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EXPERIMENTAL INVESTIGATION ON TRIBOLOGICAL CHARACTERISTICS OF SILICON NITRIDE REINFORCED ALUMINIUM METAL MATRIX COMPOSITES

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Abstract

Aluminium alloy (LM25) reinforced with silicon nitride was fabricated by liquid metallurgy route. The fabricated composite was investigated for dry sliding wear behaviour by conducting experiments using pin-on-disc tribometer. Set of experiments were planned using Taguchi's technique and data analysis was carried out using L_{27} orthogonal array. Analysis of Variance (ANOVA) technique was used to determine the significance of parameter with respect to wear rate. Signal-to-Noise ratio was employed to detect the most and least influential parameter as well as their level of influence. 'Smaller the wear' characteristic was chosen for the analysis of dry sliding wear. Results implied that, the load has the primary effect on the wear succeeded by the effect of sliding velocity and sliding distance. Scanning Electronic Microscopic studies were carried out on worn surfaces to understand the wear mechanism.Tribological results indicated that LM25 aluminium alloy could be better utilized as a material for piston, rotor and bearings for long life in low speed applications.

Keywords**:** Silicon nitride, Stir casting, Taguchi technique, Wear rate, Analysis of variance.

1. Introduction

Composites were the combination of materials that have distinct physical and chemical properties. It combines both the properties of matrix and reinforcement and this characteristic feature of composites made it a preferable material among manufacturers and researchers. Metal matrix composites (MMCs) were widely accepted in automotive industry for the properties of wear resistance, expansion coefficient, fatigue strength and damping property [1]. Aluminium metal matrix

Nomenclatures

D	Sliding Distance, m
F	Frictional force, N
L	Load, N
P	Pressure, N/m^2
V	Sliding velocity, m/s

Greek symbols

μ	Coefficient of friction

Abbreviations

AMMC	Aluminium Metal Matrix Composite
ANOVA	Analysis of Variance
LVDT	Linear Variable Differential Transformer
MMC	Metal Matrix Composite

composite (AMMCs) led to the development of advanced materials with the improved properties of lightweight, stiffness and temperature resistant [2]. Light MMCs with ceramic reinforcement have gained attention for its improved mechanical properties [3] as well as it was cost effective and has benefit towards the environment [4]. Similarly, for good castability in terms of shape and dimension, it has found application in cylinder blocks and wheel [5]. The properties of thermal expansion, damping capacity and fatigue life, have made it viable for manufacturing of connecting rod and drive shafts [6]. AMMCs with silicon based reinforcements were more resistant to wear, thereby has a significant role to play in brake pads of automobiles [7].

The possible ways to develop AMMCs and the difficulties involved in the process were investigated for automotive applications [8]. Researchers contributed several techniques for the manufacture of composites such as stir casting, plasma spraying, powder metallurgy and squeeze casting [9]. Composites fabricated using powder metallurgy process was found to exhibit weak interface bond between matrix and reinforcement [10] and in order to obtain composites with denser structure, powder metallurgy technique followed by an extrusion process were employed [11]. Mechanical alloying was an effective process to manufacture composites involving two or more reinforcement particles [12]. Stir casting technique was the cost effective method among all other techniques for fabrication of MMCs [13, 14]. Stir casting technique was preferred over other techniques for its ability to achieve uniform distribution of reinforcement particles [15, 16].Presence of hard reinforcements in the form of particles or whiskers showed a great impact on the tribological behaviour of composites [17]. Hard ceramic particles like silicon carbide, alumina reinforced composites were analyzed for dry sliding behaviour, and an increase in wear resistance was observed due to adequate bond formed by silicon carbide particles with the aluminium matrix [18]. Inclusion of alumina as reinforcement has brought improvement in wear properties [19]. Addition of ceramic reinforcements like Titanium nitride, titanium carbide and boron nitride were found to form tribolayers which promote the increase in fracture toughness and improvement in wear [20]. Improvement in wear resistance was reported to be greatly influenced by the kind of reinforcement used and its volume of fraction [21].

Increase in volume fraction of reinforcement brought an effective interfacial bond resulted in improvement related to transition wear characteristics [22, 23]. Increase in percentage of silicon carbide reinforcement showed improvement in hardness as well tribological properties [24].

From the above literature review, lot of experimental work were carried out with ceramic particles such as silicon carbide, alumina and tungsten carbide as reinforcements. It was inferred that not much experimental work was done with silicon nitride particle as reinforcement. Hence an effort was taken to explore about the silicon nitride based composites and its wear behaviour.

2.State of the art

Design of experiments was an effective statistical technique to evaluate, when the cause and the effect was due to multiple parameters. The experiments were carried out in a sequential manner followed by a series of steps. With combined variation in factors and their level, the process performance was evaluated to obtain the results. The results determined exhibit a better performance obtained with the correct combination.

Taguchi method was one of the best tool for design of system and a systematic approach to determine the optimal settings of parameter to produce results with minimum variation. Taguchi method helped to carry out limited number of experiments to achieve accurate results. The method holds well when the parameters were qualitative and discrete in nature. It involved collection of data from experiments to know the effect of parameters. Orthogonal array was usedto design the plan of experiments. The series of experiment were formulated using Taguchi method and analysis of contribution of the individual parameters were found using ANOVA technique. Ranking of parameters was done by the influence, evaluated using response table for Signal to Noise ratio.

2.1. Material selection

Due to its excellent castability characteristics, LM25 alloy was chosen as the matrix material for the investigation. LM25aluminium alloy of density 2.68 g/cm^3, was chosen as the matrix material for its property of castability, and because of this it was used as suitable material for cylinder heads [4]. LM25 alloy possess good mechanical properties, machinability and corrosion resistant, thereby has become a preferable alloy in the manufacture of brake disc. For its ability to resist high temperature, and nature of chemical inertness, silicon nitride particles (10% of wt.) of size 20μm and density of 3.44 g/cm^3 were chosen as reinforcement. Addition of silicon nitride particles improved the hardness of the composite material, thereby has an application in the form of brake pad [15]. Table 1 denotes the composition of LM25 alloy.

Table 1. Composition of LM25 alloy (in % of wt.).

Cu	**Si**	**Mg**	**Fe**	**Mn**	**Ni**	**Zn**	**Pb**	**Sn**	**Ti**	**Al**
0.20	7.50	0.06	0.50	0.30	0.10	0.10	0.10	0.05	0.02	Balance

2.2.Preparation of composite

Owing to its cost effectiveness and simplicity, stir casting method was preferred to manufacture the composite. Stir casting involved two steps namely, melting of matrix metal and mixing of reinforcement with molten metal. Initially, the matrix metal was placed in a crucible and heated to a temperature above 750℃ in a furnace, by which the metal attain molten state. In order to improve the wettingperformance, the reinforcement particles were preheated to a temperature of 300℃. Then, the preheated reinforcements were added to the molten metal and the mixture was stirred continuously at a speed of 300 rpm for 10 minutes. This continuous stir of mixture brought about the homogeneous dispersion of reinforcement particles. Finally, it was poured into the preheated mould of 300℃ and allowed to solidify.

3. Experimental Setup

The cast part was cut into specimen of length of 35 mm and has a square cross section of 10 mm. A pin-on-disc tribometer (Fig. 1) was employed for the investigation of wear rate. The machine consists of a fixed arm with pulley at one end and a stationary pin at other end. The pulley was meant to suspend weights which imposed a force at the end of stationary pin to which the specimen was fitted. The force due to load helped the specimen to maintain contact with the rotating disc which has a range of speed from 50 rpm to 2000 rpm. By sliding action against the disc the specimen suffered a change in dimension. This change in dimension caused the fixed arm to displace and this displacement was recorded by a Linear Variable Differential Transformer (LVDT) attached to the fixed arm. The output of LVDT was considered for measure of wear occurred in the surface. Weight loss of the specimen was estimated before and after the experiment using a weighing machine, which has an accuracy of 0.001g. All the experiments were carried out with a constant track diameter of 100 mm. Table 2 denotes the various process parameters along with their levels.

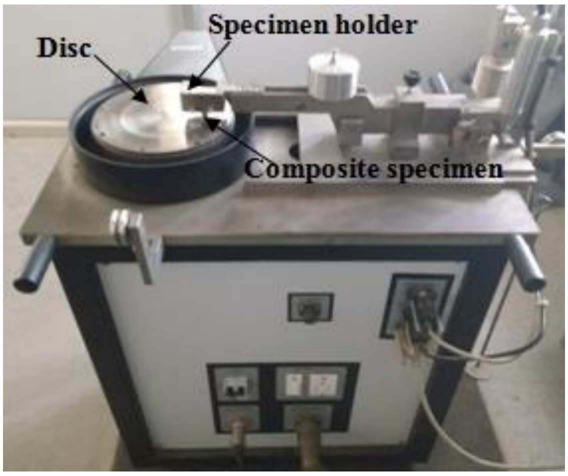

Fig. 1. Pin-on-disc tribometer.

Table 2. Process parameters and their levels.

Level	Sliding velocity, V (m/s)	Load, L (N)	Sliding distance, D(m)
1	1.5	10	400
2	3	20	800
3	4.5	30	1200

3.1. Experimental plan

The parameters considered for wear test was sliding distance, sliding velocity and load which were varied at three levels. In order to witness the performance at high temperature by inducing maximum possible friction, load of maximum 30N and sliding velocity of maximum 4.5 m/s were chosen. To determine the durability of the material surface, maximum sliding distance of 1200m was decided. The experimental plan was formulated using a standard orthogonal array. Based on the condition that degree of freedom of an orthogonal array should be greater than or equal to the sum of the wear parameters, a standard L_{27} orthogonal array was chosen. The orthogonal array has columns which were assigned for parameters. The first column denote the sliding velocity (V), second column denote the load (L) and fifth column denote the sliding distance (D) while remaining columns were dedicated for interaction. With parameters assigned, the experiment was carried out in a pin-on-disc tribometer and the graphical plots along with the numerical values were obtained through MINITAB software.

The characteristics which influence the wear rate were found using Signal to Noise (S/N) ratio. S/N ratio was grouped into three namely, ‘nominal is the best’, ‘larger the better’ and ‘smaller the better’. The response table for Signal to Noise ratio for ‘smaller the wear’ was calculated as the main objective of the investigation was to minimize the wear rate. The response table denoted the rank of influence caused by parameter on wear rate. The relative effects of the parameters were found using S/N ratio of combined parameters.

The experiments were carried out on pin-on-disc tribometer and wear rate was calculated. Table 3 shows the experimental parameters along with their calculated values of wear rate and corresponding S/N ratio.

Table 3. Orthogonal table on wear.

Exp No.	Sliding velocity, V (m/s)	Load, L (N)	Sliding distance, D (m)	Wear rate (mm^3/m)	S/N ratio
1	1.5	10	400	0.00280	51.0568
2	1.5	10	800	0.00283	50.9643
3	1.5	10	1200	0.00190	54.4249
4	1.5	20	400	0.00283	50.9643
5	1.5	20	800	0.00237	52.5050
6	1.5	20	1200	0.00310	50.1728
7	1.5	30	400	0.00283	50.9643
8	1.5	30	800	0.00374	48.5426
9	1.5	30	1200	0.00283	50.9643
10	3	10	400	0.00191	54.3793
11	3	10	800	0.00145	56.7726
12	3	10	1200	0.00161	55.8635

13	3	20	400	0.00191	54.3793
14	3	20	800	0.00230	52.7654
15	3	20	1200	0.00280	51.0568
16	3	30	400	0.00370	48.6360
17	3	30	800	0.00230	52.7654
18	3	30	1200	0.00283	50.9643
19	4.5	10	400	0.00283	50.9643
20	4.5	10	800	0.00237	52.5050
21	4.5	10	1200	0.00161	55.8635
22	4.5	20	400	0.00191	54.3793
23	4.5	20	800	0.00237	52.5050
24	4.5	20	1200	0.00252	51.9720
25	4.5	30	400	0.00283	50.9643
26	4.5	30	800	0.00237	52.5050
27	4.5	30	1200	0.00222	53.0729

4. Results and discussions

A series of experiments were conducted to estimate the effect of sliding distance, sliding velocity and applied load on wear rate at their different level of influence. Graphs were plotted using MINITAB 14 software to denote the trend followed by the corresponding parameter against their mean values of wear rate. The following comparisons were plotted.

1. Wear rate vs sliding speed.
2. Wear rate vs sliding distance.
3. Wear rate vs load.

Figure 2 represent the main effect plot for load, sliding distance and sliding velocity against the mean values of wear rate. Figure 3 represent the main effect plot for load, sliding distance and sliding velocity against the mean values of S/N ratio. From Fig. 3, the optimum parameter for better tribological behaviour was inferred as load of 10N, sliding velocity of 3m/s and sliding distance of 1200 m.

4.1. Effect of load on wear rate

Figure 2 show the main effect plot for wear rate against the load (10N, 20N, 30N). It was noted from the plot that, the wear rate became high for the increase in load from 10N to 30N and maintained a steady progress. The material loss was due to the contact pressure caused by the normal load and frictional force generated at the interface. For a load of 10N, the normal force exerts a contact pressure on the material surface which has led to rubbing action against the sliding surface. The contact pressure impose an area of contact over sliding surface as well as the constant rubbing action brought a friction proportional to contact pressure, according to the relation $F = \mu P$. Thus the resultant frictional force made the material to suffer wear loss by mechanism of friction wear at low load condition. At an intermediate load of 20N, the material loss happened due to adhesive action. Since the matrix material was soft and ductile one, it has experienced a material loss when came in contact against a hard material (disc) due to increase in contact pressure at the interface. By this the matrix surface lost its material by means of adhesion. At a peak load of 30N, the mechanism of ploughing action was responsible for increase in wear rate. For the applied load of 10N and 20N, the matrix layer with few reinforcements on the surface was exposed to wear. At the end of low and intermediate load condition, the surface was found to be semi worn with reinforcement protruded out. At 30N, the protruded reinforcements acted as load transfer elements and transferred the load applied on them to the matrix surface. This action of load transfer brought the ploughing action of matrix surface which resulted in increase in wear rate.

4.2. Effect of sliding distance on wear rate

From Fig. 2, the plot between wear rate and sliding distance depicted a decreasing trend. It was observed from the plot that the slope of wear rate decreased with increase in sliding distance which can be expressed as initial running-in process and final steady state process. The cause for decrease in wear rate was mainly due to the presence of sharp asperities on the wear surface. These asperities protrude and prevent the material surface to establish contact against the sliding surface, which resulted in less wear rate. However, at short sliding distance of 400m, the sharp asperities on the surface become fractured due to sliding action against the contact surface which was the reason for high wear rate. With the progress in distance, the sharp asperities got blunt and smoothened out to enable uniform contact with the surface and that turned out to be the reason for decrease in wear rate for sliding distance of 800m and 1200m. Till 800m, the wear rate suffered a significant decrease, which was attributed as running in process. From 800m to 1200m, a mild reduction in wear rate which might probably a sign of approach of steady state in wear rate.

4.3. Effect of sliding velocity on wear rate

From Fig. 2, the wear rate suffered a decrease and increase in trend with the sliding velocity from 1.5 m/s to 4.5 m/s. The increased wear rate at 1.5 m/s was due to severe delamination of asperities on the surface happened by sliding action and the formation of Mechanically Mixed Layer (MML) could be a reason for decrease in wear rate. The repetitive sliding action might have resulted in transfer of material, led to formation of layer of wear debris. Simultaneously, the temperature rise at the interface initiated the formation of oxide layer. The oxide layer formed act as protection between the contact surfaces, caused reduction in wear rate at an intermediate sliding speed of 3m/s. However, at high sliding speed like 4.5m/s, a transition in wear rate was noticed in the graph which revealed the increase in wear rate which could be explained by the phenomenon of weak interface bond. For a defined load, at a sliding velocity of 4.5m/s, the debris layer formed at the end of 3m/s began to deteriorate due to its low shear strength and because of which the temperature at the interface became high causing localized softening of the material. Due to softening of material, the interfacial bonding strength became weak which resulted in transfer of material to the steel disc by adhesive action. This localized softening followed by adhesive action has led to increase in wear rate with the sliding velocity of 4.5m/s and a similar observation was reported [24].

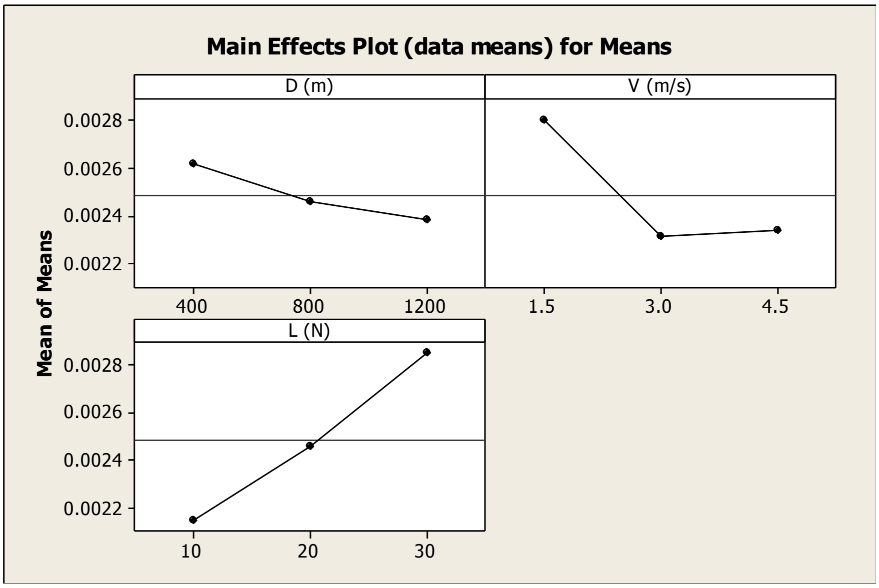

Fig. 2. Main effect plot for wear rate.

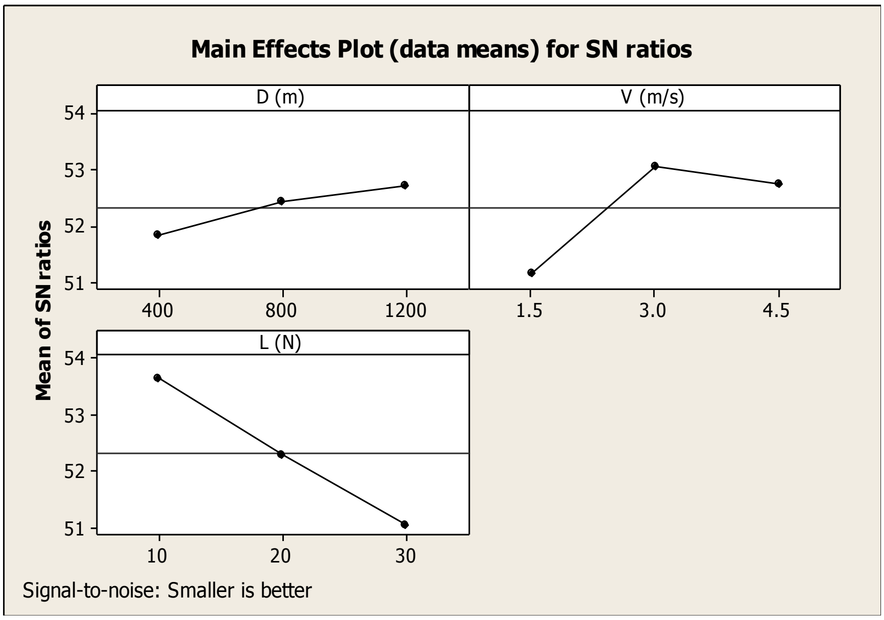

Fig. 3. Main effect plot for S/N ratio.

4.4. Analysis of variance and S/N ratio

Table 4 indicates the ANOVA results for wear rate. The analysis was performed for level of confidence at 95%.

From ANOVA results, the most influential parameter affecting wear was found to be load (25.58%), followed by sliding velocity (16.28%). The least influential parameter was sliding distance (3.49%). The combination of load and sliding velocity (19.77%) also had the severe impact on wear rate. The parameter with ‘P’ value less than 0.05 indicated the major effect on the response.

Table 5 represent the response table for Signal to Noise ratio and the ranking of parameters which influence the wear rate. Load ranked first to denote the dominant effect, followed by sliding velocity and sliding distance.

Table 4. Anova table for wear results.

Source	DF	Seq SS	Adj SS	Adj MS	F-value	P-value	Pct (%)
L	2	0.0000022	0.0000022	0.0000011	5.98	0.026	25.58
V	2	0.0000014	0.0000014	0.0000007	3.68	0.074	16.28
D	2	0.0000003	0.0000003	0.0000001	0.70	0.524	3.49
D*V	4	0.0000006	0.0000006	0.0000002	0.80	0.556	6.98
D* L	4	0.0000017	0.0000017	0.0000004	2.25	0.153	19.77
V*L	4	0.0000009	0.0000009	0.0000002	1.22	0.374	10.47
Error	20	0.0000015	0.0000015	0.0000002			17.44
Total	26	0.0000086					100

Note: DF- degrees of freedom; Seq SS-sequential sum of squares; Adj MS-adjacent mean squares; Pct-percentage.

Table 5. Response table forsignal to noise ratio– smaller is better (wear rate).

Level	D (m)	V (m/s)	L (N)
1	51.85	51.17	53.64
2	52.43	53.06	52.30
3	52.71	52.75	51.04
Delta	0.85	1.89	2.60
Rank	3	2	1

4.5. Scanning electron microscopy analysis results

In order to understand the wear mechanism, Scanning Electron Microscope images of worn surface were taken. Figures 4(a), (b) and (c) represent the Scanning Electron Microscope (SEM) images of the worn surfaces.

The worn surface images were taken for various sliding velocities such as 1.5m/s, 3m/s and 4.5m/s with load (30 N) and sliding distance (1200 m) as constant. Figure 4(a) resembles the worn surface with grooves formed as a result of adhesive wear and a view of worn out asperities on the surface. The adhesive wear marks was due to the load applied and the broken asperities which was the reason for maximum wear rate at 1.5m/s. Figure 4(b) shows the worn surfaces for a moderate sliding velocity of 3m/s, hinted about the oxide particles because of which the wear rate experienced a reduction and also the sliding marks on debris layer were visible. Figure 4(c) feature the worn out condition of wear debris layer and disappearance of oxide particles which were the sign of increase in wear rate.

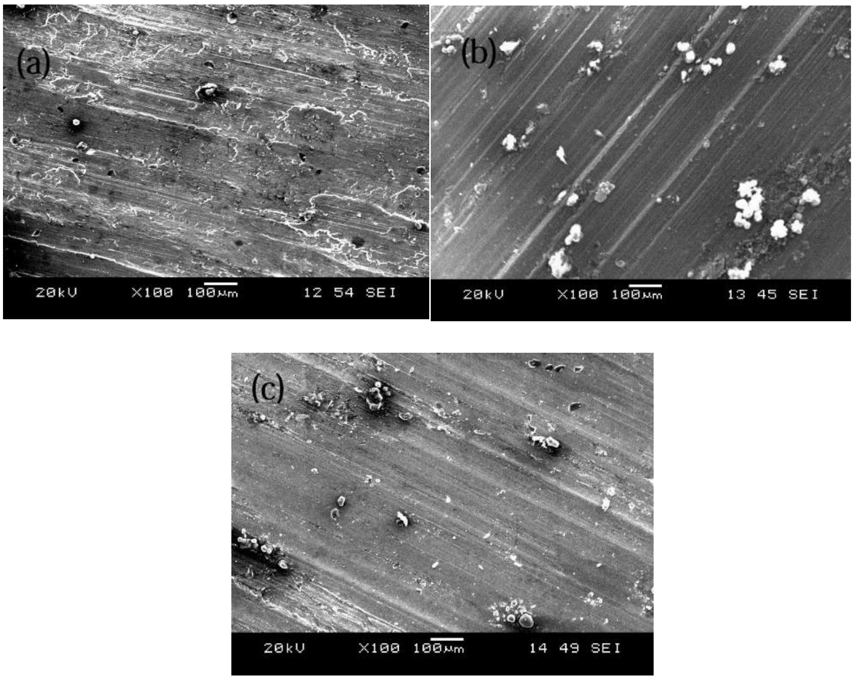

Fig. 4. SEM images of worn surfaces at different sliding velocities: a) V=1.5 m/s b) V=3 m/s c) V=4.5m/s.

5. Conclusions

LM 25 alloy reinforced with 10% wt. of silicon nitride particles was successfully fabricated by liquid metallurgy process and tested for wear behaviour. The tribological results indicated the following conclusions:

- Load was the dominant factor with a contribution of 25.58% and stayed in proportion with wear rate. Sliding velocity stood as a secondary factor with a contribution of 16.28% and sliding distance with its share of 3.49% had the least effect on wear rate.
- The optimum parameters to obtain the best tribological characteristics were low load (10 N), moderate sliding velocity (3 m/s) and higher sliding distance (1200 m).
- For the load applied, the material experienced wear by the mechanism of adhesion of matrix material and ploughing. For the parameter sliding velocity, wear took place by the mechanism of delamination of surface layer as well as by adhesive action and for the parameter sliding distance, the wear was witnessed to occur by the mechanism of plastic deformation of asperities.
- From the above investigation, aluminium reinforced silicon nitride composites were found to possess good tribological properties at low load, low speed and long distance conditions.
- Conclusively, the outcome of this work could be taken as a reference in selection of application to employ silicon nitride reinforced composites.

- It is suggested that deployment of this material in the form of piston, bearings and rotors in low load and low speed applications could favour a lasting life to the product.

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