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Investigations of the influence of hexagonal boron nitride particulates on mechanical & tribological properties of PA66

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Investigations of the influence of hexagonal boron nitride particulates on mechanical & tribological properties of PA66

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Abstract : The present research investigates the influence of hexagonal boron nitride (h-BN) particulates on PA66 composite's mechanical and tribological properties. The compounding of 5wt% h-BN and PA66 matrix was done with the help of twin-screw extruder. Specimens of pure PA66 and PA66/h-BN composites were prepared by the injection moulding process. Mechanical and tribological properties i.e. tensile strength, modulus of elasticity, Rockwell hardness, friction coefficient and wear rates were measured in this research to quantify the different properties of materials. Results shows that the modulus of elasticity and Rockwell hardness of composites were improved while the tensile strength was found decreased in negligible amount compare to pure PA66. The h-BN fillers were found to be effective in reducing wear and friction of PA66/h-BN composite in tribological testing.

Keywords: PA66; Hexagonal Boron Nitride; Extrusion; Mechanical &Tribological properties; Solid lubricant

1. INTRODUCTION

In this modern world, plastics are an essential part of our daily lives. Plastics are having very versatile applications from day to day life housewares to defence, aerospace, automobiles and in many more sectors. In an automobile industry now a days 40 % of parts are made of plastics. Polymers and polymer matrix composites proved to be effective in tribological applications due to low friction, wear, and self-lubricating properties achieved by solid lubricants like in gears, cams, bearings, and seals of components. There are various types of polyamides (PA) available in the market e.g. PA6, PA66, PA11, PA12, PA1010 and so on in which PA66 is having combinations of high mechanical & thermal properties with chemical inertness & easy processing. Many researchers have worked to improve the mechanical and tribological properties of polyamides by adding various fillers. Various organic and inorganic oxides, sulphides and fluorides were proved to be effective friction modifiers of polyamides such as Al_2O_3, TiO_2,CaO, B_2O_3, ZnS, PbS,CaS,MoS_2, WS_2, ZnF_2 and CaF_2[1-5].In one research, addition of modified montmorillonite found effective in enhancing young's modulus and tensile strength of PA66 [6].In another research, Silicon oxide (SiO2) nanoparticles of 2wt% improved tensile strength, hardness, and thermal stability of nylon 6 [7]. Clay particles as fillers in micro and nano form were used by many researchers for example, 5% nano clay particles found to be effective in enhancing tensile and flexural strength of PA6 [8]. Carbon nanotubes were also used by researchers to improve various mechanical and tribological properties of materials like in one research, 1wt% carbon nanotubes enhanced tensile strength, tensile modulus, and hardness of PA6 material by a good amount [9]. Sathees Kumar et al. found that the 20wt% graphite fillers improved the wear resistance and tribological properties of PA6. [10]. Yi-Lan You et al. found that the 15wt% glass fibres are effective in improving COF and wear rate of PA6 [11]. Haoyang Sun et al. researched on the influence of graphite fluoride and fluorographene on tribological properties of PA66 and found improvement in COF & wear rate as 37% and 46%, respectively [12]. Almond skin powder improved the wear resistance of PA6 by a good amount in one research [13]. 20 vol.% magnesium hydroxides in PA66 matrix improved the tribological properties of PA66 according to Sebastian Kamerling et al.'s research [14]. According to H. Unal et al.'s research, tensile strength, and modulus of elasticity of PA6 were enhanced with the increase of talk and kaolin filler % while on the other hand, impact strength and % elongation was found decreased due to fillers. Optimum % of fillers were found in their study was 10 to 15% [15]. In Isabel Claveria et al.'s research, 5% graphene enhanced the tribological properties of PA66/30% glass fibre hybrid composite. Also, they indicate that the addition of zirconium dioxide was not at all effective in enhancing tribological properties of PA66/glass fibre hybrid composite [16].

Hexagonal boron nitride (h-BN) is a compound of boron and nitrogen and is used in many industrial sectors and in various machine parts seals due to its low thermal expansion & high thermal conductivity [17]. A small amount of BN can enhance various thermal, mechanical and tribological properties of materials according to the research [18]. H-BN can also be used in combination with other solid lubricant to improve the tribological behaviour of any system [19]. Due to its self-lubrication properties h-BN is often used in lipsticks, pencil leads and in various paints [20-22].

In this research, the effect of h-BN fillers on mechanical and tribological properties of PA66 has been studied. Tensile strength, modulus of elasticity, % elongation and Rockwell hardness were measured to identify the mechanical performance of PA66 and PA66/h-BN composite. Coefficient of friction (COF) and wear rates were measuredon pin-on disc tribometer to check the tribological performance of materials.

2. EXPERIMENTAL METHODS

2.1 Materials

PA66 with 1.314 g/cm^{3}density, 224.3 g/mol molecular weight, KDP1000 KOPLA, was selected as a matrix material in the form of 3-5 mm granules. The h-BN fillers wereselected in the form of fine micro-particles with an average particle size of <15µm and purity of 99.98 %. 'Figure 1' represents the SEM image of h-BN micro-particles at x600 magnification. FTIR test confirms the chemical composition of fillers as boron nitride as shown in 'figure 2' with high intensity peaks at 854, 872, 1486, 2850 and 2918cm^{-1}.

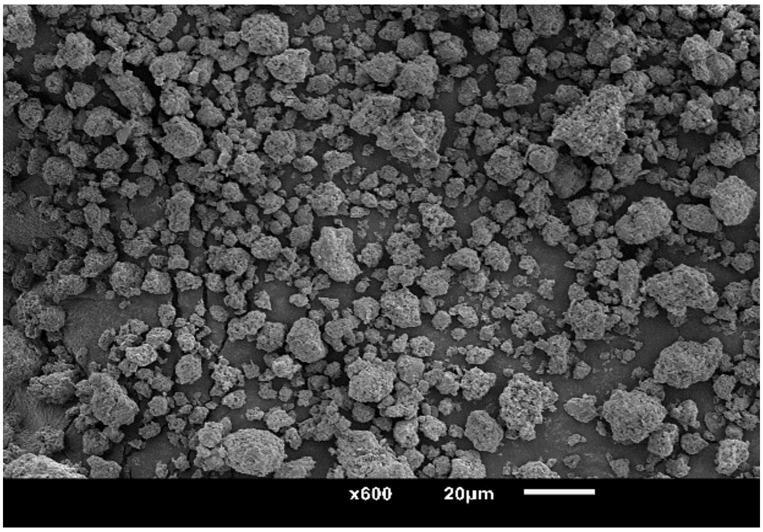

Figure 1. SEM analysis of h-BN micro-particles

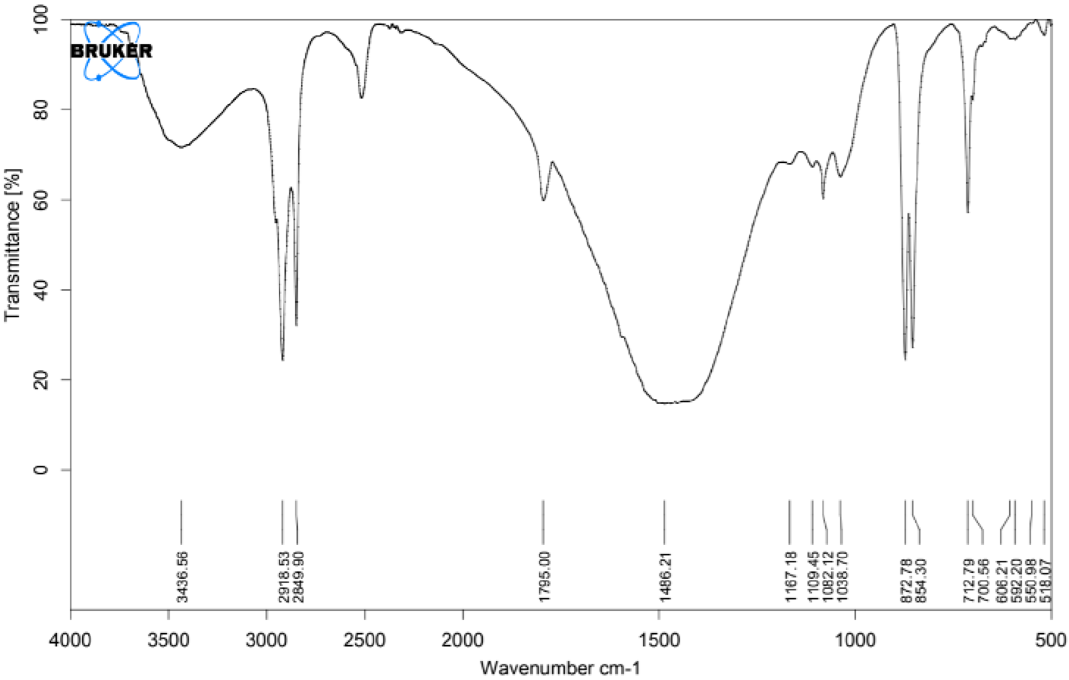

Figure 2. FTIR spectra of h-BN micro-particles

2.2. Sample Preparation

Compounding of PA66/h-BN in the wt% of 95/5 was carried out with the help of twin-screw-extruder, high-speed torque ZV20 model, M/S specific engineering & automats, at room temperature. The major benefit of using twin-screw extruder is that it is less prone to the agglomeration of the particles. The temperature profiles ofsix different zones Z1, Z2, Z3, Z4, Z5, & die were set at 240°C, 240°C, 250°C, 250°C, 270°C & 255°C, respectively. Screws of extruder were rotating at 348 RPM with low feed rate. Melting temperature and pressure were 254 °C and 12 bar respectively during the extrusion process. PA66 granules were dried in the oven before and after the extrusion process at 85 °C for 4 hours to remove any moisture from it. To fabricate the testing specimens, injection mouldings of granules were done with the help of EPK 180 injection moulding machine at room temperatures. Granules were feed into the injection moulding machine with the help of hopper. Screw of injection moulding machine was heated in the range of 250- 270 °C.

2.3. Mechanical & Tribological testing

Average tensile strength along with the elastic modulus and % elongation was noted after five specimens testingon universal testing machine,TINIUS OLSEN /L-SERIES H50KL model, according to ASTM D638 standards. Type-I sample specifications were selected for the tensile testing andall samples were tested at 5 mm/min speed.

Average Rockwell hardness afterfive specimens testing wasnoted on Rockwell hardness tester available at CIPET-Ahmedabad, according to the ASTM D 785 standard.Disks of diameter 100 mm with 3 mm thickness were the sample specifications for the hardness test. ‘Figure 3’ shows the tensile and Rockwell test specimens.

Tribological tests wereperformed on pin-on-disk tribometer, DUCOM TR-20LE-200PHM model, according to ASTM G99 standards. No external lubrication was used in the tribological testing and all tests were done at room temperature of 29^0C with relative humidity value of 40%. 30 mm length and 4 mm diameter were the specifications of samples which were mounted against the hardened EN31 steel disc. 100 mm track diameter was selected for all the experiments and surface roughness values of pins at the contact point with steel disc were maintained at 0.5-0.6 Ra before mounting on the pin-on-disc tribometer.

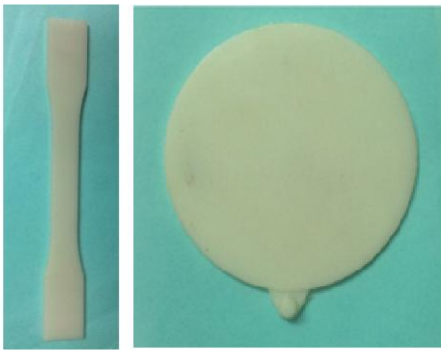

Figure 3. Mechanical testing specimens

All tribological tests were performed for constant 1000 m run with varying RPM and loads. RPMs were 100, 200 & 300 and loadswere 1, and 2kg in the different experimentations. Average value of friction coefficient (COF) and wear rate was noted after five pin specimens testing. Sliding velocitiesfor different rpms were calculated from the equation 1 and was found 31 mins, 17 mins, and 11 mins for 100, 200 and 300 rpm, respectively. COF and wear rates of pins were directly measured on friction and wear monitor screen of tribometer.

$$v(m/s) = \frac{\pi \times (track\ \ diameter\ \ \ in\ mm) \times RPM}{60{,}000} \qquad (1)$$

3. RESULTS AND DISCUSSION

3.1. Mechanical properties

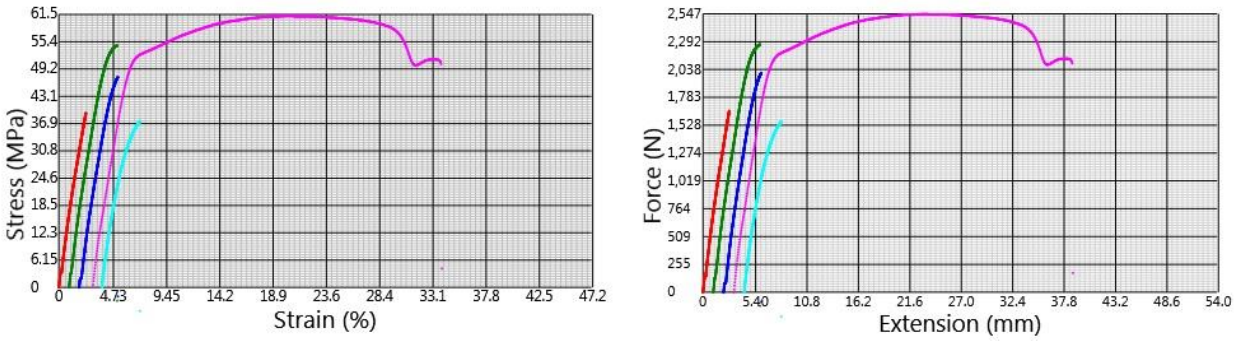

Figure 4. Tensile testing of PA66

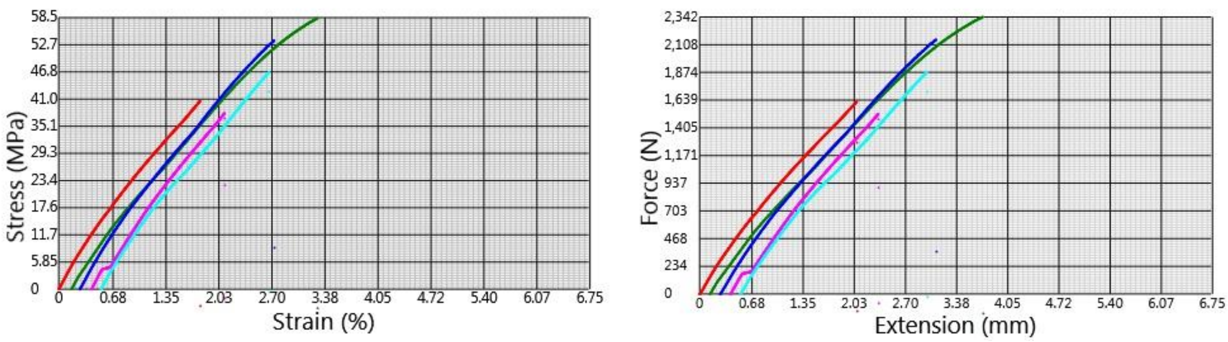

Figure 5. Tensile testing of PA66/h-BN composite

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After testing five samples, slight decrease in average tensile strength of PA66/h-BN composite was observed compare to pure PA66 which was 1.25 % i.e. 0.6 MPa as shown in ‘figure 6 (a)’. Average tensile strength of Pure PA66 and PA66/h-BN composite noticed was 48 and 47.4 MPa, respectively. Average modulus of elasticity was noticed 2010 and 2820 MPa of PA66 and PA66/h-BN composite, respectively which indicates increase of 40.3% modulus of elasticity due to h-BN fillers.% elongation was observed 6.2 and 2.25% of PA66 and PA66 composite, respectively. Decrease of 3.95% in elongation was witnessed during tensile testing of materials. ‘figure 4’ and 5’ represents the stress-strain and force vs extension results of PA66 and its composites, respectively.‘Figure 7’ shows the cross-section of tensile fractured surface of PA66/h-BN composite. Stretching of the matrix material is clearly visible in the figure with the small white dots of h-BN fillers. H-BN fillers were wrapped into the PA66 matrix which were loose out on to the fractured surface due to the tensile force on the specimen.

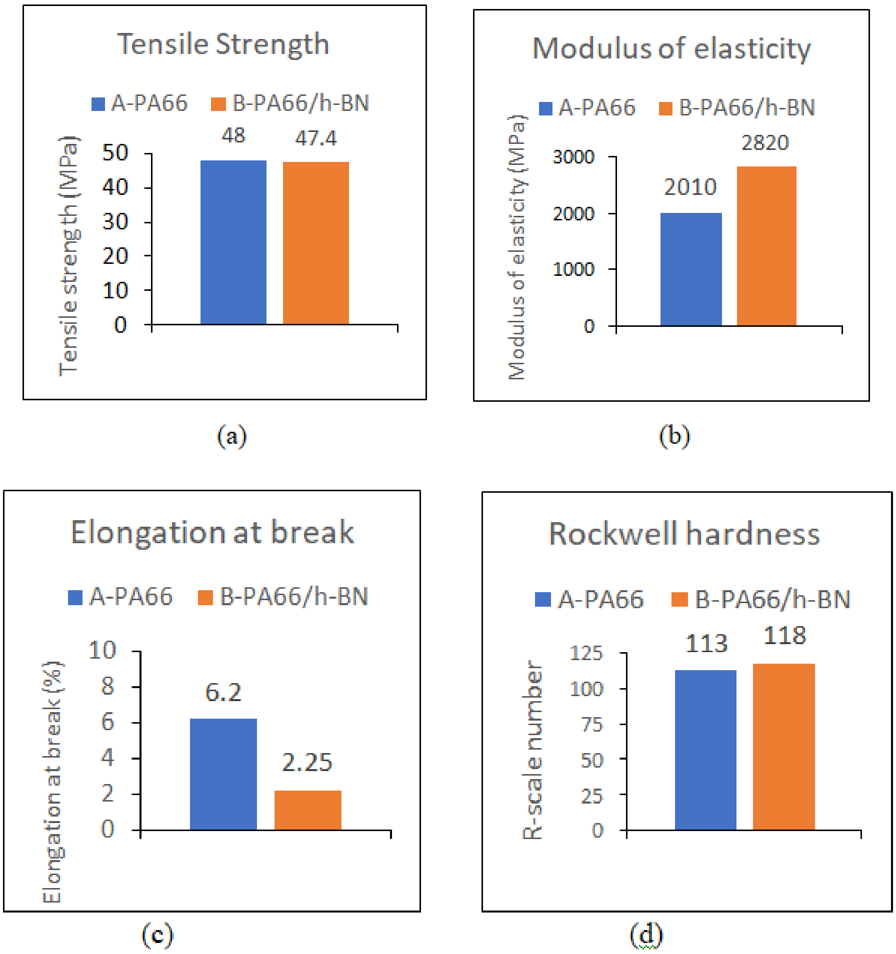

Figure 6. Mechanical testing results of PA66 and PA66/h-BN composite (a) tensile strength (b) modulus of elasticity (c) % elongation (d) Rockwell hardness

As the elongation decreased the hardness of the material was found increased of composite which was also observed in one research where 5-10% h-BN fillers in PTFE matrix enhanced the hardness of the material up to few percentages [23]. Average Rockwell hardness of 113 and 118 was observed of PA66 and PA66/h-BN composite respectively on R-scale after five samples experimentation. Increase of 4.43% in hardness was observed of PA66 composite compare to pure PA66. Table 1 represents the average mechanical testing results of PA66 and PA66/h-BN composite after five samples testing.

Table 1. Tensile and hardness test results

Sr. No.	Test name	Pure PA66	PA66/h-BN Composite
1	Tensile strength (MPa)	48	47.4
2	Elongation (%)	6.20	2.25
3	Modulus of elasticity (MPa)	2010	2820
4	Rockwell hardness (R-scale)	113	118

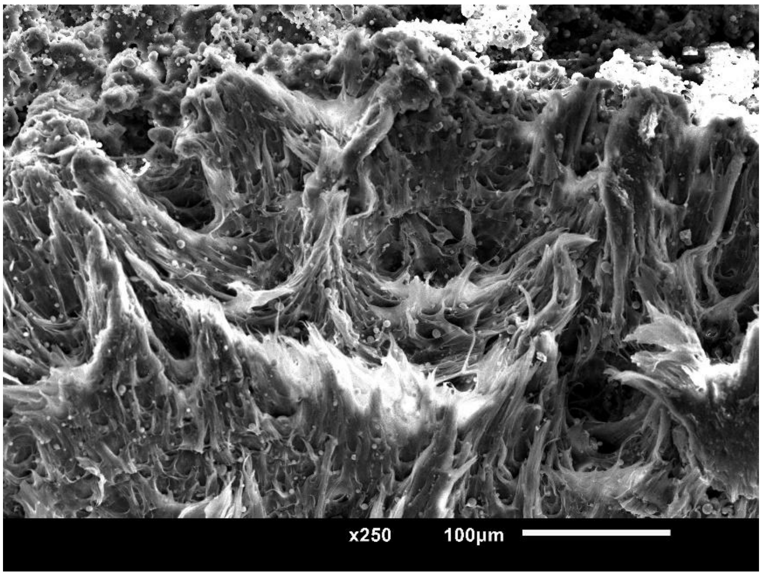

Figure7. SEM image of tensile fracture of PA66/h-BN composite

3.2 .Tribological test results

'Figure 8' represents the obtained average COF values after five samples tribological testing of materials. COF and wear testing of the samples were done by varying parameters and total 3 distinctive parameters were set to observe the COF and wear rates of the materials. First parameter was set at 100 rpm, 1 kg (9.81 N) load and sliding duration of 31 minutes. Second parameter was set at 200 rpm, 1 kg load and sliding duration of 17 minutes. And third parameter was adjusted at 300 rpm, 2 kg load and 11 minutes sliding duration. Track diameter was 100 mm in all three parameters and sliding duration was adjusted to as mentioned for different parameters to complete 1km run in all experimentations. These parameters were selected to see the material's tribological behaviour on different sliding velocities and at different loading conditions. 'Figure 9' represents the average wear rates after five samples tribological testing of materials with the mentioned parameters. As shown in the figure, average COF of 0.3534 and 0.2946 was obtained of PA66 and PA66 composite for parameter one. Decrease of 16.64% in COF was observed for parameter one due to h-BN fillers. In second parameter, 0.3845 and 0.3637 were the obtained average COF of PA66 and PA66 composite, respectively. Decrease of 5.5% in COF was noticed for parameter 2. Similarly, 0.4206 and 0.3776 were the average COF noticed after five samples testing of PA66 and PA66 composites, respectively. Improvement of 10.23 % in COF was observed for parameter three. By analysing the results, it was witnessed that, at lower sliding velocity and low load the improvement in COF was much better of PA66 composite compare to pure PA66. As the sliding velocity increased, the small difference in the COF of materials were witnessed. As the sliding velocity and load both increased, the COF of PA66/h-BN composite was improved double compare to second parameter but was observed less than first parameter. H-BN fillers were found less effective as the sliding velocity increased but found more efficient when load increased. This is due to the smooth transfer layer formation of lubricating film during first parameter of low sliding velocity and at third parameter of high load. In second parameter, the transfer film observed was not smooth and lots of debris were noticed between the pin specimens and rotating steel disc and also near the track diameter which may be the probable reason for higher COF compare to other parameters. According to mihaitiberiu et al.'s research, COF of PAs increases with the increasing rate of loads and sliding velocities and it was also seen in our study [24]. As the sliding velocities and load increased, the COF of PA66 and PA66/h-BN composite was found increased.

As shown in 'figure 9', average wear rates of 33.8 µm and 21.6 µm were obtained of PA66 and PA66 composite for parameter one. Decrease of 36% in wear rate was observed for parameter one due to h-BN fillers. In second parameter, 23 and 16.2 µm were the obtained average wear rates of PA66 and PA66 composites, respectively. Decrease of 29.57% in wear was noticed for parameter 2. Similarly, 28.67 and 23 were the average wear rate noticed after five samples experimentation of PA66 and PA66 composite, respectively. Improvement of 19.78% in wear rate was observed for parameter three.Improvement of wear resistance was probably due to the improved COF of PA66/h-BN composite at all three parameters compare to pure PA66. Table 2 represents the test results of COF and wear rates at different test parameters of tribological testing.

Table 2. Tribological test results

Sr. No.	Parameters [Track diameter = 100 mm; Sliding distance = 1000 m]			Pure PA66		PA66/h-BN Composite	
	RPM	Load (kg)	Duration (min)	COF	Wear (µm)	COF	Wear (µm)
1	100	1	31	0.3534	33.8	0.2946	21.6
2	200	1	17	0.384	23	0.3582	16.2
3	300	2	11	0.4206	28.6	0.3776	23

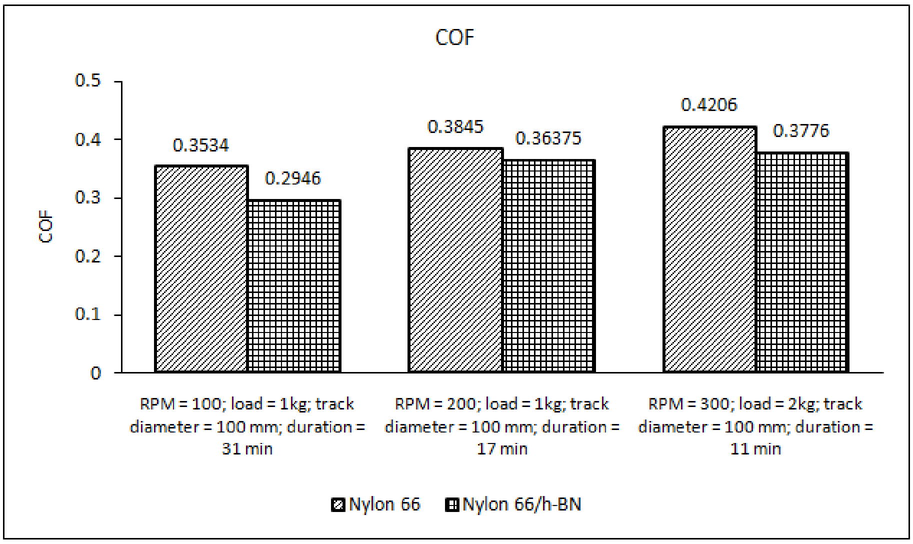

Figure 8. COF of materials at different parameters

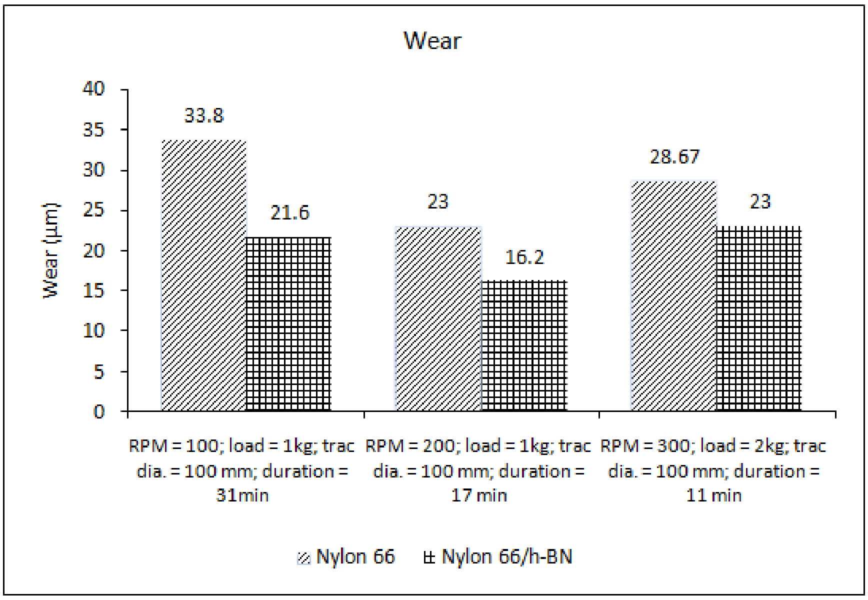

Figure 9. Wear rates of materials at different parameters

From 'figure 8' it was witnessed that, as the sliding velocity and load increases, the COF of pure PA66 and PA66 composite also increases. The wear rates of PA66 and PA66 composites found decreased with the increase in sliding velocity which increased after further increase in sliding velocity with load as shown in 'figure 9'. At 200 RPMs, COF of materials were found higher but the wear rates werestill found lower compare to the 100 RPM results of materials. The probable reason behind that was the same distance travelled with the higher (double) sliding velocities compare to previous one which reduced the sliding time as well as the wear of composite. As the sliding velocities and load further increased, the highest COF were observed of PA66 and PA66/h-BN composite. Also the wear rates of materials were found slightly increased compare to parameter 2 which probably due to the temperature rise between the pins and steel disc contact point. Rise in temperature results in the reduction of mechanical cohesion and in the erosion of the constituents of pure PA66 and its composites and ultimately in more wear [25, 26].

4. CONCLUSIONS

Inclusion of 5wt% h-BN micro particulates in PA66 matrix leads to the following conclusions:

- Almost no effect on tensile strength was observed and increase of 40.3% of modulus of elasticity was observed of PA66/h-BN composite compare to pure PA66.
- 4.43% improvement in hardness was witnessed of PA66/h-BN composite compare to pure PA66.
- The h-BN particulates improved the COF and wear resistance at all three sets of parameters by several percentages.
- Smooth transfer layer formation of lubricating film was found the main reason for improvement in COF and wear resistance of PA66/h-BN composites.

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