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

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Abstract: From the Research, it found that there is an impact of Silicon Nitride (Si3N4) on the mechanical properties of Nylon 6 or polyamide 6 based composites. Nylon 6 prepared with Silicon Nitride (Si3N4) by changing the weight proportions. The hardness and wear properties of Nylon-6/Si3N4 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/ Si3N4 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 Si3N4 upgrades the existence state of typical Nylon 6 to a more important point.

Keywords: Nylon-6, Si3N4, 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 applied 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 low densities. The development of Nylon 6/Si3N4 composites have been investigated as per Taguchi’s design. Rockwell, hardness testing device, used to observe the hardness number of different nylon-6/Si3N4 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 Si3N4 upgrades the existence state of typical Nylon 6 to a more important point.

II. MATERIALS AND EXPERIMENTAL METHODOLOGY

The Nylon-6 polymer matrix filled with nanoparticles of silicon nitride (Si3N4) whose size is 100nm. Varying different percentages of Si3N4 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/Si3N4 polymer composites. The blend of Nylon-6 and Si3N4 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 Si3N4 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 samples 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. 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.
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III. RESULTS AND DISCUSSION

A. Tensile Strength

From Fig. 3, it found that there is an effect in the edition of $Si_3N_4$. The tensile strength is higher when the percentage of $Si_3N_4$ is 4%wt. Later with an increase in the percentage of $Si_3N_4$ there is a decrease in the tensile strength. For the 12%wt of $Si_3N_4$, 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 $Si_3N_4$ 4%wt.

**Fig. 3: Strain- Stress of curves of Nylon-6/$Si_3N_4$**

Fig. 4 shows that when the content of silicon nitride ($Si_3N_4$) increased the ultimate strength decreases. A change from 4%wt to 8%wt of $Si_3N_4$ does not influence (i.e., a slight variation occurs) the tensile performance of Nylon-6/$Si_3N_4$ polymer composites. The tensile behavior of Nylon-6/20%wt of $Si_3N_4$ polymer composite is different from other compositions of Nylon-6/$Si_3N_4$ polymer composites. It observed from Fig. 5 that the strain at an ultimate tensile strength of $Si_3N_4$/20%wt of $Si_3N_4$ polymer composite was higher than the strain of other compositions of Nylon-6/$Si_3N_4$ polymer composites.

**Fig. 4: Concentration of $Si_3N_4$**

A change from 4%wt to 8%wt of $Si_3N_4$ shows a small increase in the Rockwell hardness of Nylon-6/$Si_3N_4$ polymer composites, as shown in Fig. 6. The hardness of Nylon-6/$Si_3N_4$ polymer composites was highest at a 16%wt composition of $Si_3N_4$. It increases due to resistance to the plastic deformation of the Nylon-6 matrix from comparatively hard $Si_3N_4$ nanoparticles. The significant improvement in hardness attributed to the better distribution of $Si_3N_4$ nanoparticles and good adhesion between the Nylon-6.

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**Table 1. Levels of Control parameters**

| Factor          | Symbol | Level–1 | Level–2 | Level–3 |
|-----------------|--------|---------|---------|---------|
| $Si_3N_4$ %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    |

**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 |
and Si₃N₄ nanoparticles. 12%wt to 16%wt of Si₃N₄ shows an increase in the hardness of Nylon-6/ Si₃N₄ polymer composites, and there is also a significant difference in the hardness for 16%wt to 20%wt of Si₃N₄, i.e., 16%wt to 20%wt of Si₃N₄ shows a small decrease in the hardness of Nylon-6/Si₃N₄ polymer composites.

To extremely understand their tensile failure performance, Fractography of Nylon-6/ Si₃N₄ polymer composites were taken, employing SEM images. Fig. 7(a) to 7(f) demonstrates the crack surfaces of pure Nylon-6 and Nylon-6/ Si₃N₄ 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 Si₃N₄ nanoparticles seen on the damaged surface. The Nylon-6/ Si₃N₄ composite samples, which have less substance of Si₃N₄ nanoparticles, have broken at higher stresses.

The break surfaces of Nylon-6/ 4%wt Si₃N₄, Nylon-6/ 8%wt Si₃N₄ and Nylon-6/ 12%wt Si₃N₄ 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/ Si₃N₄ polymer composites. The break surfaces of Nylon-6/16%wt Si₃N₄ and Nylon-6/20%wt Si₃N₄ 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 Si₃N₄ 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

| Parameter    | Sum1 | Sum 2 | Sum 3 | SS      | v   | V       | P      |
|--------------|------|-------|-------|---------|-----|---------|--------|
| Si₃N₄        | 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 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 form 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 wear surface highlights of the composite sample were inspected utilizing the scanning electron microscope. The SEM highlights of the worm surface samples for preliminary states of 1 and 9 and abrasive SiC papers 400 grit. Fig. 9 to 11 cutting and void development because chips out of matrix are seen dominatingly opposite to the wear direction. There is proof of matrix removal and profound wrinkles in the of Nylon-6/ Si$_3$N$_4$ polymer composites as appeared in Fig. 10(e). The Nylon-6/ Si$_3$N$_4$ 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$_3$N$_4$ composite at 400 grit SiC paper at 200m showed smooth surfaces less matrix destruction and matrix reinforced with Si$_3$N$_4$ and it secures the Si$_3$N$_4$ 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$_3$N$_4$ crust.

IV. CONCLUSIONS

The present work shows that successful production of Nylon-6/ Si$_3$N$_4$ composites with a different composition through the injection molding technique.

- The tensile strength was maximum at 4 %wt of Si$_3$N$_4$, and it gradually decreases with the increase of Si$_3$N$_4$.
- Hardness test was carried out for different compositions of Nylon-6/ Si$_3$N$_4$ composite, and the higher Rockwell hardness number of Nylon-6/ Si$_3$N$_4$ 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$_3$N$_4$) as compared to that observed in 12 and 20% wt Si$_3$N$_4$.
- 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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