

Mechanical and Tribological Behavior of Particulate Filled Silicon Nitride Reinforced Nylon-6 Polymer Composites



K. Shiva Kumar, A. Chennakesava Reddy

Abstract: From the Research, it found that there is an impact of Silicon Nitride (Si_3N_4) on the mechanical properties of Nylon 6 or polyamide 6 based composites. Nylon 6 prepared with Silicon Nitride (Si_3N_4) by changing the weight proportions. The hardness and wear properties of Nylon-6/ Si_3N_4 composites have investigated. Experiments were carried out as per Taguchi's design. Rockwell, hardness testing device, used to observe the hardness number of different nylon-6/ Si_3N_4 composites and the pin-on-disc wear test (ASTM G99) conducted with different combinations of reinforcement, sliding distance, sliding speed and normal load. Scanning electron microscopy (SEM) was used to look at the break surfaces microstructure of wear and tensile tests. The increase of Si_3N_4 upgrades the existence state of typical Nylon 6 to a more important point.

Keywords: Nylon-6, Si_3N_4 , Mechanical properties, Tensile, Hardness, Wear, SEM.

I. INTRODUCTION

Composite materials currently play a major role in a lot of applications, such as the automobile industry and aerospace applications. Polyamides are very important for designing thermoplastics utilized in modern applications such as instrument, sensor housing, packing film, bundling of gadgets, and furniture-polymer composites employed in plastic manufacturing because of their excellent strengths and low densities. The development of Nylon 6/Teflon as a matrix material and graphite as a filler is carried out material to produce thrust washer and sleeve bearing [1, 2]. Mostly short fiber reinforced thermoplastic composites is frequently used in several fields such as aerospace or aircraft and automotive industry [3-5]. The mechanical characteristics of thermoplastic polymers moreover improved with the addition of different filler materials. Polymers such as Nylon-66,6 when mixed with a suitable percentage of nanoparticles as filler materials show significant improvements such as SiC [6], Al_2O_3 [7], γ -Fe $2O_3$ [8] and used with different polymers composites.

The tensile tests explain that the modulus and yield strength of polyamide 6/layered-silicate nanocomposites (PLSN), Si_3N_4 increase with an increase in filler content [9 10].

Mechanical test outcomes show that rising carbon fiber content gives better modulus, tensile strength, and hardness but decreased strain at break values of the composite [11]. The wear performs increase with an increase in a volume fraction of different nanoparticles in different matrix materials [12-15].

In this present work, Silicon Nitride (Si_3N_4) nanoparticles filled with Nylon-6 polymer matrix composites have been investigated for varying filler content, sliding distance, normal load and sliding speed with three levels of each parameter as per Taguchi's design of experiments. The morphology of wear track also calculated and optimum circumstances of the parameters after the tribological test utilizing SEM microstructures.

II. MATERIALS AND EXPERIMENTAL METHODOLOGY

The Nylon-6 polymer matrix filled with nanoparticles of silicon nitride (Si_3N_4) whose size is 100nm. Varying different percentages of Si_3N_4 is added to Nylon-6 with 4%, 8%, 12%, 16% and 20% by weight to get better mechanical properties of the polymer composites. The injection molding machine was utilized to produce Nylon-6/ Si_3N_4 polymer composites. The blend of Nylon-6 and Si_3N_4 nanoparticles put in a hopper. The Nylon-6 warmed in the barrel with the goal that it should end up molten and delicate. The combination of Nylon-6 and Si_3N_4 nanoparticles then constrained under pressure inside a shaped cavity where it subjected to holding weight for a specific time to make up for material shrinkage. The temperature of the mold was 25 °C for all trials. After the material solidified in the mold, the sample ejected. The measurements of tensile examples shown in Fig.1. Tensometer Model PC-2000 (Fig.2) utilized for tensile tests. A scanning electron microscope (SEM) is utilized to watch microstructures at room temperature to research the brake surfaces of specimens.

A. Sliding Wear Test

The pin-on-disc type friction monitor and emery paper grit of size 400 used for conducting the sliding wear test. The disc made of EN31 hardened steel having a track radius of 80mm. The wear test was carried out at different speeds of 100, 200, and 300 rpm. Figure 2 shows the injection-molded specimens.

Revised Manuscript Received on October 30, 2019.

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Also, the sliding wear test performed under different normal loading 10N, 15N and 20N. The tests were performed for different sliding distances of 500, 750 and 1000 m.



Fig.1: Tensometer

Table 1. Levels of Control parameters

Factor	Symbol	Level-1	Level-2	Level-3
Si ₃ N ₄ %wt.	A	4	12	20
Normal Load, N	B	10	15	20
Sliding Speed, rpm	C	100	200	300
Sliding distance, m	D	500	750	1000

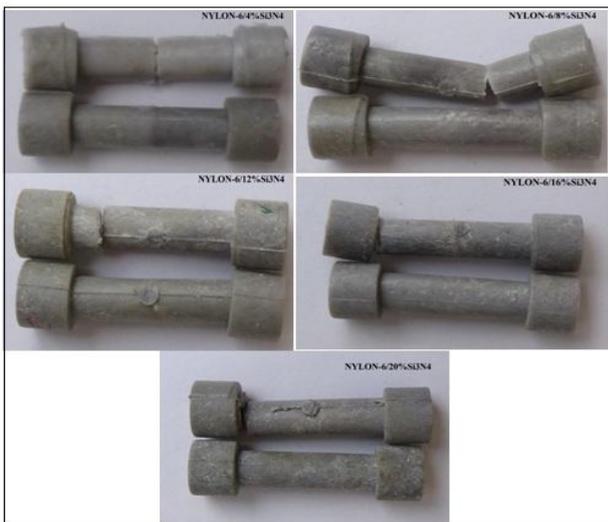


Fig.2: Tensile specimens of Nylon-6/Si₃N₄

Table 2. Orthogonal array (L9) and control parameters

Treat No.	A	B	C	D
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

III. RESULTS AND DISCUSSION

A. Tensile Strength

From Fig.3, it found that there is an effect in the edition of Si₃N₄. The tensile strength is higher when the percentage of Si₃N₄ is 4%wt later with an increase in the percentage of Si₃N₄ there is a decrease in the tensile strength. For the 12 %wt of Si₃N₄, there is least tensile strength. Tensile strength is minimum when we consider pure Nylon-6. So, to enhance the properties of the Nylon -6 with Si₃N₄ 4 %wt.

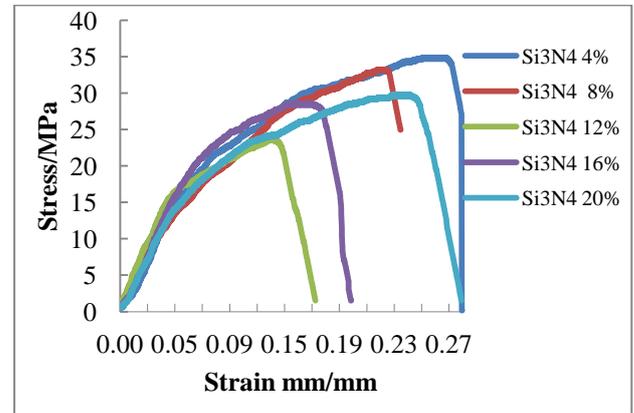


Fig.3: Strain- Stress of curves of Nylon -6/Si₃N₄

Fig.4 shows that when the content of silicon nitride (Si₃N₄) increased the ultimate strength decreases. A change from 4%wt to 8%wt of Si₃N₄ does not influence (i.e., a slight variation occurs) the tensile performance of Nylon-6/ Si₃N₄ polymer composites. The tensile behavior of Nylon-6 /20%wt of Si₃N₄ polymer composite is different from other compositions of Nylon-6/Si₃N₄ polymer composites. It observed from Fig.5 that the strain at an ultimate tensile strength of Si₃N₄/20%wt of Si₃N₄ polymer composite was higher than the strain of other compositions of Nylon-6/ Si₃N₄ polymer composites.

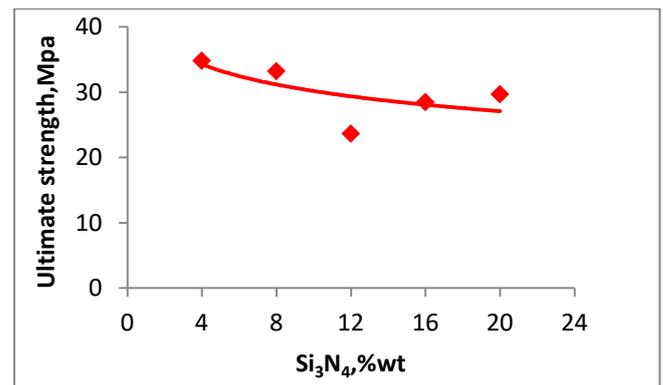


Fig. 4: Concentration of Si₃N₄

A change from 4%wt to 8%wt of Si₃N₄ shows a small increase in the Rockwell hardness of Nylon-6/ Si₃N₄ polymer composites, as shown in Fig. 6. The hardness of Nylon-6/ Si₃N₄ polymer composites was highest at a 16%wt composition of Si₃N₄. It increases due to resistance to the plastic deformation of the Nylon-6 matrix from comparatively hard Si₃N₄ nanoparticles.

The significant improvement in hardness attributed to the better distribution of Si_3N_4 nanoparticles and good adhesion between the Nylon-6 and Si_3N_4 nanoparticles. 12%wt to 16%wt of Si_3N_4 shows an increase in the hardness of Nylon-6/ Si_3N_4 polymer composites, and there is also a significant difference in the hardness for 16% wt to 20%wt of Si_3N_4 , i.e., 16%wt to 20%wt of Si_3N_4 shows a small decrease in the hardness of Nylon-6/ Si_3N_4 polymer composites.

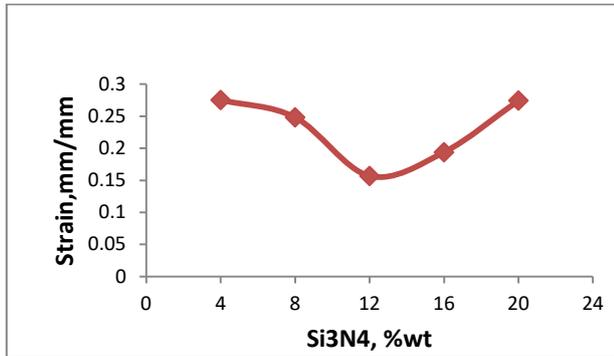


Fig. 5: Effect of volume fraction on a strain

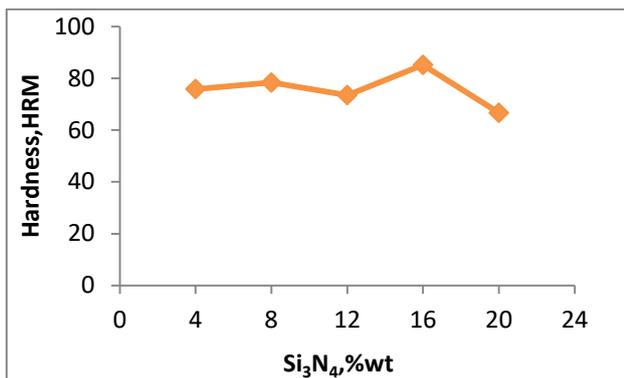


Fig. 6: Function of hardness with a volume fraction of Si_3N_4 nanoparticles

To extremely understand their tensile failure performance, Fractography of Nylon-6/ Si_3N_4 polymer composites were taken, employing SEM images. Fig. 7(a) to 7(f) demonstrates the crack surfaces of pure Nylon-6 and Nylon-6/ Si_3N_4 polymer composites. Fig.7 (a) clears the voids and fibrillations seen in the crack surface of the pure Nylon-6, which are caused by the debonding in Nylon-6. Stress brightening equally shows up on the cross-area of broken Nylon-6 samples. These are the micro-damages propagated along with the nanoparticle/matrix interface at first. These micro-damages spread until the point where the interface degradation reached, and then nanoparticle-matrix bonding was lost. For each one of these cases appeared in Fig. 7(b) and 7(f), the trademark characteristics of strengthening Si_3N_4 nanoparticles seen on the damaged surface. The Nylon-6/ Si_3N_4 composite samples, which have less substance of Si_3N_4 nanoparticles, have broken at higher stresses.

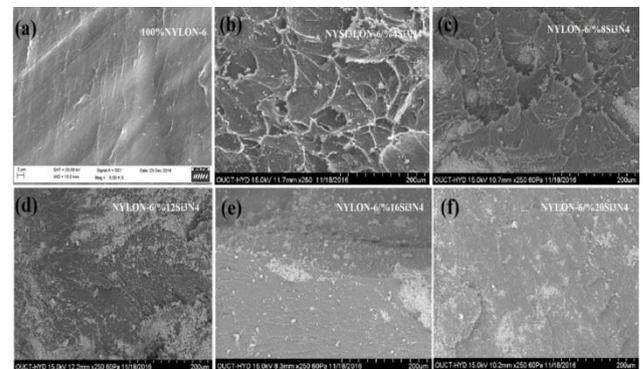


Fig.7: Fractography of Pure Nylon-6 and Nylon-6/ Si_3N_4 polymer composites

Table 3: ANOVA summary of the wear rate

Parameter	Sum1	Sum 2	Sum 3	SS	v	V	P
Si_3N_4	1066	1565	1248	42513	2	21256.5	24.26
Normal load	1330	1314	1235	1725	2	862.5	0.98
Sliding Speed	1223	923	1733	111800	2	55900	63.79
Sliding Distance	1428	1350	1102	19229	2	9614.5	10.97
Error				0.00	1	0	0.00
T	5047	5152	5318	175266	9	87633.5	100.00

Note: SS is the sum of the square, v is the degrees of freedom, V is the variance, and P is the percentage of contribution

The break surfaces of Nylon-6/ 4% wt Si_3N_4 , Nylon-6/ 8% wt Si_3N_4 and Nylon-6/ 12% wt Si_3N_4 composites have rough surfaces and more split stretching. Debonding around the imperfection edge has been seen in many break inception destinations. The debonding reflects light at the flaw periphery making the flaws to project as dull areas or fine spots on the off chance that light reflected the observer. These flaws are the agglomerates of filler material blending in the Nylon-6/ Si_3N_4 polymer composites. The break surfaces of Nylon-6/16% wt Si_3N_4 and Nylon-6/20% wt Si_3N_4 composites

have various cracks and deep routes in the matrix Nylon-6, parallel to one another.

B. Tribological Properties

Table 3 represents the calculations of the percentage contribution for wear rate using the analysis of variance.

The percentage contribution indicates that the volume portion of Si_3N_4 contributes to 24.26%. The normal load gives 0.98% of the variation in the wear rate. The Sliding speed of 63.79% of the variation observed in the wear rate. The sliding distance affords 10.97% of the total variation in the wear rate.

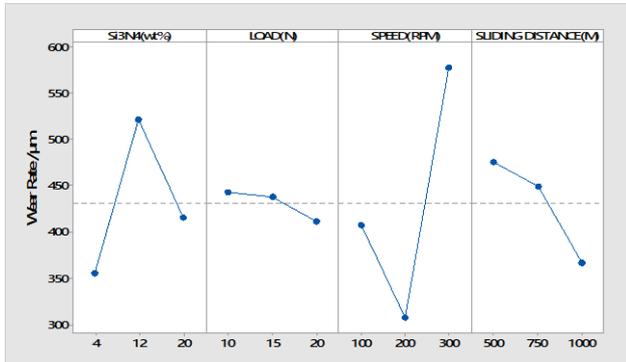


Fig.8: Variation of Wear rate

The wear rate calculated for various percentages of silicon nitride - Nylon-6 composites from the experiments that are conducted using the Taguchi (L9). The input parameter such as load, sliding speed, and sliding distance considered and outcomes all plotted to find the optimum solutions to wear rate in the Fig.8. It observed that, with the increase in the effect of normal load and sliding distance, the wear rate decreases for different % wt composites with the increase in filler load. In the case of 4 %wt composite, the lowest wear rate observed, and the wear rate reduces at 20N load, the sliding speed increases to 300 rpm and the distance to 1000 m, and then the slope reduces. Wear rate can be decreased at a lower addition of silicon nitride and give better mechanical properties such as wear rate and the tensile strength. It gives an advantage to many mechanical applications. At higher loads, there will be a decrease in wear with the addition of silicon nitrate to the Nylon-6. Hence there is good lubrication property with little addition of silicon nitride.

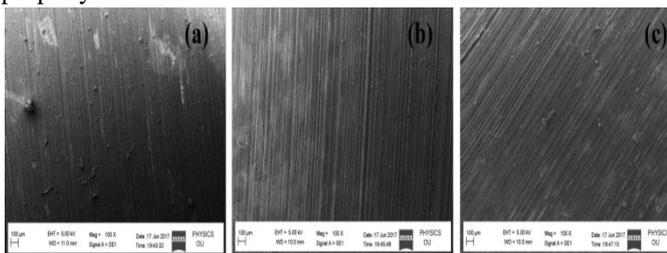


Fig.9. Worn surfaces of specimens for trial conditions of 1, 2, and 3.

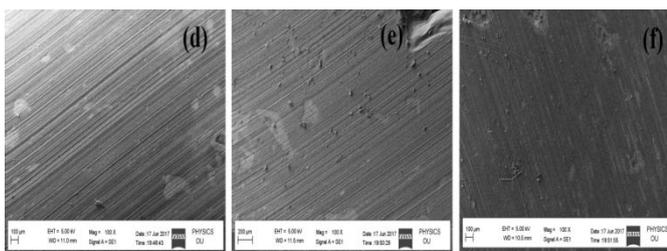


Fig.10. Worn surfaces of specimens for trial conditions of 4, 5, and 6.

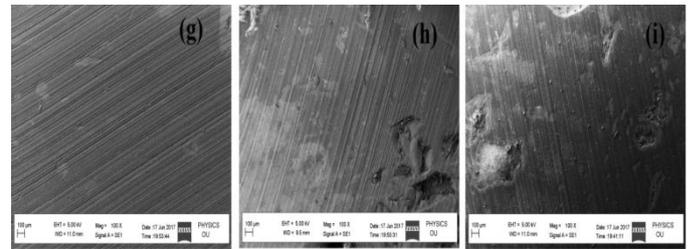


Fig.11. Worn surfaces of specimens for trial conditions of 7, 8, and 9.

The worn surface highlights of the composite sample were inspected utilizing the scanning electron microscope. The SEM highlights of the worn surface samples for preliminary states of 1 to and 9 and abrasive SiC papers 400 grit. Fig. 9 to 11 cutting and void development because chips out of matrix are seen dominantly opposite to the wear direction. There is proof of matrix removal and profound wrinkles in the of Nylon-6/ Si_3N_4 polymer composites as appeared in Fig. 10(e). The Nylon-6/ Si_3N_4 composites framework indicates extreme matrix failure at the underlying phase of abrasion, and hard abrasive particles were in contact with delicate matrix bringing about serious grid harm, and the rate of material expulsion was high. SEM pictures of the worn surface of Nylon-6/ Si_3N_4 composite at 400 grit SiC paper at 200m showed smooth surfaces less matrix destruction and matrix reinforced with Si_3N_4 , and it secures the Si_3N_4 in position as appeared in Fig. 9(b). Whereas SEM pictures at higher abrading separation 300 m indicated rough surface. Fig. 10(e) also reveals the formation of Nylon-6/ Si_3N_4 crust.

IV. CONCLUSIONS

The present work shows that successful production of Nylon-6/ Si_3N_4 composites with a different composition through the injection molding technique.

- The tensile strength was maximum at 4 %wt of Si_3N_4 , and it gradually decreases with the increase of Si_3N_4 .
- Hardness test was carried out for different compositions of Nylon-6/ Si_3N_4 composite, and the higher Rockwell hardness number of Nylon-6/ Si_3N_4 composite observed at 16% wt.
- The wear rate increased with increases in speed. However, the composites have shown a lower rate of wear (up to 4% wt Si_3N_4) as compared to that observed in 12 and 20% wt Si_3N_4 .
- Also, it was observed using scanning electron micrographs that most of the nanoparticles variation occurs homogeneously from the matrix. SEM images help to understand the surface of composites at various input parameters.
- The sliding speed shows a higher percentage contribution, that is 63.79 % for the variation in the wear rate.

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