPS composites reinforced with nano-Al₂O₃ in amounts of 3%-5%, implying that they exhibited similar wear-energy relationships.

As a final remark, an important advantage of the energetic approach used in this study is that it can be used to analyze the wear mechanisms of different polymer materials and determine whether they are similar. Further, it can be a very useful tool for designing materials for mechanical equipment.

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