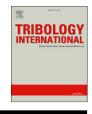
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Improvement of dry sliding tribological properties of polyamide 6 using diamond nanoparticles



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ARTICLE INFO	A B S T R A C T
<i>Keywords:</i> Polyamide 6 Nanodiamond Surface functionalization Abrasive wear	In the present study, it was revealed that incorporation of nanodiamond containing carboxylic group (ND-COOH) decreased considerably specific wear rate (~30%) and COF (~60%) of polyamide 6 (PA6) at 1 wt% loading. Compared with ND-COOH, amino functionalized ND enhanced further wear performance of PA6 which was associated with its finer dispersion as well as greater influence on the improvement of mechanical properties, toughness and crystallinity of PA6. Surface temperature increment caused by frictional heat was in close agreement with COF and wear characteristics of the samples. Optical microscopy images revealed that NDs promote the abrasive wear mechanism in PA6/ND composites.

1. Introduction

Polyamides (PAs) as engineering thermoplastics with good mechanical and tribological properties are widely employed in various applications including bearings and gears [1,2]. High resistance to abrasion and lubricating characteristic alongside with relatively low price and appropriate processability make this polymer suitable for widespread applications [3,4]. Practically, polyamide 6 (PA6) is the most desirable PAs due to a reasonable balance of cost, performance, versatility and processability.

It is well known that the wear performance of PA6 is greatly dominated by molecular weight, degree of crystallinity and water absorption [5]. Improvement of wear behavior of PA6 by incorporating various conventional solid lubricants and fibers was also reported by researchers which were found to be effective at high loadings (greater than 15 wt%) [6–9]. Kumar and Kanagaraj [10] observed that by adding 20 wt% of graphite in PA6, the best tribological performance was achieved; thanks to the enhancement of mechanical properties and heat dissipation. In this context, nanoparticles have shown better improvement compared with micron sized particles [11–13] at lower concentrations. Zhao et al. [12] reported that the wear rate of PA6 in presence of nanoalumina decreased much higher than micron sized alumina.

Up to now, efficiency of various nanoparticles on tribological performance of PA6 has been examined. Srinath and Gnanamoorthy [14] used nanoclay to improve the wear properties of PA6 in presence of water. The lowest coefficient of friction (COF) was achieved for PA6 filled with 5 wt% nanoclay which was associated with the increment of degree of crystallinity and reduction of plasticization effect of water. Effect of CNT on the wear performance of PA6 was also reported in literature [15,16]. In such case, specific wear rate at both wet and dry conditions decreased by incorporating 1 wt% CNT. Frictional behavior of PA6/SiO₂ nanocomposites was also investigated by Garcia et al. [17] who reported a significant reduction of COF from 0.5 to 0.18 by adding 2 wt% SiO₂ nanoparticles. Pikhurov and Zuev [18] achieved lowest COF of PA6 nanocomposites by adding 1 wt% of fullerene. According to them, alteration of crystalline phase of PA6 (formation of α -crystalline phase) was the reason of enhancement of tribological properties. Very few studies have reported the role of nanoparticle surface modification on wear properties of nanocomposites. Campo et al. [19] showed that CNT functionalized with amino group can improve tribological properties (wear rate) of epoxy resin more than untreated one.

Detonated ND is a kind of carbon nanoparticle which has been taken into consideration recently by researchers due to its impressive properties such as high strength, high thermal conductivity, biocompatibility and scalable production technique [20,21]. Rich surface chemistry of ND provides excellent flexibility for surface functionalization with wide range of chemical modifiers [22,23]. Furthermore, superior mechanical and lubricating properties of ND originated from its diamond core motivate the use of ND in polymer nanocomposites with multifunctional properties [24–27] as well as improvement of lubricating efficiency of fluids [28].

The research works on ND filled polymer nanocomposites dealing

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with wear performance are limited to thermosetting polymers such as epoxy resin [29]. Ayatollahi et al. [30] achieved noticeable reduction in wear rate (84%) and COF (50%) by adding only 0.1 wt% ND particles into epoxy resin. They claimed that enhancement in these parameters is related to the increment of hardness and lubrication effect of ND.

Recently, we reported on the role of NDs on the morphological and mechanical properties of PA6 processed by melt mixing method [31]. The objective of the present article is to examine the role of ND particles on friction and wear performance of PA6 under dry sliding condition. To this end, various parameters such as weight fraction of ND, surface modification and normal load are considered. Furthermore, morphological characteristics of worn surfaces are investigated to clarify the wear mechanisms.

2. Experimental

2.1. Materials and processing

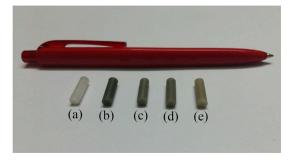
PA6 (Akulon F223D) was obtained from DSM Co., Netherland. ND powder was supplied from NaBond Technologies Co., limited, China with average particle size of 4–6 nm, purity of 99% and density of 3.05–3.3 g/ cm³. First, as received ND was oxidized at 420 °C under air atmosphere to enhance carboxyl group content of ND [32], which is referred to as ND-COOH hereafter. Amino functionalization of ND-COOH particles using ethylene diamine (EDA) was performed using wet chemistry (see details in Ref. [31]). Amino functionalized ND was designated by ND-EDA in this article.

PA6/ND composites were prepared by melt mixing method in two steps including preparation of PA6/ND at high filler loading using internal melt mixer (30/50 E, Brabender, Germany) and temperature of 220 °C followed by dilution of the mixture with PA6 using counterrotating twin-screw extruder (SHJ-20 Nanjing Giant, L/D = 20) at barrel temperature profile of 220–235 °C [31]. Before any processing steps and any tests, samples were dried in a vacuum oven at 80 °C for 16 h to avoid undesirable effects of moisture. The composite specimens were prepared by ND-COOH at variable concentrations of 0.25 wt%, 0.5 wt% and 1 wt%. To investigate the role of amine functionality of ND, PA6/ND-EDA composite was prepared at 0.25 wt% loading where the maximum improvement in mechanical properties was already achieved [31].

2.2. Characterization

Wear and friction properties of samples were investigated with a pinon-disc tribometer, according to ASTM G99. Cylindrical pin specimens with 5 mm diameter and 15 mm length (see Fig. 1) were prepared from injection molded samples and then dried in a vacuum oven before and after wear tests to measure mass loss precisely.

Counterface disc was stainless steel (52100 AISI bearing steel) with hardness of 64 HRC and surface roughness of 0.4 μ m. The disc was polished and washed with acetone after each test to eliminate possible wear tracks. Schematic of pin-on-disc tribometer is shown in Fig. 2. COF was



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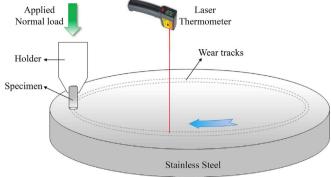


Fig. 2. Schematic of pin-on-disc tribometer.

recorded during the wear test permanently by data acquisition card connected to a computer. So the curves of COF versus sliding distance were plotted by computer. Specific wear rate (K_w) was calculated with the following equation:

$$K_{\rm w} = \frac{\Delta m}{\rho.F.l} \tag{1}$$

where Δm is the mass loss (mg) (measured precisely with 4 decimal numbers balance), ρ is the density of specimens (g/cm³), F is the normal force (N) and l is the sliding distance (m). All the tests were conducted for 2000 m sliding distance with disc linear velocity of 0.26 m/s. Applied normal loads were 20 N, 40 N and 80 N for different tests which provided wear intensities (pv value) of 0.27 MPa m/s, 0.53 MPa m/s and 1.06 MPa m/s, respectively, where p is applied pressure and v is linear velocity.

The disc surface temperature close to the pin and disc contact point (shown in Fig. 2) was recorded during the wear tests using a laser thermometer (TFA 31-1122, Germany, accuracy: \pm 1.5 °C). As shown in Fig. 2, the thermometer was held perpendicular to the disc with a distance of almost 20 cm. The surface temperature was measured at specified time intervals (5 min) and surface temperature increment ($\Delta T = T - T_{in}$ where T_{in} is the disc temperature at the start of the test which was almost the room temperature) was plotted versus sliding distance. To investigate the morphology and wear mechanisms, worn surfaces of PA6 and its composites were observed with optical microscopy (OM) images using Olympus BX51 m microscope in reflection mode.

3. Results and discussion

3.1. Effect of ND on wear and frictional performance of PA6

Variation of COF with sliding distance at the normal load of 80 N (pv = 1.06 MPa m/s) for all the samples is illustrated in Fig. 3a. It is inferred that there is a transition distance (region) after which COF shows an abrupt increase and then it approaches to steady value. COF transition of neat PA6 shows somewhat different behavior compared with PA6 composites. For neat PA6, transition is observed at short sliding distance of ~200 m, consistent with literature [33], which is followed by gradual increment of COF until it steadies off at ~1500 m. However, transition distance of PA6 composites occurs at longer distances but COF steadies off immediately after the transition region is met. This suggests that ND alters the development of friction forces at the contact surface area. According to literature, developing transfer layer in the contact surface from torn up materials and drawn up nanoparticles can be considered as the main reason for transition behavior of COF in PA6 composites [9,33,34].

It is evidenced that COF at the steady condition (average values of COF after the distance of 1500 m) decreases from 0.94 for neat PA6 to 0.36 for PA6 containing 1 wt% ND-COOH showing 62% reduction. It is to

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