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Research Article

Correlation of Wear Rate with Surface Hardness of Pure Nylon and Graphite-Reinforced Nylon Composites

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Abstract

Comparative tribological performance of pure nylon and 1 % (w/w) micron sized graphite particle reinforced nylon composite has been investigated. The influence of increasing sliding velocity and time on the wear rate has been revealed in this study. Wear test was carried out in a pin-on-disc type arrangement using 2 kg dead load under 0.43, 0.86 and 1.26 m/s sliding velocities in dry sliding condition. Interestingly, wear rate seems to show a downward trend with time in the composite sample but increases with the sliding velocity for both cases, although the effect is less pronounced in the composite samples. The addition of graphite was found to cause up to 7 fold decrease in wear rate and enhance tribological properties. The trends in wear rate variations at all conditions were found to be well correlated with surface hardness which were further complemented by surface roughness and morphological examinations.

Keywords: Nylon; Graphite; Sliding wear; Hardness.

Introduction

In recent years, researchers are trying to use polymeric materials in place of metallic materials in tribological applications because polymers have certain advantages such as ease of fabricability, good corrosion resistance, low cost etc. Their friction co-efficient value is also low compared to metals because they have low interfacial bonding energy [1]. Moreover, they have self-lubricating properties which reduces their need for oil and grease in their application as gear and cam shafts [2]. So, when we further introduce self-lubricating agents, need for lubrication is minimized further. Nylon is a polymer which is known for its wonderful combination of tribological, mechanical and thermal properties. Its flame retardation property is very good and products can be prepared by thermoforming, extrusion or molding [3].

It is not without disadvantages however; high co-efficient of friction, low temperature instability, high water absorption, dimensional instability [3-6] etc. are a few drawbacks. Due to these, problem arises during its application in high loading condition and low temperature environment. Reinforcing nylon with various short fibers has been proved to be useful in increasing the mechanical properties. And reinforcement with solid lubricant such as graphite, PTFE, MoS_2 can lessen the high wear rate values as they help in reducing slip stick motion [7]. Srinivas et al. studied the abrasive wear properties of the polyamide 66 polymer reinforced with micron sized graphite particles [8]. They conducted the test at various load and found that with increasing load, there was an increase in wear rate values. Various researchers also reinforced polyamide with things such as MoS_2, lamellar structured expanded nano graphite, graphite particles, wax etc. and found that reinforcement addition significantly improved the tribological properties of the composites and highest wear rate and friction coefficient is found for the pure sample [9-12] . Wang et al. tried to investigate surface roughness effects on the wear behavior of the carbon fiber reinforced polyamide 1010 composite and found that, with increasing surface roughness, wear rate increases. Lowest wear rate was found at 0.11-013 μm surface [13]. From the literature review, it is found that various reinforcements generally improve tribological properties. Among them, Graphite, due to its structure, acts as an excellent reinforcement. They can be easily fragmented under gliding conditions by shear

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forces. It produces a transfer film on the part being worn and thus improves wear [14]. This kind of composite can also withstand temperatures up to 300° C [15]. It has low cost, light weight and ease of availability. Thus, micron sized graphite powder was added to the nylon matrix and pure and composite samples were prepared by hot pressing. To have accurate comparison, testing conditions were identical for both samples. The aim was to produce composite material that can be an alternative to the traditional bearing; bushing and automobile materials. These automotive parts are traditionally produced using metallic materials which are very heavy. Researchers are trying to produce light weight material to reduce fuel consumption and making greener world. Polymeric materials, due to light weight, can be an option. In modern times, light vehicles may contain more than a thousand polymer parts and the demand for polymer parts are increasing day by day.

Materials and methods

Materials

In this study, nylon beads (density 1.13gm/$cm^{3)}$ were used as matrix material and micro graphite particles (density 2.2 gm/cm^3) were used as the reinforcement. Both these things were collected from the local market in Dhaka.

Sample preparation

For producing pure sample, cleaned aluminum die was sprayed with silicon mold releasing agent who prevents sticking. 220 grams of nylon grains were placed in the female part of the die. The die was then placed in a furnace at 110°C for moisture removal. After 2-3 hours the die was removed from the furnace and the upper (Male) part of the die was placed above it before finally placing the die in a Hot Press. A pressure of 30 KN was applied. Initially the temperature was allowed to rise to 200°C and was kept at 200°C for 10 minutes. Then the temperature was again increased to 220°C and kept at this temperature for 10 minutes. Finally the temperature was increased to 225°C for 5 minutes. Subsequently, the water supply line was turned on and the die was cooled to room temperature. Following the same procedure, multiple pure samples were prepared.

For producing composite samples, 1% graphite by weight was taken in a beaker and thoroughly mixed with acetone with the use of stirrer. Nylon beads were then added to the blend and it was vigorously mixed to obtain a uniform mixture without any lumps. Subsequent steps were similar to the pure ones for producing the final composite sample.

Wear test procedure

To investigate wear and frictional characteristics of the pure nylon and graphite reinforced composite, pin-on-disc type test apparatus was used following ASTM G-99 test standards [16]. The test was conducted under dry sliding condition at ambient air with a relative humidity and temperature of 77 ± 5% and 31 ± 2°C, respectively. Both sample and the counter body were of same material for each test and were cleaned with acetone to remove dirt or any kind of greasy materials before each test. Samples were of cylindrical shape with 6 mm diameter and 20 mm height. Initial sample weights were taken every time before conducting the test. Samples were then mounted into the apparatus so that it pressed against the counter body disc, and a 2 kg dead load was applied normal to the mating surfaces which was kept fixed. Specimen was kept static and disc was rotated at 0.43, 0.86 and 1.26 m/s sliding velocities in atmospheric conditions. Test duration was varied for each rotation speed to explore the wear rate variation with time. Test periods of 1, 2, 4 and 8 hours were chosen. After every test period, weight of the sample was taken and compared with the initial value to compute weight loss and wear rate.

Optical microscopy

In addition to visual inspection, samples were imaged using B-600MET Optical microscope at 100x magnification to examine the condition of the worn specimen surface.

Study of surface roughness

Roughness values of the wear track were taken using TR 200 surface roughness tester to see if there was any difference in the smoothness of pure and composite samples after wear. Roughness values of virgin samples were taken for comparison.

Study of surface hardness

Shore A hardness was taken before and after wear along the wear track using shore durometer. The durometer, similar to other hardness measurement schemes, measures the depth of an indentation in the material created by a given force on a standardized presser foot. This depth is dependent on the hardness of the material, its viscoelastic properties, the shape of the presser foot, and the duration of the test. In our case, force was applied for 11 seconds.

Results and discussion

Figure 1a 1b and 1c compare the wear rate of pure and composite samples as a function of time for 0.43, 0.86 and 1.26 m/s sliding velocities respectively. Wear rate was obtained from the mass loss data which was measured using precision electronic balance having accuracy of ±0.01 mg. From the figure 1, it is apparent that, pure samples have higher wear rate than composite samples at all sliding velocities and wear periods. Literature survey already confirms this phenomena [11, 17]. This is mainly due to the self lubrication effect of graphite powders which accumulate on the wear track and acts as a lubricating and heat dissipating medium. These actions reduce friction co-efficient and hence reduce wear. Now, if wear rate is compared against time, then it is seen that wear rate shows an upward trend with time at all sliding velocities for the pure sample. But in the composite sample, wear rate increases initially, then decreases after 2 hours. Similar behavior was seen at all sliding speeds.

In the pure sample, when sliding initially begins, few particles get worn out from the surface. This abrasive wearing is mainly due to the local shear stress and localized heating by friction between contacting surfaces. When these particles get attached to the interacting surfaces, a transfer film continues to form. Shyam bahadur explained this mechanism of transfer film forming in a study [18]. With unremitting rubbing action, more tearing occurs and the transfer film thickness increases gradually. At this stage, friction co-efficient continues to increase. Wear mechanism is two body abrasion mechanism because particles get restricted in between the interacting surfaces. But in case of composite, transfer film mainly contains loose graphite powder. It is known that, graphite has mica sheet like layered structure. Atoms lying in the same layer are strongly bonded to each other and closely packed. But the bonding forces between the layers are weak Van der Waals force and layers are relatively far apart.

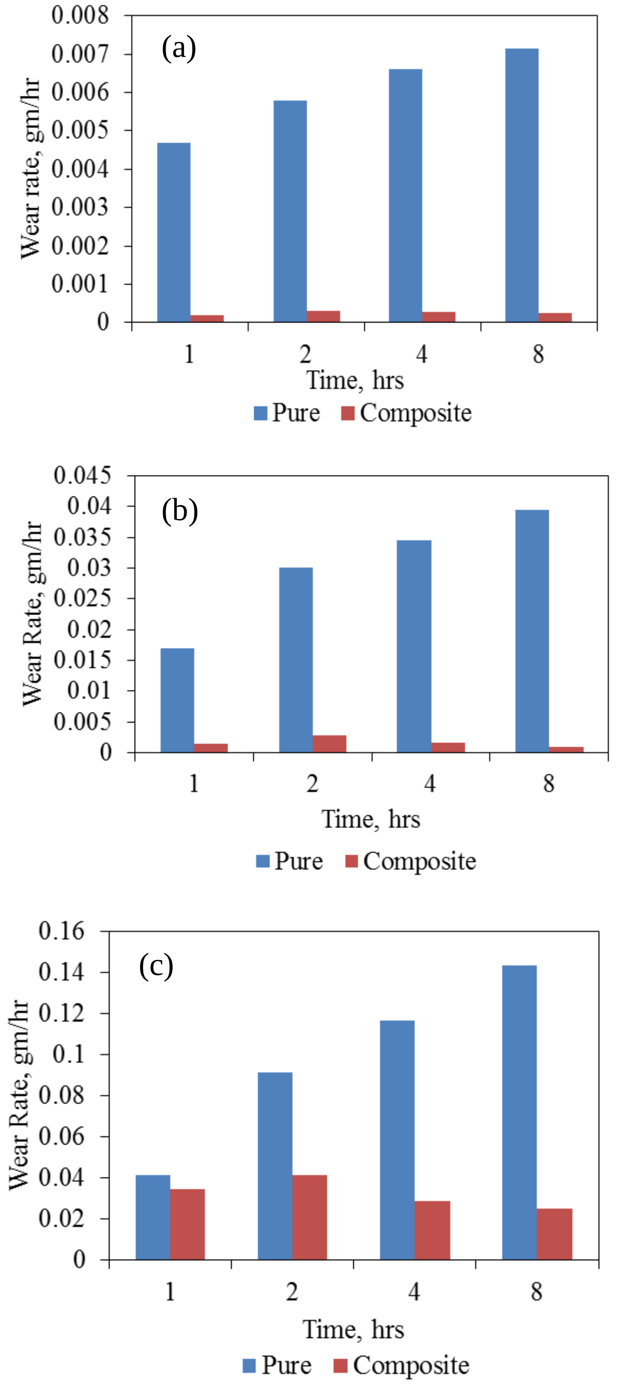

Figure 1. Wear rate of pure and composite sample at (a) 0.43 m/s (b) 0.86 m/s (c) 1.26 m/s

So when rubbing action occurred during wear test, composite surface got worn out. Worn graphite powder gets accumulated in the wear track. Their sheet like structure enables them to be aligned perpendicular to the applied load which would then make sliding against each other relatively easy. They are free to roll and slide between the contacting surfaces causing three body abrasion. Initially wear rate increased because surface was rough and caused high wear. It also takes some time to form transfer film. Again, experimentally it is found that, three bodies abrasion is slower than two body abrasion [19] and no such self lubricating medium is present. This can be the probable explanation for wear rate increase with time in pure nylon and decrease in composite sample.

Figure 2 gives the wear rate obtained after 8 hours plotted against sliding velocity. Wear rate increases at higher sliding speeds as common sense would dictate. More interestingly however, if we compare pure sample with the composite one, we can see that, wear rate is much lower in the composite one than the pure sample. The reduction in the wear rate is again due to micro graphite reinforcement which acted as a solid lubricant and heat dissipating medium.

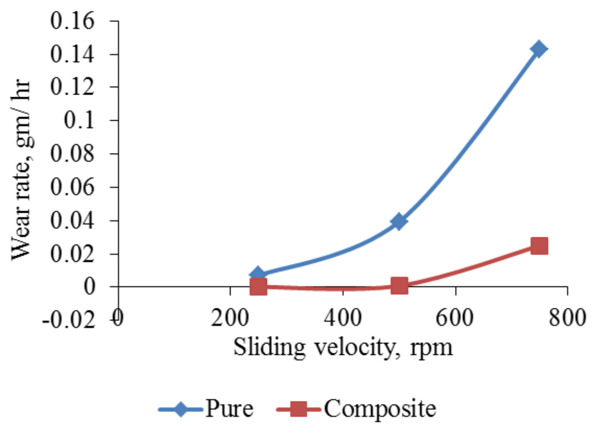

Figure 2. Wear rate versus sliding velocity of the pure and composite sample after 8 hours

Wear behavior of polymer and polymer based composites are associated with the surface temperature and viscoelastic properties. When sliding occurs, heat is generated at the asperities between the sliding surfaces and hence the temperature rises. Adhesive wear of materials depend upon the amount of heat generated, how the generated heat is dissipated and the ability to retain their integrity at the working temperature. If heat cannot be dissipated quickly, surface temperature increases and causes more wear. Bin-Bin Jia et al. used the formula of heat generation which is Q= μPV (watts) where μ= friction co-efficient, P= applied load and V= sliding velocity [20]. From the formula it is readily apparent that higher sliding speed will generate more heat. Watanabe et al. also stated that, with the increase of sliding speed, friction coefficient increases because of temperature rise at the nylon- metal interface [21].

Wear rate is directly correlated with friction coefficient and so we see that the increased surface temperature indirectly induces more wear. Same result is found in this study where increasing sliding speed increased wear rate because of higher friction coefficient associated with higher surface temperatures. But as mentioned previously, the effect is less in the composite. Graphite is the main reason for this improvement. Because of its high anti frictional characteristics, graphite acts as a lubricating medium. As a result, less heat is generated due to less friction. The heat that is still generated is quickly dissipated due to the high thermal conductivity of the graphite powder. Moreover, when these particles are strongly bonded to the matrix polymer, their strong interfacial characteristics help to transfer the load from the matrix to the reinforcement and hence protect from serious destructive action. Shin et al. also said that heat dissipation increases in the composite due to graphite reinforcement in the Nylon matrix [22].

Heat dissipation is mandatory to reduce wear because, as previously discussed, accumulated heat in the interface induces more wear. How heat accumulation induces more wear can be explained by the fact that this accumulated heat can cause subsurface melting and recrystallization. During melting, polymer chains become amorphous, crystallinity is decreased on that sub surface portion as only the crystalline portion of polymer experiences melting. Since the actual portion below the surface that experiences melting are very small compared to the overall sample, it solidifies very quickly. The cooling rate influences the amount of crystallinity. Slow cooling provides time for greater amounts of crystallization to occur. Fast rates, on the other hand, such as rapid quenches, yield highly amorphous materials [23]. As a result, surface and sub surface portion has less crystallinity than the rest of the bulk. It is well established that crystalline polymers have higher tensile strength, density, hardness etc. than their amorphous counterparts. Hence the lower crystallinity in the sub surface region compared to the bulk may as well be responsible for the decrease of hardness after wear. But in composite, such localized high temperature is not reached due to the lubricating and heat dissipating nature of graphite which tends to minimize friction between the contacting pin and disc. To see this phenomenon has occurred or not, hardness values were taken at multiple locations on the wear track before and after wear at all sliding velocities and time. Average surface hardness values of the pure and composite sample are shown in figure 3.

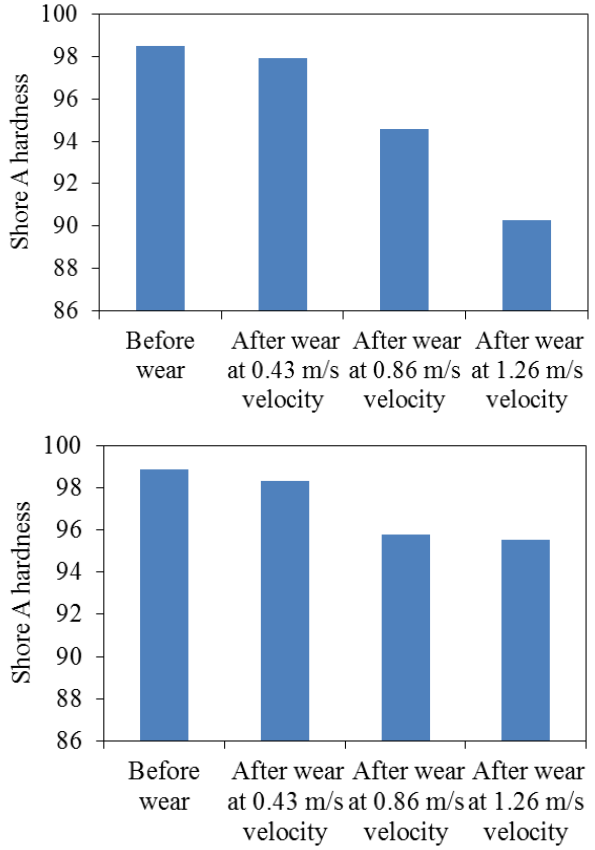

Figure 3. Average surface hardness values of (a) pure (b) composite sample

For the pure sample, average hardness was 98.48 before wear. At higher sliding velocities hardness tends to decrease, falling to 90.26 in case of 1.26 m/s sliding velocity corresponding to a decline of about 8%. In the composite sample, hardness also decreased with increasing sliding velocity, but the diminishment is not as significant as that of the pure one. Hardness difference between pristine sample and that worn at 1.26 m/s is only about 3%. Before wear hardness condition was almost same for both pure and composite sample.

Average hardness at the composite surface after wear at 0.86 m/s is 95.76 and after wear at 1.26 m/s is 95.55. Literature gives the idea that this slight decrease of hardness may be due to the internal void exposure to the surface after wear or very limited sub surface recrystallization [24]. Increasing sliding speed does not create massive sub surface melting and recrystallization and so the hardness value did not decrease as significant an extent like the pure sample. After wear at 0.43 m/s sliding velocity, hardness values remain almost the same as the before indicating that, 0.43 m/s sliding velocity generates insufficient heat to cause sub surface melting and cannot produce an impact on hardness. And so the wear rate is minimal at 0.43 m/s velocity. Again since hardness value is lowest after wear at 1.26 m/s sliding speed, highest amount of wear occurred during that time, because it is easier for a soft material's surface to get worn out. Hardness data completely acts as the supporting tool to give probable explanation for wear rate relation with sliding velocity.

To see localized melting has occurred or not, micrographic images of the worn sample surface of both pure and composite sample were taken after 8 hours of wear. Figures 4, 5, 6 show these micrographs for 0.43, 0.86 and 1.26 m/s sliding velocities respectively.

A close examination of the micrographs reveals that, after 8 hours of wear, pure sample's surface has undergone moderate wear under dry sliding condition at 0.43 m/s velocity. Wear marks are left in the worn surface in a continuous pattern. With further increase of sliding speed to 0.86 and 1.26 m/s, wear marks become denser. Composite samples on the other hand present no such continuous wear pattern. Increased sliding velocity, nonetheless, did manage to produce more wear marks. From the micrographs, it can be said that localized melting may have occurred due to the heat generation at the pure Nylon interface. At higher sliding velocity, more melting occurred. Due to this, Nylon 6 has shown higher wear rates than composites. Small fragments may have become separated from the specimen's interface. Such detached fragments get attached to the counterface. These molten fragments solidify as a brittle amorphous layer which increases wear severely [25]. But in the composite sample, there is no continuous pattern and very few localized melting may have occurred. Locally molten spot increased with the sliding speed. Decrease in the molten spot also proves that, graphite resulted in improved heat dissipation resulting in less wear.

Surface roughness values of the counterbody wear track were taken at 10 different locations both before and after wear had occurred. Results are shown in figures 7 and 8.

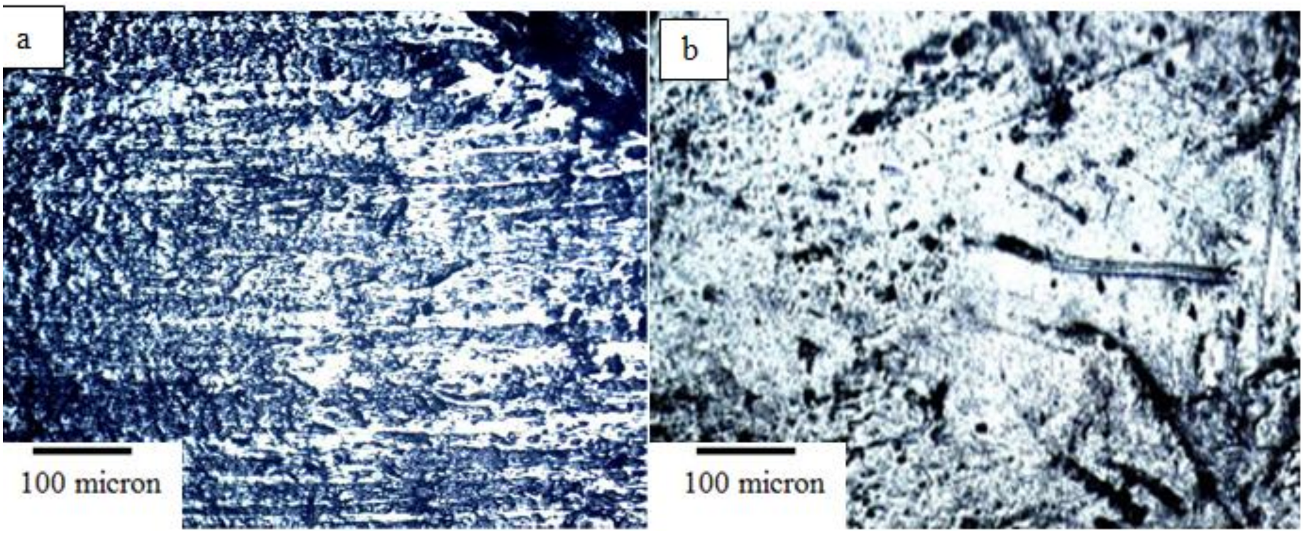

Figure 4. Optical micrograph of the worn surface after 8 hours of wear with disc speed 0.43 m/s of (a) pure (b) composite sample

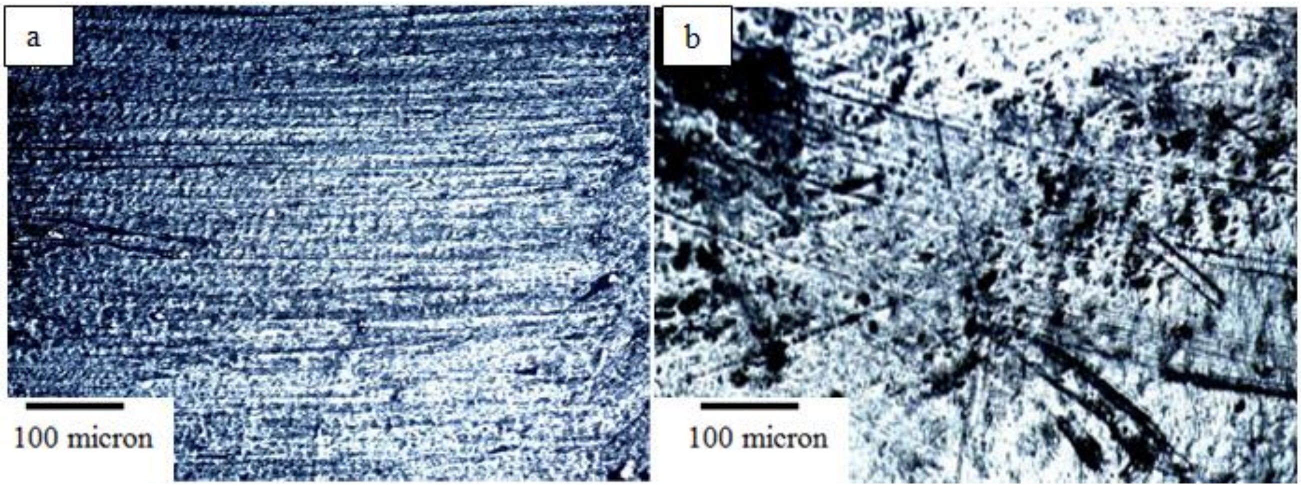

Figure 5. Optical micrograph of the worn surface after 8 hours of wear with disc speed 0.86 m/s of (A) pure (B) composite sample

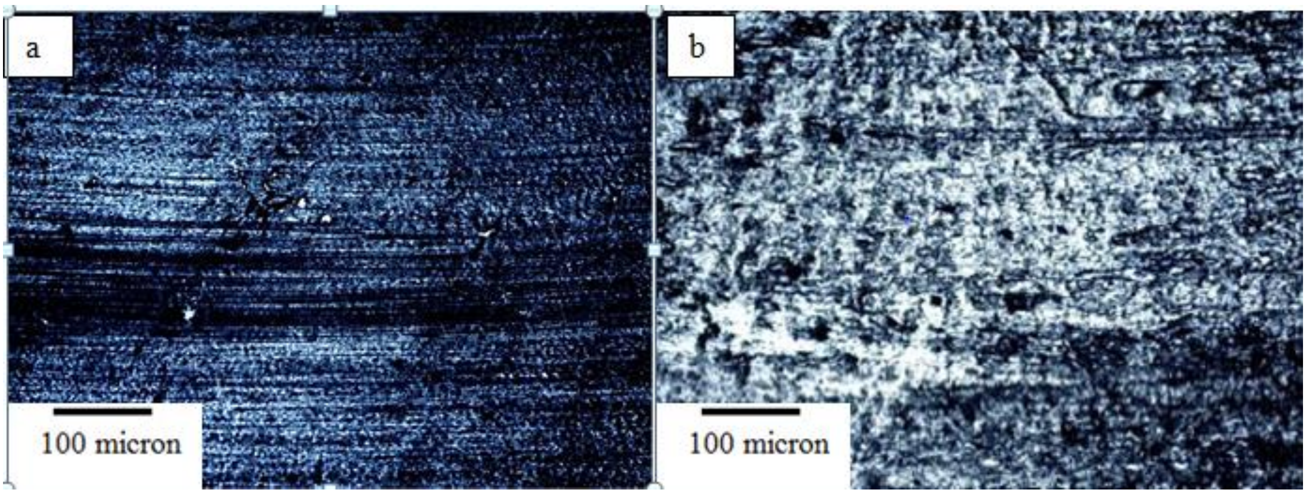

Figure 6. Optical micrograph of the worn surface after 8 hours of wear with disc speed 1.26 m/s of (a) pure (b) composite sample

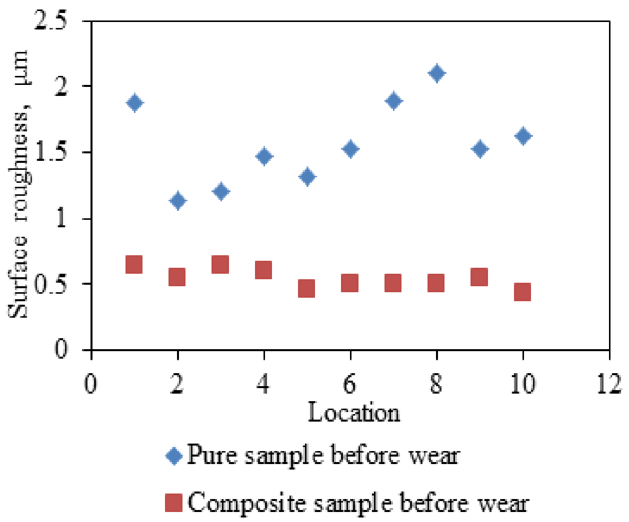

Figure 7. Surface roughness values at various locations of the counter body before wear

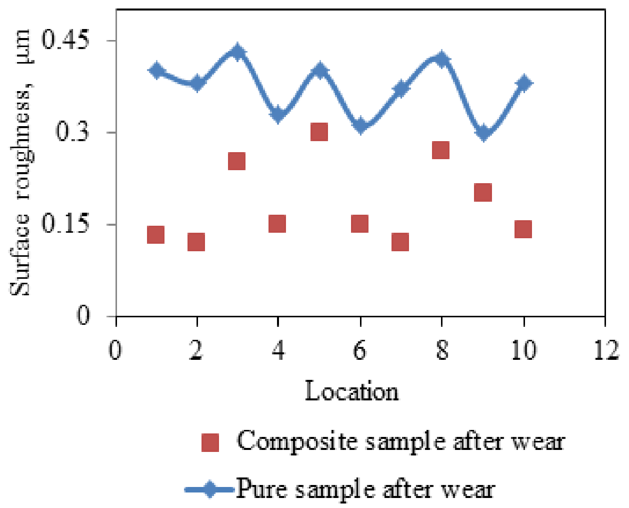

Figure 8: Surface roughness values at various locations of the counter body after wear

Before wear, pure sample had an average roughness of about 1.57 µm and composite had 0.54 µm indicating that composite had a comparatively smoother surface finish. During wear, surface asperities get flattened or are worn out, thus increasing the smoothness of the samples as evidenced by the after wear roughness values; average surface roughness drops to0.37 µm and 0.18µm for pure and composite samples respectively. Surface roughness values play an important role for good tribological properties. From literature, it is clear that higher counter body surface roughness causes greater wear rate [26,27]. Between pure and the composite disk, pure sample had higher average roughness value. That is why; it caused high wear of the pure nylon sample. Roughness values on the wear track after wear also goes with the reasons explained above for the wear rate variation with time and sliding velocity. Pure sample got periodically stuck during sliding along the wear track. During the unsticking process large portions of the material got torn off from the wear track. On the other hand in the case of the composite, the graphite film greatly lessens the chance of adherence occurring between pin and disk resulting in less tearing in the wear track and producing smoother surface finish. That is why, after wear surface roughness value is lower in the composite sample than the pure one.

Conclusions

In this experiment, wear behavior of micron sized graphite powder reinforced nylon composite has been investigated and compared with the wear profile of pure nylon sample. From the experiment, it is found that graphite inclusion creates a lubricating medium between the dry sliding contacting surfaces which acts as a heat dissipating medium and dramatically reduces the friction between the contacting surfaces. As a result, mass loss due to wear is greatly reduced by this self-lubricating nature of graphite. Composite also faces less hardness variation after wear due to heat dissipation. Sliding speed of the counter body significantly affects the wear rate of both pure and composite sample, though the effect is less prominent in the composite sample. Surface of the composite also got smoother with the graphite reinforcement causing lower wear rate. Thus, the tribological property has greatly improved due to graphite particle reinforcement.

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Conflict of interest

Authors declare there are no conflicts of interest.

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