Tribological behaviour of PA6/diborontrioxide composites

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Abstract. PA6 micro composites with different weight percentage of diboron trioxide (B$_2$O$_3$) were fabricated by a twin-screw extruder and analysed for tribological behaviour. The weight percentage of B$_2$O$_3$ were 2, 4, and 8wt% of PA6 matrix. Tribological tests were performed on pin-on-disc apparatus under different loads, sliding distances, and sliding velocities. Results were shown for individual varying parameter as well as with the combination of two and all three varying parameters conditions. Results of the tribological study shows that the PA6 composite with 2wt% diboron trioxide fillers exhibits lowest friction coefficient (COF) and wear rate compare to others and followed by PA6 with 4wt% diboron trioxide filler composite. PA6 with 8wt% diboron trioxide fillers were not found effective in reducing COF and wear rate, infect it increased the COF and wear rate of PA6 and found maximum compare to pure PA6 and PA6 with 2 and 4wt% diboron trioxide fillers.

Keywords: PA6 composite; Diboron Trioxide; Friction; Wear; Solid Lubricant; Tribology

1. Introduction

Polyamides (PA) are having vast application areas like in automobile, electronics, electrical components, and household appliances. PA6 is one of the common subtypes of polyamide family and having pleasant mechanical properties. Lots of research have already done on PA family for further improving their mechanical and tribological properties. According to one research, fibres and particulate reinforcements are advantageous in reducing friction and wear in only dry conditions when rubbing with the smooth surfaces[1]. According to Lalit Guglani and T.C. Gupta’s research, wear, and COF of PA66 was reduced when titanium dioxide fillers were used less than 6wt% and they got the best tribological results for 2wt% fillers [2]. According to the research of Abdullah, Unal, and Yetgin, addition of nano clay in PA6/PP blend improved tribological properties of blended material.COFL with wear rates of PA6/PP blend reduced by the addition of nano clay fillers [3]. Sathees Kumar and Kanagaraj found that the 20wt% graphite enhanced the wear resistance and tribological properties when encompassed in PA6. The addition of 20wt% graphite fillers enhanced the life of pure PA6 by a greater extent according to their research [4]. Yi-Lan You et al. investigated the influence of various fillers on tribological behaviour of PA6. In their results, they found that the glass fibres are more efficient in reducing COF and wear rate compared to talc when used as 15wt% and the COF and wear rate found increased with increasing the load [5]. To achieve better tribological properties of polyamides, various metal oxides were used by the researchers like copper oxide, calcium oxide, zinc
oxide whiskers, aluminium oxide, and nano titanium dioxide [6-10]. Haoyang Sun et al. investigated the influence of new class of solid lubricant i.e. graphite fluoride and fluorographene on tribological properties of PA66 and they found reduction of 37% in COF and 46% in wear rate of PA66 [11]. Various new materials were also used by researchers to improve the tribological properties of polyamides like almond skin powder reduced the wear rate of PA6 by significant amount (453-200 µm) in one research [12]. Sebastian Kamerling et al. found the enhancement in tribological properties after incorporating 20 vol.% magnesium hydroxides in PA66 matrix [13].

Diboron Trioxide is one of the oxides of boron and its crystalline form composed of BO$_3$ triangles. Its 2D properties are like graphite and hexagonal boron nitride i.e. as the force applies, the weak bonds get breaks and BO$_3$ triangles slides over each other. Generally, it is used as a fluxing agent for glass and as a catalyst in organic synthesis like applications. According to one research, the reinforcement of diborox trioxide improved the mechanical properties and abrasion resistance of PA6. Abrasion resistance was improved when diboron trioxide reinforcement was used up to 2wt% and thereafter it found decreased [14]. In one research, 5wt% of diboron trioxide improved the hardness and strength of hydroxyapatite which utilized in human hard tissue implants [15]. In another research, bending strength and hardness of dimond composite were improved by 10 mol.% diboron trioxide reinforcement [16]. As diboron trioxide properties are similar to 2D materials, it can be used as a solid lubricant to provide self-lubrication properties to various polymers. The only difference between the other 2D materials and the diboron trioxide is the melting temperature. As diboron trioxide is having low melting temperature near to 500°C it can effectively be used as a solid lubricant to polymer matrix materials of low melting/processing temperatures as fillers and not in metal matrix materials. The use of diboron trioxide as a self-lubricating agent in polymer matrices is a new area of research and described in this paper with broad discussion. Due to its low melting temperature and self-lubrication properties, various researches been conducted to provide friction reducing coatings on different materials [17, 18].

In this paper, the influence of diboron trioxide on tribological behaviour of PA6 has been studied. Different parameters have been set to evaluate the COF and wear rate of PA6 and PA6 composites. The parameters consist of different loads, sliding distance, and sliding velocities individual and with combinations.

2. Experimental Methods

2.1. Materials
PA6 as a matrix material with 99% purity, 3-6 mm granule size, molecular weight 10,000g/mol and density 1.13-1.15 g/cm$^3$ was selected for experimentation.Diboron trioxide micro-particles with the purity of 99%, average particle size (APS) <50 µm, density 2.46 g/cm$^3$ and molecular weight 69.63 g/mol were selected as fillers in PA6 matrix.PA6 and diboron trioxide micro-particles were imported from nanoresearch elements Inc., USA. Diboron trioxide micro-particles were ball milled to average particle size <50 µm. Table 1 represents the particle size distribution according to the ASTM standards. 98.2% diboron trioxide micro particles passed through 325 mesh while 89.6% micro particles passed through 400 mesh.

| Mesh no. | Mass retained (gram) | Cumulative % retained | Cumulative % passed | Particle size (µm) |
|----------|----------------------|-----------------------|---------------------|--------------------|
| 270      | 0                    | 0%                    | 100%                | 53                 |
| 325      | 18                   | 1.8%                  | 98.2%               | 44                 |
| 400      | 104                  | 10.4%                 | 89.6%               | 37                 |
Figure 1. SEM observation of diboron trioxide micro-particles

‘Figure 1’ shows the morphology of diboron trioxide micro-particles observed by scanning electron microscope (SEM) at x220 magnification. Average particle size observed was approximately less than 50 µm in SEM. Powder SEM characterization of diboron trioxide was carried out at PD Patel Institute of Applied Sciences, CHARUSAT, Gujarat, India.

2.2. Sample Preparation
Compounding of PA6/diboron trioxide composites was carried out with the help of twin-screw extruder with wide kneading block, model high speed torque ZV 20, M/S specific engineering & automats at 250 rpm. PA6 was dried in the oven for 5 hours at 60°C before compounding to remove moisture from it. Barrel temperatures of extruder were adjusted to 200, 210, 220, 230 and 240°C for zone 1, 2, 3, 4 and 5, respectively. Die temperature of extruder was adjusted at 230°C during the process. A water bath was used as the cooling media for extruded material and at the other end, pelletizer was used to convert the string of PA6 composites into pellets. After extrusion, composites pellets were dried again at 60°C for 5 hours to remove moisture from it. Dried pellets were injection molded to fabricate specimen pins for the tribological testing. Compounding and injection molding of PA6 and PA6 composites were performed at CIPET, Gujarat, India. Wide kneading block in twin-screw-extruder with processing at low rpm were selected for enough residence time and better dispersion quality of fillers in PA6 matrix [19]. The filler content used in this research was less than 10wt% i.e. 2, 4 and 8wt% of PA6 to see the effect of small quantity of diboron trioxide on tribological properties of PA6 matrix material.

2.3. Tribological Testing
DUCOM, TR-20LE-PHM-200Pin-on-disk apparatus was used to measure the wear and COF when matting and rubbing with the other surface of hardened EN31 steel metal disc in dry conditions. Diameter of nylon 6 and nylon 6 composite pins was 4 mm with the length of 32 ± 0.2 mm and the diameter of steel plate was 165 mm with the thickness of 8 mm. All tests were carried out at the room temperature of 27°C and on the track diameter of 100 mm. Tests were carried out according to the standard ASTM G99. PA6 and PA6 composite pins matting surfaces with steel were made flat and smooth before the tribological testing and were measured on surface tester. The average surface roughness was found 0.6 Ra of PA6 and PA6 composite pins. Table 2 represents the different test parameters for tribological testing of PA6 and PA6 composites.

Table 2
Table 2. Tribological tests parameters

| Steel disc rotating speed in rpm | Loads | Travel distance |
|--------------------------------|-------|----------------|
| 200                            | 0.5 kg (5 N) | 1 km (1000 metres) |
| 300                            | 1 kg (9.81 N) | 2 km (2000 metres) |
| 400                            | 1.5 kg (14.71 N) | 3 km (3000 metres) |

Full factorial design of experiments method was selected for tribological testing of PA6 and PA6 composites and total numbers of experiments were found 27 for each batch as shown in table 3.

Table 3. Total numbers of experiments by combination of different parameters

| Exp. No. | Combination of parameters | Exp. No. | Combination of parameters | Exp. No. | Combination of parameters |
|----------|---------------------------|----------|---------------------------|----------|---------------------------|
| 1        | 200 rpm – 4.9 N load – 1000 metres travel distance | 10       | 300 rpm – 4.9 N load – 1000 metres travel distance | 19       | 400 rpm – 4.9 N load – 1000 metres travel distance |
| 2        | 200 rpm – 4.9 N load – 2000 metres travel distance | 11       | 300 rpm – 4.9 N load – 2000 metres travel distance | 20       | 400 rpm – 4.9 N load – 2000 metres travel distance |
| 3        | 200 rpm – 4.9 N load – 3000 metres travel distance | 12       | 300 rpm – 4.9 N load – 3000 metres travel distance | 21       | 400 rpm – 4.9 N load – 3000 metres travel distance |
| 4        | 200 rpm – 9.81 N load – 1000 metres travel distance | 13       | 300 rpm – 9.81 N load – 1000 metres travel distance | 22       | 400 rpm – 9.81 N load – 1000 metres travel distance |
| 5        | 200 rpm – 9.81 N load – 2000 metres travel distance | 14       | 300 rpm – 9.81 N load – 2000 metres travel distance | 23       | 400 rpm – 9.81 N load – 2000 metres travel distance |
| 6        | 200 rpm – 9.81 N load – 3000 metres travel distance | 15       | 300 rpm – 9.81 N load – 3000 metres travel distance | 24       | 400 rpm – 9.81 N load – 3000 metres travel distance |
| 7        | 200 rpm – 14.71 N load – 1000 metres travel distance | 16       | 300 rpm – 14.71 N load – 1000 metres travel distance | 25       | 400 rpm – 14.71 N load – 1000 metres travel distance |
| 8        | 200 rpm – 14.71 N load – 2000 metres travel distance | 17       | 300 rpm – 14.71 N load – 2000 metres travel distance | 26       | 400 rpm – 14.71 N load – 2000 metres travel distance |
| 9        | 200 rpm – 14.71 N load – 3000 metres travel distance | 18       | 300 rpm – 14.71 N load – 3000 metres travel distance | 27       | 400 rpm – 14.71 N load – 3000 metres travel distance |

Running time for samples were calculated by the formula 1 –

Total run = circumference of track dia. * rpm * running time

Therefore,

Running time = total run / (2π * rpm)
Total running time for different experiments are shown in table 4. Running time shown in table is in minutes and rounded up.

**Table 4. Running time of experiments**

| Experiment No. | Test running time in minutes | Experiment No. | Test running time in minutes | Experiment No. | Test running time in minutes |
|----------------|-----------------------------|----------------|-----------------------------|----------------|-----------------------------|
| 1              | 16                          | 10             | 11                          | 19             | 8                           |
| 2              | 32                          | 11             | 22                          | 20             | 16                          |
| 3              | 48                          | 12             | 33                          | 21             | 24                          |
| 4              | 16                          | 13             | 11                          | 22             | 8                           |
| 5              | 32                          | 14             | 22                          | 23             | 16                          |
| 6              | 48                          | 15             | 33                          | 24             | 24                          |
| 7              | 16                          | 16             | 11                          | 25             | 8                           |
| 8              | 32                          | 17             | 22                          | 26             | 16                          |
| 9              | 48                          | 18             | 33                          | 27             | 24                          |

Total numbers of material batches were 4 i.e. pure PA6, PA6 with 2wt% fillers of diboron trioxide, PA6 with 4wt% fillers of diboron trioxide and PA6 with 8wt% fillers of diboron trioxide. Total 4 * 27 = 108 numbers of experiments were conducted in this research to investigate the influence of diboron trioxide fillers on tribological properties of PA6. COF and wear rate were directly measured on the wear and friction monitor screen for each pin.

Sliding velocity in meter per second (m/s) calculated by following equation (2) and was found as follows for 200, 300 and 400 rpms:

\[
\text{Sliding Velocity} = \frac{\pi \times \text{Track Diameter} \times \text{RPM}}{60 \times 10^3} = \frac{\pi \times 10^3}{60000} = 1.05 \text{ m/s}
\]

Similarly, for 300 and 400 rpm it was found 1.57 m/s and 2.1 m/s respectively.

3. Results and Discussion

3.1. COF

3.1.1 COF vs load/ sliding distance/ sliding velocity
Figure 2. COF vs (a) sliding distance (b) Load (c) sliding velocity

‘Figure 2 (a)’ represents COF of PA6 and PA6/diboron trioxide composites at different sliding distances of 1000, 2000 and 3000 metres at which load and sliding velocity were made constant and of 4.9 N and 1.05 m/s, respectively. As shown in figure, PA6 with 2 and 4wt% diboron trioxide filler exhibits lowest COF compared to pure PA6 and PA6 with 8wt% diboron trioxide fillers. PA6 with 2wt% diboron trioxide shows superior performance under all sliding distances while PA6 with 8wt% diboron trioxide exhibits higher COF compared to others at all sliding distances. ‘Figure 2 (b)’ represents COF of PA6 and PA6/diboron trioxide composites at different loads of 4.9, 9.81 and 14.71 N. In this, sliding velocity and sliding distance were made constant and of 1.05 m/s and 1000 metres, respectively. PA6 with 2wt% diboron trioxide shown better performance under different loading conditions followed by PA6 with 4wt% diboron trioxide composite. PA6/diboron trioxide composite with 8wt% filler content exhibits highest COF compared to others and not found effective in reducing the COF of PA6. ‘Figure 2 (c)’ represents COF of PA6 and PA6 composites at different sliding velocities of 1.05, 1.57 and 2.1 m/s where load and sliding distance were made constant and of 4.9 N and 1000 metres, respectively. In this, 2wt% filler PA6 composite shown reduced COF compared to PA6 at all sliding distances while 4wt% filler composite shown improvement in performance at higher sliding velocities and reduction of COF was witnessed at sliding velocity higher than 1.57 m/s. PA6 with 2wt% diboron trioxide filler composite shown lowest COF in all three variable parameters of sliding distances, load and sliding velocities which was followed by 4wt% diboron trioxide filler PA6 composite. As the load, sliding distance and velocity increases, the COF of PA6 and PA6 composites also found increasing.

3.1.2 COF at combinations of different parameters
Figure 3. COF at (a) constant velocity and varying load & sliding distance (b) constant sliding distance and varying load & sliding velocity (c) constant load and varying sliding distance & velocity (d) varying all parameters i.e. load, sliding distance & velocity

‘Figure 3 (a)’ represents COF of PA6 and PA6 composite at varying loads and sliding distances while sliding velocity was made constant and of 1.05 m/s. ‘Figure 3 (b)’ represents COF of PA6 and PA6 composite at varying loads and sliding velocities while sliding distance was made constant and of 1000 metres. And ‘figure 3 (c)’ represents the COF of materials at constant load of 4.9 N with varying sliding distances and velocities. ‘Figure 3 (d)’ represents the COF when load, sliding distances and sliding velocities, all varied of PA6 and PA6 composites. In all four cases, as shown in diagrams, 2wt% filler PA6 composites exhibits lowest COF followed by 4wt% filler PA6 composite. In ‘figure 3 (c)’ where load was constant and sliding distance and velocity was varying, the 2 and 4 wt% filler composites almost shown same lowest COF. PA6 with 8wt% diboron trioxide fillers not found effective in reducing the COF and found maximum compare to other materials. COF of PA6 with 2wt% diboron trioxide found approx 10 to 12 % less than pure PA6.

The increased value of COF of PA6 with 8wt% diboron trioxide filler was due to the dislodged debris between the contact surfaces of pin and rotating steel disc. Also, as the load, sliding distance and velocity increased, the dislodged debris quantity also increased between the contact surfaces of pin and steel disc. That is the reason of increasing COF of PA6 and PA6 composites at higher sliding velocities, distances and loads and it found almost linearly increasing at increasing different parameters. The erosion of the constituents of PA6 and PA6 composite pins at higher load, sliding velocities and distances was found the main reason for more debris. According to mihai tiberiu et al., the value of COF of polyamides increases with increasing rate of load, temperature and sliding velocities which was also witnessed in our study [20].

3.2. Wear

3.2.1 Wear vs load/ sliding distance/ sliding velocity
Figure 4. Wear vs (a) Load (b) sliding distance (c) sliding velocity

‘Figure 4 (a)’ represents the wear rate in micrometres at different loads of 4.9 N, 9.81 N and 14.71 N where sliding distance and velocity were made constant and of 1.05 m/s and 1000 metres, respectively. ‘Figure 4 (b)’ shows the wear rate at different sliding distances of 1000, 2000 and 3000 metres where load and sliding velocity were made constant and of 4.9 N and 1.05 m/s, respectively. ‘Figure 4 (c)’ represents the wear rate at different sliding velocities of 1.05, 1.57 and 2.1 m/s where sliding distance and load were made constant and of 1000 metres and 4.9 N, respectively. In all three conditions of parameters, as shown in figures, 2wt% and 4wt% diboron trioxide fillers PA6 composites exhibited lower wear compared to pure PA6 and PA6 with 8wt% diboron trioxide fillers. As the load, sliding distance and velocity increases, the wear of PA6 and PA6 composites also found increasing.

3.2.2 Wear at combinations of different parameters
Figure 5. Wear at (a) constant velocity and varying load & sliding distance (b) constant sliding distance and varying load & sliding velocity (c) constant load and varying sliding distance & velocity (d) varying all parameters i.e. load, sliding distance & velocity

‘Figure 5 (a)’ represents wear rate of PA6 and PA6 composite at varying load and sliding distance while sliding velocity was made constant and of 1.05 m/s. ‘Figure 5 (b)’ represents wear of PA6 and PA6 composite at varying load and sliding velocities while sliding distance was made constant and of 1000 metres. And ‘figure 5 (c)’ represents the wear of materials at constant load of 4.9 N load with varying sliding distance and velocity. ‘Figure 5 (d)’ represents the wear rate of materials when load, sliding distances and sliding velocities, all varied. In all four cases, as shown in diagrams, 2wt% filler PA6 composites exhibit lowest wear followed by 4wt% filler PA6 composite. In figure 5 (b) and 5 (c), the 2 and 4 wt% filler composites almost shown same minimum wear rate. PA6 with 8wt% diboron trioxide fillers not found effective in reducing the wear and found maximum compare to pure PA6 and PA6 with 2 and 4wt% diboron trioxide filler composites. The wear rate of 2wt% diboron trioxide filler PA6 composite was found approx 12 to 15% less than pure PA6. Wear rate of PA6 with 8wt% diboron trioxide filler was found high due to the high value of COF. At high sliding velocities, sliding distances and loading conditions, the contact surface temperatures of pins increases which results in reduction of mechanical cohesion and erode the constituents of materials which cause more wear [21, 22].

‘Figure 6’ represents the transfer layer generation during the tribological testing of PA6 composite pins. In 2 and 4wt% diboron trioxide fillers PA6 composites, the generated transfer layer was smooth and consistent with less burrs around the track diameters. While in case of 8wt% diboron trioxide filler PA6 composite, the transfer layer was not found smooth and lots of burrs were found near the track diameter during testing. Pure PA6 was not showing any smooth layer generation but scratch marks with very low intensity and with few burrs were witnessed during tribological testing of it.

Figure 6. Transfer layer generation on the steel disc of pin-on-disc apparatus
As the transfer layer of lubricating film getting smooth, the COF of the materials got reduced compared to initial reading of few fraction of seconds to the letter on. Higher loads, higher RPMs (sliding velocities), and higher travelling distances cause heating of the contact area of pin and rotating steel disc which reduced the mechanical cohesion strength of the material. Due to this, material from the pins got eroded and can be seen as debris or burs between the contacts of pins and steel disc and around the track diameter of testing. This cause increase in COF as well as wear rate of materials.

4. Conclusion

Diboron trioxide fillers improved the tribological properties of PA6 matrix when used up to 4wt%. The best results of COF and wear resistance were achieved with 2wt% filler content. Smooth transfer layer generation of lubricating film was found the probable reason for the effectiveness of 2, and 4wt% diboron trioxide filler PA6 composites in improving tribological properties of PA6.

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