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RESEARCH ARTICLE



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Enhancing mechanical and functional properties of LM25 alloy through squeeze cast hybrid nanocomposite incorporating breadfruit seed husk ash and graphite nanoparticles

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ABSTRACT

In this research, effective utilization of agro waste breadfruit seed husk ash particle (SHA) as reinforcement purpose in LM25 alloy matrix enhanced with the inclusions of 3, 6, and 9wt% of graphite (Gr) nanoparticle (50nm) via squeeze cast liquid processing executed with 200MPa applied compressive pressure. This developed cast examined LM25 alloy and its composites for density, percentage of porosity, microstructure, ultimate tensile strength, abrasion, and corrosion behaviour. From the experimental result, the LM25/10wt% SHA/9wt% Gr hybrid nanocomposite has low density (2.554g/cc), an excellent ultimate tensile strength of (181±1.8 MPa), superior abrasion resistance (0.332mg/m) under 40N load at 0.75m/sec, and good corrosion resistance (0.00382mm/year) compared to cast LM25 alloy. The inclusions of SHA and Gr proved their presence in LM25 alloy has good mechanical strength and maximum abrasion and corrosion resistance to overcome the drawbacks of conventional stir-casted LM25 alloy, which is poor wear resistance.

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KEYWORDS

Breadfruit seed husk ash; characteristics behaviour; LM25 alloy; squeeze cast

1. Introduction

Metal matrix composites were the trend for the current scenario and are used in various engineering applications to overcome the behaviour drawbacks of monolithic alloy [1]. The aluminium alloy-based mono and hybrid composites gained significance in various engineering applications like aerospace [2] and automotive [3] due to low density, good castability behaviour recycling ease, better thermal stability and good elongation percentage [4]. It was synthesised through a solid-state [5] and liquid-state process [6]. Due to important aspects, the Metal Matrix Composite (MMC) was synthesized by the liquid-state process and was familiar with complex shape production with low fabrication cost compared to the solid-state process [7]. The Al-Fe-Si alloy hybrid composites were prepared with goat dung ash and Si₃N₄ liquid stir cast technique, and their particle distribution (microstructure) and mechanical properties were investigated. The output results of this investigation reported that the particle distribution was homogenous and facilitated maximum tensile and impact strength compared to cast Al-Fe-Si alloy [8]. Coconut shell ash-developed aluminium alloy (AA7075) composite performance was enhanced by adding alumina particles via ultrasonic support stir casting process. Its microstructure showed the uniform distribution of reinforcement results in increased tensile strength, damping factor and better corrosion resistance [9]. Waste fly as reinforced Al–10 Mg alloy composite mechanical and thermal behaviour was enhanced by introducing silicon carbide nanoparticles via gravity stir cast and found the enhanced value of tensile, hardness and high thermal stability compared to cast Al– 10 Mg alloy [10]. Recent research has worked towards attaining specific objectives like lightweight, superior tensile strength, good abrasion wear resistance and excellent corrosion resistance with an increase in the contents of reinforcements during the fabrication of aluminium alloy hybrid composite via a liquid-state process.

2. Literature review

2.1. Based on reinforcements

In past decades, the aluminium alloy nanocomposite prepared with inorganic particles like nitride [11], oxide [12], carbide particles [13] and organic waste ash particles [14] found progressive improvement in mechanical, thermal and wear behaviour. Due to this, most of the research investigations focus on synthesising the hybrid nanocomposite with different types of

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reinforcement found to have superior tensile strength and thermal and wear characteristics compared to conventional alloy [15]. The ash-based reinforcement is prominent in aluminium alloy composite prepared with inorganic particle-enhanced mechanical and wear behaviour. Meanwhile, the composite weight was reduced due to ash particles [16]. The waste ash particle-reinforced aluminium hybrid nanocomposite was developed with tungsten oxide and silicon carbide particles via the conventional method. The results showed higher tensile strength, good ductility and high hardness. In addition, the composite cost was reduced by adding waste ash particles [17]. The waste sugar cane bagasse ash utilised as reinforcement in Al-Si10Mg alloy composite synthesised with silicon carbide particle through stir casting technique. The experimental results revealed that the composite contained 9 wt-% sugar cane bagasse ash and 9 wt-% SiC, which offered superior tensile strength, impact and hardness compared to cast Al-Si10Mg alloy [18]. The effect of ilmenite on the corrosion and wear behaviour of aluminium alloy (AA6061) hybrid composite was studied, and it was found that 10% ilmenite has good corrosion and wear resistance [19]. The corrosion behaviour of vanadium carbide and fly ash-reinforced aluminium alloy (Al-Si) hybrid composite was studied, and homogenous particle distribution enhanced the composite's corrosion resistance [20].

2.2. Based on liquid stir processing

Moreover, apart from reinforcement, the selection of liquid-state processing parameters was one of the factors for enhancing the mechanical strength [21], compression strength [22] and microstructural composite properties [23]. The trend for cost-effective melon shell ash-reinforced green metal matrix composites was synthesised via conventional casting. The results improved the composite's hardness and tensile strength on increased melon shell ash trend as 0-12 wt-% [24]. The mechanical and wear behaviour of the aloe vera powder-reinforced aluminium alloy (AA6061) hybrid composite was studied. The impact of aloe vera powder in AA6061/SiC matrix composite offered high hardness and low wear rate with an increased coefficient of friction of 0.242 [25]. In addition, inorganic and organic reinforcements were suitable for aluminium alloy hybrid composite fabrication. They found that the presence of organic waste, like ash-based material, has economic and reduces composite weight [26]. The fly ash and alumina-reinforced LM25 alloy hybrid composites were prepared, and their mechanical and wear behaviour was studied. The results revealed good tensile, compression strength and high hardness values resulted in a low wear rate [27]. Rajan et al. [28] studied the tensile fractography, microstructural and tribological properties of scrap aluminium engine head (LM25) composite developed with alumina catalyst via stir casting route. The results showed good tensile and wear resistance properties. The aluminium alloy composite was synthesised via an optimised stir and squeeze cast technique. The optimised composite specimen found the lowest porosity of 5.29% [29]. In addition, the stir squeeze cast-developed aluminium alloy from scrap waste was found to have good mechanical and microstructural behaviour [30]. The scrap aluminium alloy wheel considered as the source of matrix and developed with 5% alumina, 3 and 6% SiC and 1, 3 and 4% Gr via stir squeeze cast process found maximum tensile and compressive strength on 6% alumina/4% Gr/3% SiC [31].

The various kinds of literature on ash-based reinforcement utilised aluminium alloy hybrid composite were prepared with inorganic ceramic particles via liquid-state processing found microporosity, which resulted in variations in mechanical and wear properties of the composite. In addition, LM25 alloy has the drawback of poor wear resistance. Based on this, the present research novelty is to develop the LM25 alloy hybrid nanocomposites containing 10 wt-% of seed husk ash (SHA) and varied weight percentages of Gr particles through squeeze cast liquid process with an applied pressure of 200 MPa found to reduce the porosity level and obtaining homogenous particle distribution. It increases tensile strength (157-181 MPa), abrasion (0.411-0.321 mg/m) and corrosion resistance (0.0412-0.00382 mm/year).

3. Materials and methods

3.1. Matrix and reinforcements

Advantageous like fluidity (suitable for fairly thin casting), good mechanical properties, low patternmakers' shrinkage (1.3%), good machinability and high corrosion resistance reasons, the LM25 grade alloy was selected as matrix material and had the chemical compositions of 90.15-91.55% Al, 0.2% Cu, 0.5% Fe, 0.1% Pb, 0.2-0.6% Mg, 0.3% Mn, 0.1% Ni, 6.5-7.5% Si, 0.05% Sn, 0.2% Ti and 0.1% Zn [27]. The waste agro breadfruit seeds were collected and washed with water. It was cut into small pieces and heated via an electric oven at 100°C for 1 hour. It helps to remove the moisture content [9]. After the heat-treated seeds were subjected, drying process and milling via a ball milling machine found fine powder containing 35.80% Al₂O₃, 5.06% Cr₂O₃, 30.34% Fe₂O₃, 0.52% K₂O, 1.20% MgO, 0.45% Na2O, 15.45% SiO2, 0.22% MnO and 0.05% ZnO [32]. Ultimately, the acquisition of particles measuring 50 µm occurred through a sieving procedure. In a similar trend, fly ash [10], industrial waste [14] and sugar cane bagasse ash [18] were prepared as earlier research and utilised as reinforcement in an aluminium alloy matrix. The 50 nm Gr particles are received from

		Composition details (wt-%)				
Identification of	Aluminium alloy	Volume of composite	Breadfruit seed husk ash	Graphite		
sample	cm ³	LM25	SHA	Gr		
Cast LM25	150	100	0	0		
S1		87	10	3		
S2		84	10	6		
S3		81	10	9		

Table 1. Composite fabrication details.

the research lab, weighted by 3, 6, and 9 wt-% and used as reinforcement. The squeeze cast type liquidstate stir cast technique was adopted to cast the LM25 alloy and their hybrid nanocomposites tracked by Table 1. It denotes the cast sample identification number, weight and compositions.

3.2. Squeeze cast set-up and its process parameters

The LM25 grade aluminium alloy with melting furnace, along with the squeeze cast liquid stir cast setup, is used to synthesise the above-mentioned composite samples, as shown in Figure 1. It consists of an electrical furnace with a maximum operating temperature of 1200°C, 7 kg capacity steel crucible, electronic control panel, mechanical stirrer operated with servomotor, twin-blade stirrer blade and hydraulic set-up with a maximum capacity of 500 MPa



Figure 1. Actual stir-squeeze cast set-up.

(compressive force). Past research reported that the aluminium-based composites were developed using the liquid-state stir cast process due to economical, efficient and suitable for complex shape production [13]. In this conventional process, casting defects like porosity and internal casting defects were found in the microstructure [15]. To overcome this drawback, the squeeze cast process was recommended [26], and its process parameters are shown in Table 2. Previously, Venkatesan et al. [33] studied the optimisation of the stir and squeeze cast process for tensile properties of aluminium alloy composite and reported that the 800°C melting temperature with 500 rpm stir speed for 5-10 min offered maximum tensile strength. While stirrer action, the temperature of molten metal was reduced to 600°C, which helps to increase the particle distribution [10,11]. In addition, the applied squeeze pressure of 200 MPa showed a good surface with improved bonding strength between the matrix and reinforcements [34]. Based on this reference, the following process parameters are chosen.

3.3. Synthesis of LM25 alloy and its hybrid nanocomposites

Using a portable power hacksaw, the aluminium alloy grade of LM25 was hacked into small ingots and weighed according to Table 1. Based on the tool steel (split type) die dimensions ($30 \text{ cm} \times 5 \text{ cm} \times 1 \text{ cm}$), the total volume for each composite casting was reserved 150 cm³, and its density was varied due to the weight of the matrix and reinforcements. The measured quantity of LM25 alloys was kept in a steel crucible, and its temperature was gradually raised to 800°C through an electronic control panel. After reaching, it was stirred uniformly (500 rpm) for 5 min. It helps to increase wettability and reduce slag formation [8,9]. In the meantime, the externally preheated (300°C for 30 min) SHA and Gr nanoparticles were placed in a reinforcement feeder. It leads to removing moisture content and restricting particle agglomeration during the casting process [13].

After the melting process, the temperature was reduced to 600°C as a semisolid stage, and the quantity of preheated SHA and Gr feed to molten metal was measured. It assists in enhancing the particle distribution during the mixing of reinforcement [15].

Table 2. Squeeze cast process parameters.

Process parameters	Melting temperature	Stirrer temperature	Stirrer speed	Stirrer time	Squeeze pressure	Squeeze time
Values and units	800°C	600°C	500 rpm	5 min	200 MPa	30 sec



Figure 2. Developed composite samples.

Continued, a constant stirrer speed of 500 rpm was applied for mixing the molten matrix and reinforcements for 5 min and then allowed to bottom pouring runway. It is linked to preheated tool steel die (400°C) and helps resist casting shrinkage [26]. Through hydraulic set-up, 200 MPa pressure was applied for 30 seconds over the cast and cooled by natural convection under ambient temperature for 1 hour. Finally, it was removed from the die and cleaned manually. The synthesised cast LM25 alloy and its hybrid nanocomposites are illustrated in Figure 2.

3.4. Characterization of cast LM25 alloy and its hybrid nanocomposites

3.4.1. Density and porosity

Synthesized composites were prepared by $3 \times 1 \times 1$ cm³, and their density was evaluated. The experimental and theoretical density of the composite was evaluated by Archimedes' principle and rule of mixture [11]. From Equation 1, the porosity percentages (*P*%) of developed composites were measured [21].

$$P\% = (1 - \frac{\text{measureddensity}}{\text{theoreticaldensity}}) \times 100$$
 (1)

3.4.2. Microstructure

Influences of the squeeze cast process on the microstructure of developed composites contained constant weight percentages of SHA with varied weight percentages of Gr were analysed via TESCAN make VEGA3 model scanning electron microscope with 3.00 kx magnification. Before, the composites were hacked into $1 \times 1 \times 1$ cm³, and their surfaces were polished using different grit emery papers. The glass polish was made with a rotating polishing machine, and the Keller's reagent was applied for etching [8].

3.4.3. Mechanical behaviour

The tensile samples were machined using wire-cut electrical discharge machining using the standard of ASTM E8 ($100 \times 10 \times 6 \text{ mm}^3$) [11,13,16]. Fuel Instruments & Engineers Pvt. Ltd.(FIE) made a UT4O model configured with a 40-ton capacity universal tensile testing machine was used to evaluate the tensile strength of composites.. With the help of an electronic plotter, the composite's maximum elongation and tensile strength were noted. During the evaluation, three trials from each sample are executed, and the average values of three are considered the mean.

3.4.4. Abrasion resistance

The squeeze cast-synthesised cast LM25 alloy and its hybrid nanocomposites were machined by ASTM G99-05 standard of 35 mm length and 8 mm diameter [11,19] for abrasion resistance study. Ducom pin-ondisc wear tester with hardened steel counter disc with an average applied load of 10, 20, 30 and 40 N under 0.75 m/sec sliding velocity is utilised to wear study behaviour. The three test samples from each composite sample are tested, and the average value is fixed as the mean.

3.4.5. Corrosion behaviour

During the corrosion resistance test, the machined samples ($1 \text{ cm} \times 10 \text{ cm} \times 0.3 \text{ cm}$) are tested by electrochemical impedance spectroscopy test using 3.5% NaCl solution maintained at 20 mV oscillation [19].

4. Result and discussions

4.1. Density and porosity percentage

Figure 3 represents the density (measured and theoretical) and porosity percentages of squeeze cast liquid stir cast synthesised cast LM25 alloy and its hybrid nanocomposites containing 10 wt-% of SHA and 3, 6 and 9 wt-% of Gr nanoparticles.

The primary Y-axis indicates the density, and the secondary Y-axis depicts the porosity percentage. From Figure 3, the measured density of developed composites was lower than the theoretical density. Microporosity was found inside the composite, and its values are illustrated in Figure 3 secondary Y-axis as green. During the casting process, the thermal mismatch was the reason for microporosity [21]. Here, the measured density of cast LM25 allot was noted as 2.655 g/cc, and the hybrid nanocomposite contained



Figure 3. Measured and theoretical density and porosity percentage of composite samples.

10 wt-% SHA with 3, 6 and 9 wt-% in LM25 alloy found the decreased density of 2.576, 2.565 and 2.554 g/cc. It was due to the additions of lightweight SHA in the LM25 matrix. Moreover, the composite density varied due to matrix and reinforcements [26]. A similar trend was reported by previous research [10]. Based on the SHA presence in LM25 alloy, the S3 hybrid nanocomposite was 4% lower in weight than cast LM25 alloy. In addition, the porosity of squeeze cast-developed cast LM25 alloy was 0.93%, and the additions of 10 wt-% SHA with 3 wt-%, 6 wt-% and 9 wt-% Gr nanoparticles showed decreased porosity percentages of 0.81%, 0.77% and 0.70%, respectively. A similar trend was reported by Krishnan et al. [35] during the evaluation of alumina-reinforced scrap aluminium composite. During the casting process, the applied uniform stir



Figure 4. Surface morphology of composite samples: (a) cast LM25 alloy, (b) S1 sample, (c) S2 sample and (d) S3 sample.

speed of 500 rpm performed with the semisolid condition under 200 MPa compressive force was the prime reason for the reduced porosity of the composite. In past research, the aluminium alloy composite developed with a liquid stir casting process without applied compressive force cast increased porosity [10]. However, the developed composites' porosity was maintained at lower than 1%, and reduced porosity results in enhanced composites' mechanical and abrasion behaviour [11,36].

4.2. Microstructure

Based on squeeze cast-developed composite surface morphology with their details of particle distribution, slag formation and its structure formation are shown in Figure 4(a-d). The grey median and small particles showed the identity of SHA and Gr. The darkened surface phase denoted that the LM25 matrix consists of eutectic Al–Si phase grains.

Figure 4(a) represents the surface morphology of cast LM25 alloy with slag-free microstructure. It was due to the uniform stir speed (500 rpm) and the applied compressive pressure of 200 MPa. Moreover, there was no chain reaction found in the coarse grain structure. It reduced porosity, as evidenced in Figure 3. The liquid-state stir cast process selection fixed the composite structure [5]. Here, squeeze cast developed S1 hybrid nanocomposite surface morphology is shown in Figure 4(b). It confirms the SHA and Gr presence in the LM25 alloy matrix. SHA showed the distribution in all the directions of the LM25 alloy matrix, and Gr noted an even distribution. A few slag amounts were recorded due to thermal mismatch during the casting process. Based on the past literature,

even particle distribution was attained by uniform stir speed under the semisolid stage [13].

Figures 4(c) and 4(d) show the homogenous particle distribution with good interfacial bonded structure due to applied uniform stir speed and compressive pressure. The Gr particle speared in the multi directions of the LM25 matrix with SHA particles. The SHA particles create a coarseaggregated structure with a reduced particle gap, which increases the bonding strength [12]. The composite's reduced porosity is evidenced in Figure 3. Maintaining molten metal temperature as semisolid limits the particle damage during the constant stir speed, and obtaining the uniform particle distribution and applying 200 MPa compressive pressure help limit the internal thermal stress. It increases mechanical and wear behaviour [21,26]. Moreover, the four-blade stirrer design found increased particle distribution, which results in increased mechanical properties [37].

4.3. Mechanical behaviour – ultimate tensile strength and elongation

The tensile performance of squeeze cast-synthesised cast LM25 alloy and 10 wt-% with varied (3, 6 and 9) weight percentages of Gr incorporated LM25 alloy hybrid nanocomposite is illustrated in Figure 5, and the ultimate tensile strength of LM25 alloy hybrid nanocomposite was higher than the tensile strength of cast LM25 alloy. It was due to the presence of SHA and Gr particles and limits the failure during the tensile load.

According to Figure 5, the ultimate tensile strength of cast LM25 alloy was 157 ± 3.14 MPa at an elongation rate of 2.67%. While the additions of 10 wt-% SHA with



Figure 5. Ultimate tensile strength and elongation percentage of composite samples.

3 wt-% of Gr in the LM25 alloy matrix showed a 2.72% increased elongation rate, it resulted in a 7% improvement in tensile strength compared to cast LM25 alloy. Effective interfacial bonding between the matrix and reinforcement limits the localised damages against the applied load [11,13]. After this, the content of Gr was increased as 6 wt-% and 9 wt-% in the LM25 matrix showed a significant improvement in ultimate tensile strength. A similar trend was reported in previous literature [36]. The maximum tensile strength of 181 ± 1.8 MPa was recorded in LM25/10 wt-% SHA/9 wt-% Gr alloy hybrid nanocomposite with increased elongation of 2.88%. While the ultimate tensile strength of this composite was compared to cast LM25 alloy, it was improved by 15.28%. While compared to past literature [35], the S3 sample was improved by 44.8%.

It was due to reduced porosity with homogenous particle distribution of composite, as proved in Figures 3 and 4(d). In the past, investigation of aluminium alloy composite developed with fly ash particles via conventional stir cast route found variations in tensile strength [10].

4.4. Abrasion resistance

Here, Figure 6 bar chart represents the abrasion wear resistance behaviour of prepared composites evaluated by Ducom pin on disc wear tester with hardened steel counter disc with an average applied load of 10, 20, 30 and 40N under 0.75 m/sec sliding velocity. Based on Figure 6, the inclusions of SHA/Gr particles in LM25 alloy showed better abrasion resistance and a lower wear rate than cast LM25 alloy. The enhancement of bonding strength was the prime reason for improved abrasion resistance. As mentioned earlier, the aluminium alloy composite contained uniform particle distribution with good bonding that limits particle dislocation and enhances abrasion [11,15].

Abrasion resistance of cast LM25 alloy was 0.385, 0.392, 0.411 and 0.421 mg/m on a normal applied load of 10-40 N with 10 N interval. At the same time, the additions of 10 wt-% SHA and 3, 6 and 9 wt-% of Gr particle in the LM25 alloy matrix showed a decreased abrasion wear rate on increased load. It was due to the presence of ash-based reinforcement leading to withstand the maximum frictional heat and Gr particle resist the scratch against the frictional force. In the previous investigation, the composite's wear rate gradually increased with the increase in load, and the hard inorganic particle offered the maximum resistance against the frictional force [19]. The composite containing 9 wt-% Gr with 10 wt-% SHA recorded the better abrasion resistance (0.332 mg/m) on 40 N normal load with the frictional force of 22.78 N at a maximum sliding speed of 0.75 m/sec. However, effective interfacial bonding with reduced particle spacing in the LM25 alloy matrix limits particle dislocation during high frictional force. Compared to cast LM25 alloy, the S3 hybrid nanocomposite facilitated a 26.8% improvement in abrasion resistance.

4.5. Corrosion behaviour

The influences of SHA and Gr particles on the corrosion behaviour of cast LM25 alloy and 10 wt-% SHA with 3, 6 and 9 wt-% Gr nanoparticle reinforced LM25 alloy hybrid nanocomposite is shown in Figure 7. It showed that the variations in polarisation effect due to the variations in current density, polarisation resistance, potential and resistance to corrosion were measured by electrochemical impedance, and its values are mentioned in Table 3.



Figure 6. Abrasion resistance of composite samples.



Figure 7. Corrosion behaviour of composite samples.

Table 3. Polarisation effect on electrochemical impedance corrosion behaviour of composite samples.

	Current density	Potential	Polarization resistance	Corrosion rate
	lcorr	Ecorr	Rp	-
Sample	A/cm ²	V	$\Omega \text{ cm}^2$	mm/year
Cast LM25	4.265×10^{-6}	-0.645	5.122	0.0412
S1	1.278×10^{-6}	-0.678	3.129	0.0211
S2	2.343×10^{-7}	-0.687	5.678	0.00229
S3	2.612×10^{-7}	-0.691	6.702	0.00382

The potential curve of cast LM alloy and its hybrid nanocomposite showed better corrosion resistance, and the composite contained LM25/10 wt-% SHA/9 wt-% Gr has higher corrosion resistance than others. The S1 and S2 hybrid nanocomposite showed a similar range but was lower than the S3 corrosion resistance value. Due to the presence of SHA leads to withstand the chemical reaction, there was no corrosion failure in the NaCl process. However, the hybrid nanocomposite containing 9 wt-% Gr and 10 wt-% SHA found maximum corrosion resistance, and its life was extended with its minimum corrosion rate of 0.00382 mm/year.

5. Conclusions

The LM25 alloy hybrid nanocomposite is successfully developed with 10 wt-% SHA and 3, 6 and 9 wt-% Gr nanoparticle via squeeze cast liquid processing under 200 MPa compressive force. Among the various compositions, the S3 (LM25/10 wt-% SHA/9 wt-% Gr) hybrid nanocomposite showed fewer porosity percentages of less than 1%, and its weight is saved by 4% compared to cast LM25 alloy. The ultimate tensile strength of the S3 (LM25/10 wt-% SHA/9 wt-% Gr) hybrid composite was improved 15.28% compared to

cast LM25 alloy. The S3 hybrid nanocomposite offered maximum abrasion resistance at 40 N normal load at a high sliding speed of 0.75 m/sec, and its resistance was improved to 26.8% compared to cast LM25 alloy. In addition, the corrosion rate of LM25/10 wt-% SHA/9 wt-% Gr hybrid nanocomposite composite was found with a minimum corrosion rate of 0.00328 mm/year. According to the present investigation results, LM alloy's mechanical, abrasion and corrosion resistance were enhanced by adding 10 wt-% SHA and 9 wt-% Gr and applied for brake disc applications. However, this technology was limited due to costlier than the conventional process and required machining operation for the final product. In future, the developed composite with optimum behaviour of composite S3 sample will be subjected to machining studies.

Disclosure statement

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Data availability statement

All the data required are available within the manuscript.

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