Tribological, Thermal and Corrosive Behaviour of Aluminium Alloy 2219 Reinforced by Si₃N₄ Nanosized Powder

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Abstract

The Metal Matrix Composite (MMC) technique is the most effective contrast method when compared with other techniques. By using the method of high energy stir casting, Aluminium alloy Al2219 is reinforced with various percentages of Si₃N₄ (0, 3, 6, and 9 %) particles. X-ray diffraction along with Scanning electron microscope was performed to characterize the composite. The mechanical and thermal behaviours such as differential thermal analysis thermo gravimetric analysis, tensile, wear and hardness behaviours were investigated. By using electro chemical potentiodynamic polarization test, the consequence of heat treatment on the corrosion behaviour of the composites when compared to its matrix in 3.5 % NaCl when at 600 rpm was also investigated. In this experimental study, the wear of the aluminium composites was significantly decreased on addition of Si₃N₄ particles. The study also revealed that, since the inclusion of Si₃N₄ in the samples and compared to the base aluminium alloy, the mechanical properties of the composites, such as wear resistance, hardness and tensile strength increased by percentage. The surface morphology and Scanning electron microscope analysis of worn surfaces in the test pieces unfold that with the increase in reinforcement content, wear rate decreases.

Keywords Si₃N₄ · Thermal analysis · Wear · Corrosion analysis · SEM

1 Introduction

In the recent times, the technology space in aircraft, aerospace, and automotive industries is rapidly advancing which in turn increases the demand for composite materials which is widely being used in the above-mentioned areas. The special properties of Composites materials such as low specific gravity makes the material highly superior in modulus and strength when compared with most traditional engineering metallic materials [1, 2].

Metal Matrix Composites (MMCs) are explicitly utilized composite materials which is made with generally two constituents, one of the constituents is a metal matrix and the other material is a reinforcement. In all the cases, the matrix is outlines with a metal and the use of pure metal as a matrix is mostly avoided. In general, the matrix is constituted by the use of an alloy. The general synthesis of the composite involves the mixing of the matrix and the reinforcement together.

Aluminium is extensively used in MMCs. This is main reason for this is the unique features of aluminium such as good mechanical properties, low density, good machinability properties, low electrical resistance, high strength and good corrosion resistance. Although comparatively substandard wear resistance of these alloy has restrained its use in specific tribological applications [3, 4]. In the recent studies, both particulate and fibre reinforced aluminium alloy composites fabricated have shown appreciable improvement in their tribological properties, including but not limiting to sliding wear, seizure resistance, friction, and abrasive wear [5, 6]. The production and utilization of Si₃N₄ was stimulated by increasing demand for low-cost reinforcement [7–9].
Wear based damages to the material is an important aspect in tribology buy even though it is highly important, it is one of the least understood field. It is also a youngest topic, lubrication, friction and wear which needs more scientific attention. The practical significance of the same is being identified through the years [10–12].

Wear is responsible for a huge annual outlay by the consumers as well as the industries. Most of this goes in replacing or repairing equipment’s that is worn out and does no longer perform its function [13]. In many parts of the machine the damage is seen only after a percentage of the entire volume of the part has worn out [14, 15].

Number of studies have been conducted to characterize tribological behavior of Aluminium-based MMC: Suresh and Sridhara [16] reported on the impact of Gr and SiC (up to 10 % wt%) on the wear properties of LM25-based aluminium composites. It was discovered that increasing the weight fraction of reinforcement particle in MMC reduced wear until it reached 7 %, after which it increased. Wear of matrix material composites increased with increasing load due to an unstable tribo-layer, but decreased with increasing speed due to the presence of Mechanically Mixed Layer (MML) [17]. The effect of SiC and B4C on the wear efficiency of the Al7075-based MMC was studied by Uvaraja and Natarajan [18]. Wear rate was found to decrease as the volume fraction of reinforcement, speed, and time increased, whereas it increased as the applied load increased. The reinforcement content was found to be the most important factor, followed by the applied load and sliding speed. Surface morphology and SEM analysis of the test pieces’ worn surfaces showed that the wear rate decreased as the reinforcement material increased. The EDS findings were used to classify the MML that forms on the test specimen’s worn surfaces [19, 20]. The zinc effect on the microstructure was investigated by keeping the ratio of zinc in the mixture at different ratios for each mixture. X-ray diffraction (XRD) analysis was carried out to investigate the different phases. Analyzes of the microstructures of alloys with different alloying times were investigated by scanning electron microscopy (SEM) and optical microscope [21, 22].

The effects of reinforcement on composite corrosion activity are still unknown. It has been found that in the presence of reinforcement, the corrosion current density rises, decreases, or remains unchanged. Furthermore, reinforcement has been shown to influence the open circuit potential (OCP) by increasing, decreasing, or having no effect [23, 24]. Heat treatment results are discovered to be a crucial factor in evaluating the corrosion behaviour of aluminium alloy composites. Kolman and Butt [25, 26] examined the corrosion properties of an aluminum–silicon alloy composite reinforced with in-situ TiB2 particulate after heat treatment. It was discovered that as the amount of TiB2 in these composites increased, their corrosion resistance decreased. The effect of heat treatment of the reinforcing BN, Al2O3, and Ti (C, N) particles in the EN AW–AlCu4Mg1(A) aluminium alloy on its corrosion resistance in the presence of NaCl water solution was investigated by Wodarczyk-Fligier et al. [27, 28]. The corrosion resistance of composite material heat treated in a 3 % NaCl solution was found to be noticeably improved [29]. The tensile strength of the samples decreases, the elongation of the extruded samples increases. Consequently, reciprocating extrusion is an effective method for improving the mechanical properties of metal matrix composites (MMC) [18, 30].

In the context of the above discussion, it has been found that no work has been carried out by the combination of Al2219/ Si3N4 with different composition. Also, characterization such as TGA, Corrosion have not been conducted by other researchers. So, the aim of the present work is to study the Tribological, Thermal and Corrosive behaviour of aluminium alloy 2219 reinforced by Si3N4 nanosized powder. The reinforcement is embeded by high energy stir casting method with various percentage of Si3N4 (0,3,6 and 9) particles. The structural characterization was carried out by SEM and XRD tests. Tensile, hardness and corrosion test are carried out for determining the mechanical behaviour of this material. TGA analysis has been carried for the purpose of thermal analysis. The wear behaviour of AA2219/ Si3N4 MMC has not been explored so far and they are fabricated by two-step stir casting method. Characterization of microstructure and worn surface morphology of the composites was done by Scanning electron microscope (SEM). Tensile fractured samples were made to undergo fractography study. Mechanically mixed layer (MML) of the test specimens were evaluated by EDS.

2 Experimental Aspects

2.1 Casting Process

In this study the electrical furnace setup was replaced with a stir set up by using a simple blower furnace and the vertical drilling machine assembly as shown in the Fig. 1. As the high operating cost of electrical furnace is annihilated, the variable speed of drilling machine is obtained using the stirrer. The addition of Si3N4 particles in terms of ratio was done at 0, 3, 6, and 9 % by the overall weight. Formulation process begins with the stirring out in a graphite crucible in a coal-fired furnace. Ceaseless stirring of the molten metal-matrix gives homogeneous mixture of the composite. This is instantly poured into the mould to get solidified. For melting the alloy Coal was used as a fuel. Al2219 was kept in a crucible and liquefied by melting it in a blower furnace at a temperature of 670°C for 15 min. The Si3N4 powder was preheated to a temperature of 670 °C using a separate muffle furnace. The temperature of the furnace was first increased above the temperature of the liquid.
2.2 Tensile Test

Ductile test examples were made according to the ASTM standard and tried in a Universal Testing Machine. In request to gauge the rigidity of the examples, they were made in a round and hollow shape as per ASTM E8. The information estimations of the four examples acquired from the ductile test were utilized. The fortifying stage in the metal network composites bears a huge division of the worry as it is commonly a lot stiffer than the grid. The molecule joining brings about an expansion in the work solidifying of the material. The higher work solidifying rate saw in the composites is because of the mathematical imperatives forced by the presence of the support. The expanding weight % of Si₃N₄ builds the work solidifying rate. The tensile samples before and after test is shown in Fig. 2(a) and (b).

2.3 Hardness Test

The composites materials hardness was estimated by utilizing a Brinell hardness machine following the ASTM E10 standard. All the examples were applying a heap of 500 to 3000kgf for a time of Ten seconds. The test was done at room temperature and the estimation of hardness was taken at 3 distinct areas to keep away from the conceivable impact of indenter laying on the hard support particles. The midpoints of the apparent multitude of four readings were accounted for.

2.4 Pin-on-disc Experimental Set Up

A pin-on-disc equipment is used to carry out the experiment which is coupled with a wear monitor and type friction with a data acquisition system. The above setup is used in measuring the wear behavior of composite by testing it against the hardened ground steel disc (EN-24) which is has a hardness value of 65HRC and surface roughness (Ra) of 0.5 μm. the equipment is highly versatile which is designed in such a way to study the wear characteristics only under sliding conditions. In the equipment, sliding usually occurs between the rotating disc and the stationary pin. A D.C motor was used to rotate the disc; the motor is having a speed range of 100–1500 rpm with wear track diameter (50mm*160mm), which would yield
sliding speed 0 to 10 m/sec. The load has to be applied on pin sample (specimen) by deadweight through a pulley string arrangement. The system’s maximum loading capacity is 100 N. Figure 3(a) & (b) shows Wear specimens before and after test.

2.5 TGA

Differential thermal analysis (DTA) and thermogravimetric analysis (TG) were carried out simultaneously using a TG/DTA EXSTAR 6300 instrument (SII Nanotechnology Inc.). Approximately 10 mg of the specimen is weighed on the alumina crucible and heated from 30 to 800° in a flow of air atmosphere (100 ml/min). The heating rate was 10°/min. α-Alumina was used as reference standard.

2.6 XRD

XRD is commonly utilized to assess the samples’ purity, phase and crystal structure [25]. Phase analysis of prepared specimens was done by XRD model D2 PHASER (Bruker AXS) using Cu/Kα radiation (λ = 1.54060 Å). Over the 2θ range of 20°-80° with a step size of 0.02°, peak values were obtained. Different shapes correspond to a different growth of crystal structures resulting on different intensities of the XRD peaks. Characteristics peaks has been observed in the XRD pattern, and the relative intensity rate corresponding to the samples.

2.7 Corrosive Test

Corrosion measurement were carried out using CHI 604D Electrochemical Workstation (CH Instruments, Inc) in 3.5 % NaCl Solution using an Ag/AgCl reference electrode and
platinum wire as a counter electrode. The Inhibitive efficiency is calculated using the below Eq. (1).

\[
\text{Inhibition Efficiency} = \frac{C_{ra} - C_{rs}}{C_{ra}} \times 100
\]  

(1)

In the above equation for Inhibition Efficiency, \(C_{ra}\) denotes the corrosion rate of the unreinforced Al2219 alloy and \(C_{rs}\) is used to denote the corrosion rate of Al2219 alloy reinforced with Si\(_3\)N\(_4\). Each run’s output consists of a polarisation curve from which the corrosion parameters can be calculated using a manufactured software package using the Tafel extrapolation technique. The samples are shown in Fig. 4.

Fig. 5  Effect of tensile strength on wt% of Si\(_3\)N\(_4\)

Fig. 6  (i): Fractography of Al 2219 Alloy + 0 % Si\(_3\)N\(_4\), (ii): Fractography of Al 2219 Alloy + 3 % Si\(_3\)N\(_4\), (iii): Fractography of Al 2219 Alloy + 6 % Si\(_3\)N\(_4\), (iv): Fractography of Al 2219 Alloy + 9 % Si\(_3\)N\(_4\)
3 Result & Discussion

3.1 Effect of Tensile Analysis

Mechanical conduct of the composites was explored by pliable tests which are done utilizing a mechanized widespread malleable testing machine. The examples when the ductile test has appeared in Fig. 5. Four test examples were utilized for each run. The malleable properties, for example, rigidity, extreme elasticity, and modulus were deciphered from the pressure strain bends spoke. It is additionally obvious from the malleable test that expansion in the measure of support builds the elasticity, whereas there exists a lessening in pliability huge enhancement in the mechanical properties of the composite as contrasted and the Al2219 can be ascribed to the Si3N4.

The tensile tested specimen of AA2219 alloy with 0 %, 3 %, 6 and 9 % of Si3N4 reinforced composite is subjected to fracture morphology and the result is as shown in Fig. 6(i–iv). It has been noted that sample with 3 % wt demonstrated a high breakage point. Scanning electron microscope image of AA2219 alloy in the Fig. 6(i–iv) unravels that the AA2219 alloy has equally distributed, large voids, with a good number of comparatively equalled fibrous ligaments and dimples present on its surface. This discloses the ductile mode of the fracture. The composites 0 %, 3 %, 6 and 9 % of Si3N4 as shown in figure are small and minimal. Within the dimples the Si3N4 particles are excellently harboured. This confirms a good bonding between the matrix and the Si3N4 [30]. Because of the existence of Si3N4 particles, the dimples were found to be much shallower, and their size was decreased by growing a fraction of Si3N4 particles. This shows that the particles are uniformly distributed and strongly bound to the matrix. Despite the fact that fracture morphology shows signs of ductile fracture, ductile fracture is heavy in the AA2219 alloy and low in the composite. These fractography clearly demonstrate that the matrix and reinforcement have a strong bond, resulting in improved composite hardness and ultimate tensile strength [13, 30]. It confirms that as the percentage of Si3N4 increases, the percent of elongation decreases. This is mostly due to the Si3N4 particle’s hardness. Many other researchers have found similar patterns [1, 13, 21, 30].

3.2 Brinell Hardness Analysis

The after-effects of Brinell hardness tests directed on AA2219 and the composite containing diverse wt% of Si3N4 particles have appeared in Fig. 7. They uncover critical improvement in the hardness with the expansion of Si3N4 fortification in the AA2219. The AA2219 composite with hardness esteem 63.71 HV and the aluminium-based composite containing 2 wt% Si3N4 demonstrates 65.39 HV and this goes with expanding Si3N4 content. It is seen that the hardness of 10 % Si3N4 is expanded by 73.93 HV more than AA2219. A striking accent in the hardness of the composite network can be seen with the expansion of Si3N4 particles. Hardness esteem uncovers that

### Table 1 Wear rate of all the samples with 20KN and 40KN Load

| Load (N) | Sliding speed (Rpm) | Wear rate in $10^{-7}$ mm$^3$/Nm |
|----------|---------------------|---------------------------------|
|          | 0                   | 3                               | 6                       | 9                       |
| 20       | 300                 | 1.527                           | 1.319                   | 1.041                   | 1.180                   |
|          | 600                 | 1.562                           | 1.388                   | 1.145                   | 1.249                   |
|          | 900                 | 1.666                           | 1.458                   | 1.272                   | 1.342                   |
| 40       | 300                 | 2.013                           | 1.527                   | 1.458                   | 1.527                   |
|          | 600                 | 2.326                           | 2.152                   | 1.701                   | 1.874                   |
|          | 900                 | 2.430                           | 2.360                   | 1.944                   | 2.129                   |

Fig. 7 Effect of hardness on wt% of Si3N4 particles

Fig. 8 Wear rate vs. Wt. % of Si3N4 (load: 20 N)

Fig. 8 Wear rate vs. Wt. % of Si3N4 (load: 20 N)
the higher estimation of hardness demonstrates the presence of Si$_3$N$_4$ particulates in the lattice composites. A hard fortification is consolidated into a delicate material; the hardness of the grid material is enhanced. Consequently, expanding the wt% of Si$_3$N$_4$ builds the hardness of the composites.

### 3.3 Effect of Wear Analysis

The above Fig. 8 shows effect of wt% of Si$_3$N$_4$ and sliding speed on wear behaviour of the AA2219/Si$_3$N$_4$ composites at 20 N. Table 1 shows the Wear rate of all the samples with 20KN and 40 KN Load.

The above Fig. 9 shows the Effect of wt% of Si$_3$N$_4$ and with sliding speed of disk on wear behaviour of the Al2219/Si$_3$N$_4$ composites at 40 N. The effect of Silicon Nitride (Si$_3$N$_4$) content on the wear characteristics of AA 2219/Si$_3$N$_4$ particulate for a wear test the loads of 20, and 40 N and rotational speed of 300, 600, and 900 rpm as shown in Figs. 7 and 8 which is the representative graphs plotted based on wear rate results. The following is revealed by the study of these Figures. The wear rate of the AA 2219/Si$_3$N$_4$ composites depends on the % of Silicon Nitride (Si$_3$N$_4$) dispersion. With the increase in Silicon Nitride (Si$_3$N$_4$) content from 3 to 6 wt% wear rate was found to be decreased. But there is a result of an increase in wear rate for the 9 wt% of Silicon Nitride (Si$_3$N$_4$) when compared with the 6 wt% reinforcement. The weighted rate by wear is minimum for the composite containing 6 wt% Silicon Nitride (Si$_3$N$_4$) dispersed in the as-cast as observed. At lower loads, the wear rate of the material remained almost constant with an increase in rpm. When comparing the results,
the light wear rate was shown by AA2219 without dispersoid loss decreased in a steady manner. Perhaps, due to hard particles of Silicon Nitride (Si₃N₄) dispersed in the base matrix there is relatively rapid attainment of stability in the wear resistance, as seen in the above figures.

3.4 Worn Out Surface Analysis of Wear Specimens

Figure 10(a) – (d) shows the surfaces of samples as viewed using a SEM which is cast Al2219 alloy and Al2219-3,6,9 wt% Si₃N₄ composites after applying a load of 20 N, 40 N and 600, 900 rpm sliding speed test. Figure 10(a) shows that some of the regions are damaged as seen in as cast Al2219 alloy. When higher load was applied, the degree of grooves formed at the worn surface of the matrix alloy is quite larger which causes a severe plastic deformation which causes severe wear in the specimens. Figure 5.2, 5.3, 5.4 shows the amount of grooving in the surfaces of the Al2219 alloy composite. The grooves reduce as a result in the increase content of Si₃N₄ showing the lower material removal in comparison with Al2219 base matrix material.

3.5 XRD Investigations

Figure 11(a) shows the X-ray patterns of extracted AA2219 as cast composites. The fabricated AA 2219’s X-ray diffraction pattern. The presence of aluminium (Al) and copper (Cu) of metallic compounds was discovered through the study of XRD peaks. The diffraction pattern clearly indicates Si₃N₄ particles, as shown in Fig. 11(b, c, and d). The relative fractions of the strength of the Si₃N₄ particles were measured. The relative fractions of Si₃N₄ particles found strength in the XRD pattern, with the highest peak occurring at the reinforcement particles. The particle peaks within the composite are visible using XRD. As the number of Si₃N₄ particles increases, the probability of reinforcement agglomeration in the AA 2219 matrix increases. The results show that metallic elements are present in the highest peaks and reinforcement is present in the lowest peaks. The relative strength occurs at a 38-degree angle, with strong peaks for wt% of composites. The percentage of Si₃N₄ in composites has been found to increase, resulting in a higher peak intensity level in the study.

3.6 TGA Analysis

Analyzing the thermal response of unreinforced Al2219 and developed Al2219- Si₃N₄ composites as shown in Fig. 12(a-d), has been established using a differential thermal analyzer (DTA) and thermogravimetric analyzer (TGA). The results from the thermograms shows that as the composite with incorporated Si₃N₄ has enhances the thermal stability of the aluminium alloy matrix. Apart from increase in inhibition efficiency, the addition of the nanoceramic Si₃N₄ also causes a decrease in the mass loss as the composite is heated from 30
to 800 °C, which is clearly evident from the TG/DTA result. From the graph, there was a 7.3% loss of mass for the unreinforced Al2219, whereas the addition of the incorporation of Si3N4 decreased the mass loss to 2.5% in 3 wt% Si3N4 reinforced alloy. At 6 wt% Si3N4, the material was about 4.4% and mass loss was observed in 9 wt% particulates with an 7.6% loss. This shows the positive effect of the inclusion of the Si3N4 helped in material saving for high temperature application by enhancing the reduction of mass loss with an optimal Si3N4 amount of 3%.

3.7 Corrosion

Figure 13 shows the potentiodynamic polarization graph for the Al 2219 allow with and without Si3N4 reinforcement. From the results, the corrosion rate and the inhibition efficiency were calculated and is shown in Table 2. the results clearly shows that the corrosion rate decreases as with the addition of Si3N4 which shows its positive effect on the material in preventing it from getting corroded. The inert properties of the nano ceramic Si3N4 that makes it resistant to corrosion.
was seen to influence the composite to have a low corrosion rate, its positive effect on the corrosion inhibition efficiency increases as the Si$_3$N$_4$ percentage increases from 3 to 9 %.

4 Conclusion

The tribological, thermal, morphological and mechanical characterisation of AA2219 reinforced with four wt% of Si$_3$N$_4$ composite was made by stir casting method. The study revealed the following results:

- Due to the dispersion of Si$_3$N$_4$ particles over the Aluminium alloy 2219, matrix increases the hardness value of the composites. Tensile strength increases firstly from 0 to 6 % Si$_3$N$_4$ and decreases suddenly at 9 % Si$_3$N$_4$. HBW of the material gradually increases as percentage composition of aluminium varies from 0 to 6 % Si$_3$N$_4$ and decreases in 9 % of Si$_3$N$_4$.
- Tensile strength of AA2219-Si$_3$N$_4$ composite revealed that high strength is obtained at 6 %wt addition of Si$_3$N4 when compared to other the weight %. The intensity of facts initial will increase from 0 to 6 % and then suddenly

Fig. 12 (continued)
to 9%. The results of the tests like hardness and tensile increased with increasing the wt% of Si₃N₄ in AA2219 composites [10].

- The XRD analysis of Al 2219 alloy / Si₃N₄ composites reexhibits the aluminium, Silicon Nitride (Si₃N₄) and intermetallic (Al-Cu) phases.

- The tribological characteristics mass wear, friction force and coefficient of friction, of the AA2219-Si₃N₄ composites increases with the 9% of Si₃N₄ reinforcement. The AA2219-with 0, 3, 6, and 9 wt% of Si₃N₄ composites has shown the lower rate wear compared to the original AA2219 alloy matrix. Work concluded that the 9% of Si₃N₄ reinforcement is better for the wear resistances at minimum load applications of Aluminium alloy 2219 matrix materials, the minimum amount of 6% of Si₃N₄ reinforcement is to be suggested.

- The coefficient of friction and wear rate the of Al/Si₃N₄ MMC was found to be lower than that of AA2219. The smaller and finer grain size, improved hardness, reduction in the porosity level and uniform distribution of Si₃N₄ particles are the various reasons for this. When the wt% of Si₃N₄ content increase the wear rate is found to be decreased with an increase in sliding velocity [26].

- The worn area shows a rough wear process, which is mainly the product of hard particulate exposure. In the case of AA2219/ Si₃N₄ Composites, the particulate inhibits delamination progression, while the wear resistance is supplemental.

- The corrosion resistance of the matrix alloy was improved when Si₃N₄ particles were added in a quantity of up to 9% by weight. The AA2219- Si₃N₄ alloy matrix composite showed high corrosion resistance with corrosion rates of 367.00 mil/year for 6 wt% Si₃N₄ and 170.60 mil/year 9 wt% Si₃N₄. Heat treatment of the composites and matrix alloy improved corrosion and wear resistance.

### Table 2
| Sample | Corrosion rate (mil/year) | Inhibition efficiency (%) |
|--------|---------------------------|---------------------------|
| 0 %    | 455.70                    | 12.2                      |
| 3 %    | 346.20                    | 24.03                     |
| 6 %    | 367.00                    | 19.46                     |
| 9 %    | 170.60                    | 62.56                     |

**Fig. 13** Polarization curves for Al– Si₃N₄ alloy matrix and its composites with different wt% of Al2O3 particles tested in 3.5 % NaCl
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