Evaluation of mechanical and wear properties of AA6063/(Si3N4)6%–12%/ (CuN2O6)2%–4% composite via PM route and optimization through robust design technique

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Abstract
The study is to investigate the physical, mechanical, and tribological properties of Al6063 alloy reinforced by Silicon Nitride (Si3N4) & compound Copper Nitrate (CuN2O6) processed via Powder Metallurgy (PM) Techniques. Incorporation of reinforcement in matrix material ranged from 6 to 12% Si3N4 in a 6-step interval, and 2 to 6%CuN2O6 in a two-step interval. The characterizations were made on the PM produced specimens using OM, EDS, XRD and hardness. The reinforcement particles were distributed uniformly is attributed by homogeneous mixer of matrix and reinforcements. The tests were carried out in accordance with ASTM Standards on the Al6063 alloy and its composites. The test findings show that as the reinforcing percentage of ceramic and inorganic compound is increased, properties such as hardness and density rise monolithically and considerably. The dispersion of Si3N4 and CuN2O6 reinforcement in the AA6061 matrix was ensured by x-ray diffraction patterns. In comparison to the base alloy, the hardness of AA6063/12%Si3N4/6% CuN2O6 improved by 88% due to the mismatch of thermal expansion between the Al matrix and reinforcement causes huge internal stress, causing the aluminium matrix to deform plastically to lodge the smaller volume expansion of Si3N4 and CuN2O6 particles. The dry sliding wear test was carried out on a tribometer with a pin-on-disc arrangement, and the findings show that the composite has a higher wear resistance. The Taguchi design of experiments was used to investigate the solution containing parameters employing an orthogonal array, the signal-to-noise ratio, and analysis of variance. The weight percentage of Si3N4/CuN2O6 compound and the relationship between wt% of reinforcement and applied load had the highest impact on composite wear resistance, accounted for 31.66%. Before and after the wear morphology during the wear test, images from a scanning electron microscope and energy dispersive microscopy were used to examine the manufactured composites.

1. Introduction
In the recent decades, Hybrid Aluminum matrix composite (HAMC) has been used for many applications such as aerospace, automobile, marine and light weight components due to their attractive properties such as good specific strength, specific modulus. Because of its light weight and great strength, aluminium is widely employed
in the automotive and aerospace sectors. To create light weight Al and Mg components, traditional production techniques such as casting and powder metallurgy (PM) are frequently used. Due to environmental conditions, speed, and mechanical loading, these light weight Industrial components are prone to wear. Material loss happens in components as a result of repeated rubbing operations, potentially increasing maintenance costs and repair time. The best way to enhancement of mechanical and tribological behavior of hybrid aluminum composite has been attained through ceramic reinforcement such as SiC [1, 2], B₄C [3, 4], Al₂O₃ [5, 6], TiC [7, 8], and AlN [9, 10]. The MMC’s wear performance is influenced by microstructural features such as volume fraction, particle size, shape, and reinforcing distribution and quantity. Many researchers have sought to produce MMC pieces with various reinforcements in order to increase their strength. They’ve also looked into the mechanical and microstructural characteristics of MMCs according to the reinforcement. Aravindan et al. [11], investigated Al6063-SiC 10 wt% MMCs and found that the microstructure with good particle distribution and mechanical properties such as hardness, Ultimate Tensile Strength (UTS), Percentage (% Elongation and Yield Strength) properties increased with increasing reinforced weight (wt) fraction of SiC. Ramesh et al. [12] synthesized Al6063-TiB₂ in-situ composites and performed dry sliding wear tests at various loads and sliding velocities, finding that the composites have a lower coefficient of friction (COF) and wear rates than Al6063 alloy. Nonetheless, the rates of wear were seen to increase as the weight applied and the sliding velocity increased.

The liquid metallurgy processed AA6063–10% Si₃N₄ reinforced composite has given an 86 percent increase in hardness when compared to the base alloy, according to Veereshkumar et al. [13]. Using an in-situ powder metallurgy (IPM) technique, Mahdavi et al. [5] examined the effect of SiC content on the processing, compaction behaviour, and characteristics of Al6061/SiC/Gr hybrid composites. Basavarajappa et al. [14], concluded that composite wear resistance improved when ceramic and metallic reinforcement increased and the transition load from mild to severe wear increased. Hossein Abzideh et al. [15], discussed the aluminum composite reinforced with ZrSiO₄. With increasing zircon content, yield stress and compressive strength change. The specimen with 5% zircon concentration in 650 C has the highest compressive strength, which is 248 MPa as a result of preventing plastic deformation and causing greater strain hardening. Erdemir et al. [16], investigated the mechanical properties of functionally graded Al2024/SiC composites and found that increasing the SiC content to 35 percent and 40 percent increased microhardness, but increasing the SiC content to more than 50 percent and 60 percent reduced microhardness due to high porosity. Ravichandran et al. [17], investigated the mechanical characteristics of the hot extruded Al-TiO₂ powder metallurgical composites. The results reveal that adding 5% TiO₂ increases tensile strength while adding 7.5 percent TiO₂ increases hardness.

Mehdi Rahimian et al. [18], investigated the hardness of Al-Al₂O₃ composites and found that adding 15% Al₂O₃ reinforcement results in a hardness increase. As a result of the above, the researchers concentrated on ceramic reinforcement as a means of improving the properties of aluminum composites. Fathy et al. [19] investigated the microstructure, mechanical, and magnetic properties of Al matrix composites reinforced with 5, 10, and 15% iron powder using powder metallurgy. Due to dispersion strengthening of the Al matrix by the Fe powders and the development of intermetallic compounds as the mass fraction of the Fe powders. 15 percent iron powder had a compressive strength of 550 MPa and a plastic strain of 65 percent. The Al-15Fe composite showed the best mechanical characteristics of all the composites tested.

Pal et al. [20], examined Ni reinforced Al matrix composites with different weight percentages of 10, 20, 30, and 40%. Composition of Al matrix composite reinforced with 20% Ni shows the best combination of hardness, tensile strength, and thermal conductivity. Raj et al. [21], used the pin-on-disk type system to examine the tribostest characteristics of an aluminum LM13/12 wt percent Si₃N₄/3 wt percent Gr composite produced using liquid metallurgical processing. Lotfy et al. [22], used a melt stirring procedure to create Al-5 percent Cu alloy reinforced BN/Si₃N₄ and examined the impact of the BN/Si₃N₄ reinforcement content on the microstructure, mechanical, and thermal properties of the composite. Sharma et al. [23], investigated the effects of Si₃N₄ on AA6082/Si₃N₄ composite on the tribostest and mechanical properties of AMCs. The goal of this research is to make an AA6351/Si₃N₄ composite using a melt stirring method and to investigate the impact of Si₃N₄ reinforcing content and weight percentage on the microstructure, tribological characteristics, and mechanical properties of AMCs. SEM analysis was used to characterize the manufactured composite. The taguchi technique, on the other hand, has been shown by researchers to be an effective tool for composite wear study [24–29]. The main goal of this study was to fabricate the hybrid aluminium matrix (HAM) composites (ceramic and inorganic reinforcement) using powder metallurgy (PM) and analyze their mechanical and tribological properties. Furthermore, using Taguchi design of experiments, the influence of applied load, sliding speed, and sliding distance on the wear behaviour of hybrid composites was investigated. The proportion of influence of various factors and their interactions was determined using analysis of variance. To further understand the wear mechanisms, the SEM morphology of worn surfaces, wear debris, and the EDX of worn surfaces were examined. However, the use of ceramic and inorganic compound reinforcements is novel in this work. The process parameter of both varying reinforcement processed by powder metallurgy technique was analyzed using the robust design technique.
2. Experimental work

2.1. Materials and methods
The powder metallurgy (PM) method was employed to fabricate the hybrid aluminium composites (HAC). The matrix used in this study was aluminium 6063, and the composition is described in the table 1. Because of its mechanical properties, heat treatability, and weldability, the matrix was chosen for a wide range of applications including architectural, automotive, and marine parts. In this analysis, inorganic metallic reinforcement of CuN_2O_6 and ceramic Si_3N_4 were used as reinforcements as shown in table 2. The matrix powder was purchased by M/s Metal Powder Company Ltd, Tamilnadu, India. Four combinations of nitrate-based inorganic element and ceramic-based element aluminium composites were prepared for the mechanical property analysis. For tribological studies three combination of mixture were used.

The Al elemental powders were dried at 100°C for one hour using muffle furnace. To achieve uniform mixing and reduce powder size, the powder was mixed in a planetary tumbler mixer with stainless steel balls with a diameter of 10 mm and a ball to powder weight ratio of 15:1, with a mixing time of up to 4 h. To avoid oxidation, mixing was done at 250 rpm with toluene. The AA6063 powder was then combined with varied weight fractions of silicon nitride (6 and 12 wt%) and copper nitrate (2 and 4 wt%) in a tumbler mixer. To make green compacts, the combined powder was pressed at 750 MPa in a compression-testing machine (CTM). The experimental setup for the composite preparation is shown in figure 1. The green compacts were sintered at 520°C for 1 h at a steady temperature. The sintered composite was solution treated in a furnace at 530°C for 2 h and then water quenched before being naturally aged for 72 h.

Table 1. Chemical composition of AA6063.

| Element | Si   | Mg   | Fe   | Cu  | Mn  | Cr  | Zn  | Ti  | Al  |
|---------|------|------|------|-----|-----|-----|-----|-----|-----|
| Content%| 0.48 | 0.47 | 0.34 | 0.1 | 0.1 | 0.08| 0.1 | 0.05| Balance |

Table 2. The composites used for this study.

| S.No. | Specimen compositions                |
|-------|-------------------------------------|
| 1.    | AA6063/6%Si_3N_4                     |
| 2.    | AA6063/12%Si_3N_4                   |
| 3.    | AA6063/12%Si_3N_4/2%CuN_2O_6        |
| 4.    | AA6063/12%Si_3N_4/4%CuN_2O_6        |
2.2. Characterization
The density of the AA6063 matrix composite specimens was measured using the Archimedean principle. The specimens were immersed in distilled water for density calculations. The weight of the specimen was determined before and after immersion. The microstructure of the AA 6063/\text{Si}_3\text{N}_4/\text{CuN}_2\text{O}_6 composite generated by powder metallurgy was investigated. On the PM manufactured composites, sectioning was done, and then a standard metallographic study was used to achieve the mirror finish. The samples were ground with SiC sheets from 400 to 1500 grits, then polished with an alumina velvet cloth to achieve a mirror-like finish. To disclose the microstructure, the samples were etched with Keller’s (HCl + HNO$_3$ + HF) etchant. Optical microscope was used to analyze the structure and distribution of the specimens reinforcement (OM). The phases contained on the PM composite specimens were identified using an x-ray diffractometer (Bruker D8 Discover). In order to evaluate the influence of reinforcement addition on the matrix, the hardness of the composites was determined using a Rockwell hardness tester in accordance with ASTM E10. The indentation was made with a spherical hardened steel ball indenter with a diameter of 10 mm. A 500 gf load was applied, and a 15 s dwell duration was maintained. The hardness experiment was carried out in ten separate locations before the average was calculated.

2.3. Wear test
The ASTM G99 standard was used to measure the tribological properties of composites, such as wear and coefficient of friction, using a pin-on-disc test under unlubricated dry sliding conditions. The weight percentage of reinforcement, applied load, sliding distance, and sliding speed are the process parameters being investigated. The photograph of Pin-on-Disc wear test setup is shown in figure 2. The sliding disc is made of EN32 steel with an HRC 65 hardness and a diameter of 165 mm. Because, the EN32 steel used for the counterpart of the pin due to the higher hardness as compared to the pin material. In all of the experiments, the sliding pin is used on a track with a diameter of 110 mm. The EN32 steel has a 1 m initial surface roughness. The pin is composed of aluminium hybrid metal matrix composites and has a diameter of 10 mm and a height of 25 mm. Prior to wear testing, it is critical to ensure that the test sample, namely the pin end surfaces, are flat and polished using metallographic procedures.

The test specimen was prepared by manually grinding the composite aluminium surface with 240, 320, 400, and 600 grit silicon carbide papers, then polishing with 5, 1, and 0.5 m alumina on a low speed-polishing machine. Surface relief between hard and soft aluminium matrix is achieved using this approach. The specimen and counter face disc was cleaned with acetone after each test. This was done before and after the test to remove any traces of composite. Weighing the specimen before and after testing with an accuracy of 0.1 mg can be used to calculate wear loss. Each test was carried out five times, with the average value being used for analysis. SEM is used to examine the wear track on the worn surface. The following parameters were considered during the wear test: (i) reinforcing weight percentage, (ii) applied load, (iii) sliding distance, and (iv) sliding speed.

2.4. Taguchi technique
The Taguchi technique is a powerful experimental tool that allows determining optimal parameters in a straightforward, quick, and systematic manner. When compared to traditional experimentation, this technique significantly decreases the number of experiments required to model response functions. Traditional experimentation involves one-variable-at-a-time tests, in which one variable is altered while the others remain constant. The main drawback in this method is that it ignores any potential interactions between the parameters. An interaction occurs when one element fails to have the same influence on the response of another factor at various levels. In addition, studying all of the components and determining their principal effects (i.e., individual
impacts in a single experiment is unfeasible. All of these flaws are resolved by the Taguchi approach. The response function’s average value at a given level of a parameter is the principal output. The departure a factor level creates from the overall mean response is its effect. The Taguchi methodology is used to optimize processes and find the best combinations of elements reactions. The signal-to-noise (S/N) ratio is calculated from the experimental data. Depending on the sort of characteristics, different S/N ratios are available. Smaller S/N ratios are better, greater S/N ratios are better, and nominal S/N ratios are the best. The S/N ratio for the smallest wear loss and coefficient of friction follows the smaller-the-better rule. The S/N ratio is a logarithmic ratio that can be determined as seen below, transformation of the loss function

\[
\frac{S}{N} = -10 \log \left[ \frac{1}{n} (y_1^2 + y_2^2 + \ldots + y_n^2) \right]
\]

where:
- \(n\) = repeated number of trial
- \(y\) = response the sliding wear characteristics

| Table 3. Process parameters with their different Levels. |
|-----------------------------------------------|
| Process Parameters | Units | Level I | Level II | Level III |
| Si\(_3\)N\(_4\) + Cu(NO\(_3\))\(_2\) reinforcement wt% | 6 + 0 | 12 + 0 | 12 + 4 |
| Load N | 10 | 15 | 20 |
| Sliding Distance m | 1000 | 1500 | 2000 |
| Sliding Speed rpm | 200 | 300 | 400 |

| Table 4. Taguchi Design using L\(_{27}\) orthogonal matrix. |
|-----------------------------------------------|
| L\(_{27}\)(2^13) | wt%of reinforcement | Load (N) | Sliding distance (m) | Sliding Speed (rpm) | Wear Loss (g) | Wear rate (10\(^{-7}\)m\(^3\) Nm\(^{-1}\)) | S/N ratio | Mean |
| 1 | 6% Si\(_3\)N\(_4\) | 10 | 1000 | 200 | 0.018 | 6.6915 | 34.8945 | 0.018 |
| 2 | 6% Si\(_3\)N\(_4\) | 10 | 1500 | 300 | 0.010 | 2.4783 | 40.9151 | 0.009 |
| 3 | 6% Si\(_3\)N\(_4\) | 10 | 2000 | 400 | 0.009 | 1.6729 | 40.9151 | 0.009 |
| 4 | 6% Si\(_3\)N\(_4\) | 15 | 1000 | 300 | 0.006 | 1.4870 | 44.4370 | 0.006 |
| 5 | 6% Si\(_3\)N\(_4\) | 15 | 1500 | 400 | 0.009 | 1.4871 | 40.9151 | 0.009 |
| 6 | 6% Si\(_3\)N\(_4\) | 15 | 2000 | 200 | 0.019 | 2.3544 | 34.4249 | 0.019 |
| 7 | 6% Si\(_3\)N\(_4\) | 20 | 1000 | 400 | 0.030 | 5.5762 | 30.4576 | 0.030 |
| 8 | 6% Si\(_3\)N\(_4\) | 20 | 1500 | 200 | 0.040 | 4.9566 | 32.0412 | 0.040 |
| 9 | 6% Si\(_3\)N\(_4\) | 20 | 2000 | 300 | 0.035 | 3.2169 | 32.3958 | 0.035 |
| 10 | 12% Si\(_3\)N\(_4\) | 10 | 1000 | 200 | 0.024 | 8.8235 | 32.3958 | 0.024 |
| 11 | 12% Si\(_3\)N\(_4\) | 10 | 1500 | 300 | 0.015 | 3.6765 | 36.4782 | 0.015 |
| 12 | 12% Si\(_3\)N\(_4\) | 10 | 2000 | 400 | 0.025 | 4.5956 | 32.0412 | 0.025 |
| 13 | 12% Si\(_3\)N\(_4\) | 15 | 1000 | 300 | 0.035 | 8.5784 | 29.1186 | 0.035 |
| 14 | 12% Si\(_3\)N\(_4\) | 15 | 1500 | 400 | 0.005 | 0.8170 | 46.0206 | 0.005 |
| 15 | 12% Si\(_3\)N\(_4\) | 15 | 2000 | 200 | 0.026 | 3.1869 | 40.9151 | 0.026 |
| 16 | 12% Si\(_3\)N\(_4\) | 15 | 2000 | 300 | 0.020 | 1.8181 | 34.4249 | 0.020 |
| 17 | 12% Si\(_3\)N\(_4\) | 20 | 1000 | 400 | 0.017 | 3.1250 | 39.1721 | 0.017 |
| 18 | 12% Si\(_3\)N\(_4\) | 20 | 1500 | 200 | 0.006 | 0.7353 | 44.4370 | 0.006 |
| 19 | 12% Si\(_3\)N\(_4\) | 20 | 2000 | 300 | 0.035 | 3.6364 | 40.9151 | 0.035 |
| 20 | 12% Si\(_3\)N\(_4\) + 4% CuN\(_2\)O\(_6\) | 10 | 1500 | 300 | 0.005 | 1.2121 | 46.0206 | 0.005 |
| 21 | 12% Si\(_3\)N\(_4\) + 4% CuN\(_2\)O\(_6\) | 10 | 2000 | 400 | 0.004 | 0.7273 | 47.9588 | 0.004 |
| 22 | 12% Si\(_3\)N\(_4\) + 4% CuN\(_2\)O\(_6\) | 15 | 1000 | 300 | 0.008 | 1.9394 | 41.9382 | 0.008 |
| 23 | 12% Si\(_3\)N\(_4\) + 4% CuN\(_2\)O\(_6\) | 15 | 1500 | 400 | 0.010 | 1.6162 | 40.9151 | 0.010 |
| 24 | 12% Si\(_3\)N\(_4\) + 4% CuN\(_2\)O\(_6\) | 15 | 2000 | 200 | 0.003 | 0.3637 | 50.4576 | 0.003 |
| 25 | 12% Si\(_3\)N\(_4\) + 4% CuN\(_2\)O\(_6\) | 20 | 1000 | 400 | 0.011 | 2.0000 | 39.1721 | 0.011 |
| 26 | 12% Si\(_3\)N\(_4\) + 4% CuN\(_2\)O\(_6\) | 20 | 1500 | 200 | 0.010 | 1.2121 | 40.9151 | 0.010 |
| 27 | 12% Si\(_3\)N\(_4\) + 4% CuN\(_2\)O\(_6\) | 20 | 2000 | 300 | 0.005 | 0.4455 | 46.0206 | 0.005 |
Table 3 shows the three levels of investigation for the four process parameters. The studies were carried out under the following conditions: table 4. ANOVA analysis was used to evaluate the percentage of contribution of testing parameters, and mean-response graphs were drawn using Minitab-16 software.

3. Result and discussion

3.1. Density
Theoretical densities for various compositions of base to reinforcement added composite were determined using the law of mixtures, whereas practical densities were determined using the Archimedes principle. In the graph illustrated in figure 3, both theoretical and practical density values were examined. The theoretical density was clearly higher than the practical density. As the percentage of Si₃N₄ in the AA6063–Si₃N₄ composite material grows from 0 to 12 wt%, the density of the material increases by 2.42% and also in comparison to AA6063%-12% Si₃N₄, adding 4% CuN₂O₆ increased the density value by 1.57 percent. The higher density of AA6063 composite is due to the addition of Si₃N₄ CuN₂O₆, which raised the density and filled the void space. The mechanical properties of the composite formed improve as the density increases [30].

3.2. Optical microscope analysis
Optical micrographs of AA6063/12% Si₃N₄/2 and 4% CuN₂O₆ MMCs manufactured by PM method are shown in figure 4. The grey area depicts Si₃N₄ particles, while the black area depicts CuN₂O₆ particles. It was determined from OM pictures that the reinforcing particles in the AA 6063 alloy were dispersed equally. The micrograph clearly depicts the increase in CuN₂O₆ content distribution in MMCs. The propensity reaction and the alloying materials utilized in the processing are to responsible for the good bonding observed between the reinforcement and the matrix. There are no cracks or micropores in MMCs, and the presence of reinforcement particles constantly increases the density and hardness of the composites.

3.3. XRD analysis
Figure 5 shows the XRD patterns of AA6063/Si₃N₄/CuN₂O₆ composites reinforced with Si₃N₄ and 2, 4, and 6% CuN₂O₆ particles as formed by PM. The presence of metallic compounds Al, Cu, Si₃N₄ and CuN₂O₆ particles is revealed by the XRD peaks. The composite of AA6063/Si₃N₄/2%CuN₂O₆ has high Al and Si₃N₄ peaks and weak CuN₂O₆ peaks, as seen in the XRD patterns. It’s because there are fewer CuN₂O₆ particles in the mix. When the amount of CuN₂O₆ was added at 4 and 6 wt%, significant peaks of CuN₂O₆ were observed.
3.4. Hardness

The specimens were tested for hardness in hardness testing equipment. The test was carried out on specimens measuring 10 mm in length and 10 mm in diameter, in accordance with ASTM E10-07a standards and under normal temperature circumstances. The test was conducted on AA6063 composite with varying weight percentage of ceramic and inorganic reinforcement. Each value is based on a five-reading average. The results of the test on the specimen are shown in figure 6. It is clear that the composites’ hardness is substantially higher than that of base alloy. The hardness of the material increases as the reinforcing weight percent increases. The increase in hardness could be attributed to the reinforcement Si₃N₄ having a higher hardness and its presence in the matrix increasing the composites’ overall hardness. Further increase the hardness of composite by adding inorganic reinforcement of CuN₂O₆. The hardness of the AA6063 composite was improved because the reinforcement particles were incorporated into the aluminium matrix, which increased their surface area and reduced the size of the aluminium matrix grains. The presence of these hard surface areas of Si₃N₄ particles provides a significant amount of resistance to plastic deformation, resulting in an increase in the hardness of powder manufactured AMCs. Furthermore, the presence of hard and brittle Si₃N₄ particles in the soft and ductile AA6063 matrix reduces the ductility content of processed AMCs due to the low ductile percentage of matrix metal in the composite, which improves the hardness of produced AMCs significantly. With increasing
copper nitrate content, alloy formation also increases, which leads to the formation of a continuous layer between the metal matrix and the copper particles. Deformation becomes far more difficult, resulting in higher hardness values as reported [31]. In addition for improvement of hardness of composite due to a thermal mismatch between the aluminium matrix and the reinforcement, increasing the amount of reinforcement in the matrix causes an increase in dislocation density during sintering process. Because of the temperature differential, the mismatch of thermal expansion between the aluminium matrix and reinforcement causes huge internal stress, causing the aluminium matrix to deform plastically to lodge the smaller volume expansion of Si₃N₄ and CuN₂O₆ particles. Increased dislocation density at the particle–matrix interface results in increased hardness and resistance to plastic deformation. Si₃N₄ particles have a thermal expansion coefficient of 3.7 10⁻⁶ °K⁻¹, while AA6063 has a thermal expansion coefficient of 2.34 10⁻⁵ °K⁻¹. The mismatch of thermal expansion between the matrix and reinforcement particles during the sintering process at 520 °C for 1 h leads in higher dislocation density in the matrix and load bearing capability of hard reinforcement particles, increasing the composites’ hardness. The lower thermal coefficient of thermal expansion particle of Si₃N₄ increases in the higher coefficient of thermal expansion matrix of AA6063 during the process, causing the matrix’s microstructural features to change with a simultaneous contribution in increasing hardness. This is consistent with the findings of previous researchers [23]. Previous research found that increasing the quantity of reinforcement in the matrix increased the hardness of AMCs regardless of the fabrication procedure [17].

3.5. ANOVA and the effects of factors
The impact of various process parameters such as CuN₂O₆ reinforcement, Load, sliding distance, sliding speed and their interactions, to determine the order of significant factors and their interactions, an analysis of variance (ANOVA) table 5 was developed. This study was conducted with a 5% level of confidence in the validity of the results. In this study, the results of the ANOVA of hybrid composites in terms of wear are shown in table 5. Interaction between reinforcement and load (p = 31.66%), reinforcement and sliding distance, CuN₂O₆ reinforcement (p = 28.47%) had a significant impact on wear loss, as seen in table 5. The contributions of load, sliding distance and sliding speed alone were less influential on wear loss, while the interactions between reinforcement and sliding speed had no effect on wear loss.

3.6. Function of process parameters on wear loss
Figure 7 shows the main effect of the influence of process parameter on wear loss of the composites. If the line for a particular parameter in the main effect plot is near horizontal, the parameter has no significant effect. A parameter for which the line has the greatest inclination, on the other hand, has the greatest impact. The process
The parameter of reinforcement had the largest influence, according to the studies. Other testing variables including load, sliding distance, and sliding speed had a smaller impact. The relationship between the reinforcement and the load, on the other hand, has a greater impact on wear loss. Wear loss increased as the applied load and sliding distance increased, indicating that more material was removed from the surface. Furthermore, wear loss decreased as the sliding speed increased due to the creation of a copper rich passive layer on the worn surfaces, reducing wear by covering more contact area. When compared to other situations (i.e., 0% and 2%), the composite with 4% reinforcement demonstrated improved wear resistance at greater loads.

3.7. Using the taguchi method analyze and evaluate the findings of the tests

The signal/noise ratio ($S/N$) is the most important criterion in the Taguchi method for assessing experimental data which is depicted figure 7. According to the Taguchi approach, the $S/N$ ratio in this study should have a maximum value in order to provide optimal testing conditions of wear loss. The wear loss $S/N$ response table for powder processed hybrid composites is provided in table 6. The average of the selected features for each level of the components is shown in the response table. The ranks in the response table are based on Delta statistics, which measure the relative magnitudes of impacts. The Delta statistic is the sum of the highest and lowest averages for each element. The highest Delta value is assigned to rank 1, the second highest Delta value is assigned to rank 2, and so on. For a good wear resistance value, the optimal conditions were 4 wt. percent CuN$_2$O$_6$, 15 N load, 2000 m sliding distance, and 200 rpm sliding speed.

3.8. Multiple linear regression analysis and confirmation test of experiment

A multiple linear regression equation was developed using the statistical software MINITAB R14. By fitting a linear equation to the observed data, this produced model provides the link between an independent/predictor variable and a response variable. The wear rate regression equation is as follows:

$$
Wear\ loss = 0.0066 + 0.00394 \times A + 0.001617 \times B \\
+ 0.000003 \times C - 0.000046 \times D - 0.000508 \\
\times A \times B - 0.000002 \times A \times C + 0.000013 \times A \times D
$$

Where, $A =$ reinforcement, $B =$ Load, $C =$ Sliding distance, $D =$ Sliding speed

Verification tests are required after the optimum level of testing parameters has been determined in order to assess the accuracy of the analysis and validate the experimental results. Table 7 indicates the experimental values for conducting the dry sliding wear test & adopting the best condition to compare the estimated wear result with the actual wear rate. According to the analysis, the actual wear rate varies from predicted using regression equations, with error percentages ranging from 6.42% to 9.25%.

| Source                        | DF  | Adj SS   | Adj MS   | F-Value | P-Value | Percentage of contribution |
|-------------------------------|-----|----------|----------|---------|---------|---------------------------|
| wt% Reinforcement             | 2   | 0.000873 | 0.000436 | 41.92   | 0.000   | 28.74                     |
| Load (N)                      | 2   | 0.000212 | 0.000106 | 10.19   | 0.012   | 6.46                      |
| Sliding distance (m)          | 2   | 0.000143 | 0.000072 | 6.88    | 0.028   | 4.13                      |
| Sliding Speed (rpm)           | 2   | 0.000072 | 0.000036 | 3.46    | 0.100   | 1.73                      |
| wt% Reinforcement x Load      | 4   | 0.000980 | 0.000245 | 23.53   | 0.001   | 31.66                     |
| wt% Reinforcement x Sliding distance | 4   | 0.000429 | 0.000107 | 10.30   | 0.007   | 13.08                     |
| wt% Reinforcement x Sliding Speed | 4   | 0.000196 | 0.000049 | 4.70    | 0.046   | 5.23                      |
| Error                         | 6   | 0.000062 | 0.000010 |         |         | 8.95                      |
| Total                         | 26  | 0.002966 |          |         |         | 100                       |

$S = 0.0032261$, $R$-Sq $= 97.82\%$ and $R$-Sq (adj) $= 90.88\%$
3.9. Effect of reinforcement on coefficient of friction

The coefficient of friction was influenced by the three compositions with varying weight percentages of Si₃N₄ and CuN₂O₆, applied load, and sliding speed, as shown in figures 8–10. On AA6063/6%Si₃N₄, the coefficient of
friction was reduced by 56.25% when the load was increased from 10 to 20 N and the sliding speed was increased from 1000 to 2000 rpm. Furthermore, the coefficient of friction was reduced by increasing the reinforcement of Si₃N₄ from 6% to 12% and adding 4% CuN₂O₆. The presence of hard Si₃N₄ in the aluminum matrix, the coefficient of friction is lowering. Because of the smoothening of aluminum composite by adding soft CuN₂O₆ reinforcement, the coefficient of friction decreases as the load increases. At a higher load of 20 N, AA6063/12%Si₃N₄/4%CuN₂O₆ had the lowest coefficient of friction of 0.26. When the hard particles are introduced, the surface will have sharp edges, which will prohibit it from perfectly mating with adjacent sliding surfaces. The coefficient of friction was reduced as a result.

3.10. EDS analysis
Under dry sliding conditions, energy dispersive spectroscopy (EDS) of the worn surfaces of the powder treated composite was done. The presence of an oxygen peak in all EDS studies of the hybrid composites produced suggested that oxide formation of the worn surface had occurred due to the inclusion of CuN₂O₆ inorganic reinforcement. The combined action of temperature increase and environmental response can lead to oxide film deposition at the contact surfaces while the composite is sliding on a steel counter surface (EN31). Figure 11 shows that the EDAX spectrum of the worn surface of the AA6063/12%Si₃N₄. The EDAX of the worn surface reveals the presence of two-element peak intensities such as Al and Si. However, under dry sliding conditions, the Al intensity peak showed plastic deformation of the composite. Figure 11 compared to the EDAX profile of AA6063/12%Si₃N₄ worn surface, the acetone peak were observed. The intensity of acetone were increased as the CuN₂O₆ reinforcement increases. This acetone generated a protective oxide layer on the composite’s surface. As inorganic reinforcement increases, the peak of Ac rises, reducing wear rate. All fabricated composites had a carbon peak, which increased the work hardening rate of the composite, which increased abrasion resistance. However, because the steel counter surface material was abraded by the Si₃N₄ particles, a notable Fe peak was discovered. Figure 11 shows that Fe-rich materials protect the worn surfaces of wear specimens. Si₃N₄ acts as an abrasive on these surfaces, removing material from the disc’s surface. When compared to matrix alloys, the physical manifestation of Fe-rich coverings on worn surfaces significantly reduces composite wear loss.

![Figure 8. Applied load versus Coefficient of Friction on AA6063/6%Si₃N₄.](image)

| Experiment No. | Experimental wear loss (g) | Regression equation wear loss (g) | % Error |
|----------------|---------------------------|----------------------------------|---------|
| 1.             | 0.0185                    | 0.017                            | 8.82    |
| 2.             | 0.0406                    | 0.038                            | 6.48    |
| 3.             | 0.0087                    | 0.008                            | 9.25    |

Table 7. Confirmation test using regression equation model findings and experimental data.

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Figure 9. Applied load versus Coefficient of Friction on AA6063/12%Si₃N₄.

Figure 10. Applied load versus Coefficient of Friction on AA6063/12%Si₃N₄/4%CuN₂O₆.

Figure 11. EDS analysis on the worn surface.
existence of a low-intensity oxygen peak was seen in all of the composite EDX data. This peak suggested that some oxide development had occurred on the mated parts’ worn surfaces. The combined action of the increased temperature and the environmental response can cause the development of an oxide film on the contact surfaces during the sliding of the composite on the steel counter surface.

3.11. Wear surface studies

The manufactured composites have fewer concentrations of carbon, silicon, and iron. Chemical reactions, such as oxidation, are stimulated by the local heating created during a wear test. The production of oxides, particularly aluminum oxides and iron oxides, is quite likely to have happened on the worn surface, which can help reduce the wear rate of the composite as copper nitrate increases by acting as an in-situ solid lubricant.

A significant difference was detected while examining AA6063/12%Si₃N₄/2% CuN₂O₆ with the main factor governing wear being the creation of a generalized mechanically mixed layer (MML), as illustrated in figure 12. This MML, which may contain iron and aluminum oxide, appears to be able to withstand more frictional heating without adhesion. Furthermore, the hybrid composites have less plastic deformation, indicating a stronger resilience to wear, as seen by the hybrid composites’ lower wear rate.
It is well known that increasing the amount of ceramic reinforcement Si$_3$N$_4$ and inorganic copper nitrate particles in AA6063 treated by PM methods improves the tribological characteristics of the material, since Si$_3$N$_4$ and copper nitrate particles protect the matrix from wear (Silicon nitride reference). Furthermore, as compared to ceramic reinforcement alone composites, the hybrid’s greater wear resistance can be explained by thermal expansion dislocation strengthening and matrix work hardening (due to both reinforced particles). Figure 12 shows the AA6063 alloy’s worn microstructure reveals a large amount of primary Al alloy plastic movement. The amount of heat generated between the rolling pin’s surface and the counter disk’s surface, causing plastic deformation. It was discovered that the mode of wear is adhesive. The worn micrograph of the composite shows shallower grooves and fewer pits. The depth of the grooves was lowered due to the presence of Si$_3$N$_4$ and inorganic reinforcement of CuN$_2$O$_6$ particles. Furthermore, the lesser degree of plastic deformation at the groove’s borders is visible. The abrasive wear mechanism has been discovered.

4. Conclusion

The following are some of the most important findings from investigations on AA6063/Si$_3$N$_4$ CuN$_2$O$_6$ MMCs:

The production of AA6063-Si$_3$N$_4$/CuN$_2$O$_6$ MMCs with reinforcement content up to 12% of Si$_3$N$_4$ and 4% of CuN$_2$O$_6$ using powder metallurgical techniques (solid-state process) was successful. The MMCs’ densities were higher than the basic alloy’s. When 12wt% Si$_3$N$_4$ is added to a composite, the density increases by 2.47 percent when compared to the base alloy. Further, CuN$_2$O$_6$ was added to increase 1.57 percent more. The hardness of composite increased about 74.72% as Si$_3$N$_4$ added 12%, and further increased 34.71% as 4% CuN$_2$O$_6$ added as compared to base alloy due to thermal mismatch between the matrix and reinforcement. The order of percentage of reinforcement, sliding speed, applied stress and sliding time all influence the wear rate. According to the results of an ANOVA test, as the percentage of reinforcement increases, the wear rate drops dramatically. The best conditions for wear rate were 12wt% Si$_3$N$_4$ reinforcement and 4wt% CuN$_2$O$_6$ reinforcement, 15N applied load, 2000m sliding distance, and 200 rpm sliding speed, respectively. Among the wear factors, percentage of reinforcement (28.74%) has the highest physical qualities as well as statistical influence on the dry sliding wear rate of composites, compared to other parameters such as applied load (6.46%) and sliding distance (4.13%). In comparison to other parameter interactions, the interaction between reinforcement and applied load (31.66%) had a substantial impact on the wear rate of manufactured composites. The composite’s worn micrograph exhibits shallower grooves and fewer pits. The inclusion of Si$_3$N$_4$ and inorganic reinforcement of CuN$_2$O$_6$ particles reduces the depth of the grooves.

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Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

Conflict of interest

There is no conflict of interest

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