A novel approach for the production and characterisation of aluminium–alumina hybrid metal matrix composites

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Abstract

In this study, aluminium–alumina hybrid metal matrix composites were developed using stir–squeeze casting with ultrasonic stirring. Scrap aluminium alloy wheels (SAAWs) were used as the matrix material, with 1, 2, and 3 wt% of nanosized alumina (\(Al_2O_3_n\)) particles as well as 4, 5.5, and 7 wt% of microsized alumina (\(Al_2O_3_m\)) particles as reinforcement components. An experimental study was conducted using the Taguchi method with an L9 orthogonal array, and the multiobjective optimisation based on ratio analysis technique was used for optimisation. The effects of different ratios of nanosized and microsized alumina particles as well as ultrasonic parameters, namely amplitude and pulse time, on the microstructure and mechanical properties were evaluated and compared. Pin-on-disc wear tests were conducted in the dry condition under uniform load to determine the influence of nanosized and microsized alumina on the wear behaviour. The results revealed that SAAWs reinforced with 1 wt% of nanosized alumina particles and 5.5 wt% of microsized alumina particles and having an amplitude of 100% and pulse on-time of 180 s exhibited lower porosity and metal loss (wear) as well as higher hardness, tensile strength, and compressive strength than other composites.

1. Introduction

In the last decade, the use of aluminium in cars has steadily increased due to its suitable properties, such as high strength-to-weight ratio, ductility, and durability. According to a survey by WardsAuto and DuPont, aluminium is the preferred material of designers for achieving increased fuel economy and decreased emission levels by 2025. The survey emphasised that the use of aluminium parts as compared to steel reduces the fuel consumption by 6.5% and the vehicle mass by 10%. Moreover, the emission of CO\(_2\) is reduced by 8 g/km, with a reduction of 100 kg in the mass of the vehicle [1]. Furthermore, the development of electric vehicles has resulted in a high demand for aluminium. By 2030, the sale of hybrid vehicles is expected to increase by 30% in the global automotive market [2]. The demand for aluminium in the construction of electric and hybrid vehicles is expected to increase by 10 million tonnes by 2030.

Aluminium cannot be used directly in cars, and various methods are used to improve it. One traditional improvement involves adding reinforcements to aluminium, which results in the formation of composites. Hybrid composites comprise two or more different types, sizes, and shapes of reinforcements. Aluminium–alumina hybrid metal matrix composites (AHMMCs) are used in many applications due to their properties such as high tensile, high compressive strength, and lower weight [3]. Consequently, these materials have attracted considerable research attention.

Suresh et al. developed and characterised LM25 aluminium alloy composites reinforced with micro- and nano-\(Al_2O_3\) particles [3]. They found that nanoparticle-enhanced composites have a higher hardness and strength than microparticle-enhanced composites do. Ebrahimnejad et al. evaluated the effect of the addition of...
particulate nanosized alumina on the mechanical properties of Al-Al₂O₃ nanocomposites [4]. Their results indicated that the variation in the reinforcement volume fraction was the primary cause for the change in the fracture mode of the composites. Singh et al prepared Al-6061 reinforced with nano-alumina by using the squeeze casting technique. They found that the composite produced using the ultrasonic-assisted squeeze casting technique has superior mechanical properties to the composite produced using an ultrasonic-assisted stir–squeeze process [5]. Kannan et al examined the mechanical properties and microstructure of aluminium hybrid composites reinforced with nano-alumina. They combined 2 wt% of nano-alumina particles (average particle size: 30–50 nm) and 4 wt% of silicon carbide (average particle size: 5–10 μm) with grade AA7075 aluminium alloy matrix material [6]. The Brinell hardness has been reported to increase for nanocomposites strengthened with single and hybrid reinforcement. Moreover, the tensile strength of single-reinforced nanocomposites obtained through squeeze casting is higher than that of single-reinforced nanocomposites obtained through stir casting [6].

Surendran et al investigated the wear behaviour of LM 25 reinforced with varying proportions of nano-Al₂O₃ (99% LM25 + 1% nano-Al₂O₃, 98.5% LM25 + 1.5% nano-Al₂O₃, 98% LM25 + 2% nano-Al₂O₃, and 97.5% LM 25 + 2.5% nano-Al₂O₃) [7]. The composite with 2.5% nano-Al₂O₃ exhibited optimal wear reduction.

To the best of our knowledge, no attempts have been made to create hybrid metal matrix composites (MMCs) by using scrap Al alloy. Therefore, the mechanical and tribological properties of scrap Al alloy reinforced with nanosized alumina ((Al₂O₃)ₙ) and microsized alumina ((Al₂O₃)ₘ) were investigated in this study.

2. Materials and methods

Scrap aluminium alloy wheels (SAAWs) were selected as the matrix material. A combination of nano-alumina powder (US Research Nanomaterials, Inc., Houston, Texas, USA) with a particle size of 80 nm and purity of 99.5% and microsized alumina powder (Merck Life Sciences Private Limited, Mumbai, India) with a particle size of approximately 45 μm was used as reinforcement. Table 1 presents the chemical composition of the SAAWs (Honda, Japan). The composition of the SAAWs was analysed through an X-ray fluorescence analyser (Horiba XGT 5200, Japan) with a tube voltage of 50 kV and current of 1 mA.

AHMMCs were produced using a bottom tapping stir casting furnace equipped with an ultrasonic stirrer and squeeze casting attachment. To attain superior mechanical properties, the squeeze casting parameters (squeeze pressure of 100 MPa and squeeze time of 45 s) and other parameters were suitably set according to previous studies [8]. The matrix material was melted and maintained at 700 °C in a stainless-steel retort, from which slag was removed. Mg (1 wt%) was added to increase the wettability of the matrix and reinforcements. Nanosized alumina ((Al₂O₃)ₙ) and microsized alumina ((Al₂O₃)ₘ) powders were preheated to 300 °C and then mixed into the melt. After mechanical stirring at 525 rpm for 5 min, an ultrasonic horn with a tip diameter of 25 mm was inserted into the melt. The ultrasonic parameters, such as the pulse and amplitude, were varied at a constant frequency of 20 kHz. The melt was poured into the preheated mould at 250 °C after ultrasonic mixing, and the pouring temperature was maintained at 690 °C. Finally, casting ingots with a diameter of 50 mm and height of 220 mm were obtained by applying a squeeze pressure of 100 MPa. A control sample without any reinforcement was also prepared for comparison.

An experimental study was conducted using the Taguchi method with the L₉ orthogonal array (OA) to save time and resources in achieving an optimised condition for AHMMC production. The levels of the control variables were as follows: (Al₂O₃)ₙ, 4, 5.5, and 7 wt%; (Al₂O₃)ₘ, 1, 2, and 3 wt%; amplitude, 60%, 80%, and 100% of the power drawn; and pulse on-time, 60, 120, and 180 s. Table 2 presents the L₉ OA, where the control sample is represented by E10. The influence of the control variables on the physical and mechanical properties of the produced AHMMCs was analysed and compared.

The multiobjective optimisation based on ratio analysis (MOORA) technique was used to optimise multiple variables because of its simplicity. Brauers and Zavadskas [9] first introduced the MOORA technique, which has been subsequently applied to resolve many types of complex problems. The following steps are adopted for the ranking of the experiments.

## Table 1. Chemical composition of the SAAWs.

| Elements | Cu | Si  | Fe  | Zn | Sn | Pb | Ti | Al |
|----------|----|-----|-----|----|----|----|----|----|
| Mass %   |    |     |     |    |    |    |    |    |
|          | 0.09 | 11.13 | 2.92 | 0.06 | 0.19 | 0.29 | 1.70 | 83.62 |

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**Note:**
- The chemical composition of the SAAWs was analysed through an X-ray fluorescence analyser (Horiba XGT 5200, Japan) with a tube voltage of 50 kV and current of 1 mA.
- AHMMCs were produced using a bottom tapping stir casting furnace equipped with an ultrasonic stirrer and squeeze casting attachment.
- The influence of the control variables on the physical and mechanical properties of the produced AHMMCs was analysed and compared.
- The multiobjective optimisation based on ratio analysis (MOORA) technique was used to optimise multiple variables because of its simplicity.
Step 1: In the decision matrix presented in equation (1), 'p' attributes and 'q' alternatives exist for a decision. The responses in the matrix are as presented in equation (1).

\[
B = \begin{bmatrix}
    b_{11} & b_{12} & b_{13} & \ldots & b_{1p} \\
    b_{21} & b_{22} & b_{23} & \ldots & b_{2p} \\
    b_{31} & b_{32} & b_{33} & \ldots & b_{3p} \\
    \vdots & \vdots & \vdots & \ddots & \vdots \\
    b_{q1} & b_{q2} & b_{q3} & \ldots & b_{qp}
\end{bmatrix}
\]  

(1)

Step 2: The normalised responses of the matrix are obtained using equation (2) as follows:

\[
k_{lm} = \frac{b_{lm}}{\sqrt{\sum_{i=1}^{q} b_{lm}^2}} \quad m = 1, 2, \ldots, p
\]

(2)

where \(k_{lm}\) is a dimensionless number in the interval \([0, 1]\) for the ith alternative and \(m\) is an attribute for the normalised performance.

Step 3: For beneficial attributes, the maximum normalised performance values must be added. Moreover, for nonbeneficial attributes, the minimum normalised values must be subtracted.

\[
d_i = \sum_{m=1}^{m=g-1} k_{lm} - \sum_{m=g+1}^{p} k_{lm}
\]

(3)

where \(g\) is the number of attributes to be maximised, \((p - g)\) is the number of attributes to be minimised, and \(d_i\) is the normalised assessment value.

Step 4: The attribute \(w_m\), which represents the importance of responses, must be multiplied with corresponding weight.

\[
d_i = \sum_{m=1}^{m=g} w_m k_{lm} - \sum_{m=g+1}^{p} w_m k_{lm} \quad (m = 1, 2, \ldots, p)
\]

(4)

where \(w_m\) is the weighted value obtained using the entropy method. The \(d_i\) values are ranked according to the preference values, and the highest value deserves the optimal combination.

Metallographic specimens were prepared through typical processes such as cutting, specimen mounting, polishing, and etching. Specimens of standard size were cut from the produced sample, mounted in Bakelite, ground, and then polished, as per the process described in a previous study [8]. The samples were etched using Kellers reagent and subjected to field-emission scanning electron microscopy (FESEM). The phase composition of the AHMMCs in the optimised condition was analysed with a Bruker X-ray diffraction (XRD) under a scanning ranging of \(10^\circ-90^\circ\), a scanning speed of \(10^\circ/\min\), \(\text{Cu K\alpha}\) radiation \((\lambda = 1.54060 \, \text{Å})\), a tube voltage of 40 kV, and a current of 30 mA. A Bruker-binary V3 advanced software was used for peak identification. The average grain sizes reported in table 2 were estimated using the average grain intercept method described in a previous study [8].

The density of the AHMMCs was measured geometrically by using a polished specimen with a diameter of 13 mm and height of 25 mm. The masses of the AHMMCs were determined using an electronic weighing scale having a resolution of 0.1 mg. The specimen height and diameter were then determined using a Vernier Calliper with a least count of 0.001 mm. Finally, the volume of the specimen was measured. The experimental density of the specimen was estimated by dividing the measured volume by the measured mass [10]. The theoretical density of the composite was determined using the rule of mixture \((0.95 \times \text{density of matrix} + 0.5 \times \text{density of reinforcement})\). The porosity percentage was calculated by dividing the difference between the theoretical and experimental density of the specimen by the experimental density.
A typical subsize specimen was prepared according to the ASTM B557–14 standard for the tensile test, and a short specimen was prepared as per the ASTM E9-09 standard for the compression test. The average ultimate compressive strength (UCS) and ultimate tensile strength (UTS) were determined for four samples. The hardness of the AHMMCs was measured through the metallographic examination of samples as per ASTM E18-17 at room temperature. A ball indenter with a diameter of 1.588 mm (1/16") and load of 100 kg was used for 15 s in the Rockwell hardness tester using the B-scale. Five hardness readings were taken from various points on each specimen to obtain a representative average value.

Wear tests were performed in accordance with ASTM G99 on samples with a diameter of 10 mm and height of 30 mm in the optimised condition. Surface preparation was performed using fine sandpaper and by conducting alcohol washing. Pin-on-disc experiments were conducted using a DUCOM make, TL-20-model-type friction and wear monitor equipped with a data acquisition system. EN31 steel was used as the disc material. It had a hardness of 62 HRC and surface roughness (Ra) of 0.8 μm. A pin-on-disc tribometer was used under the following constant parameters: sliding load, 10 N; sliding speed, 382 rpm; sliding distance, 50 mm; and sliding time, 1000 s. A deadweight load was applied on the pin (specimen) through a pulley–string arrangement.

### 3. Results and discussion

Table 3 presents the results of the L9 OA experiments. The table indicates the rankings for the developed AHMMCs by using the MOORA technique. In table 3, d<sub>i</sub> is the normalised assessment value. As presented in the table, E8 has the highest d<sub>i</sub> value.

#### 3.1. Microstructural analysis

The FESEM image of the E8 composite (rank 1) is displayed in figure 1(a). The composite comprised well-dispersed eutectic silicon. Alumina reinforcement was observed for the E8 composite even though only a few small clusters of reinforcement particles were formed. The small clusters may have been caused due to the small...
size of the reinforcement. The alumina particles were isolated in the interdendrite zones of the eutectic silicon; thus, these particles appear as a mixture in the energy-dispersive X-ray spectroscopy (EDS) images displayed in figures 1(b) and (c). The microstructure of the composite comprised primary alpha-phase aluminium dendrites and eutectic silicon. Because of the applied squeeze pressure, a flake-like microstructure was formed throughout the AHMMC samples. The wettability between the nanoparticles and the matrix improved when breaking up the clustered particles through a strong impulsive impact produced through transient cavitation, which removed the gas layer from the nanoparticle surface [11]. Figures 2(a) and (b) depict a comparison of the optical micrographs obtained for the control sample (E10) and optimised sample (E8). The reinforcement particles were distributed uniformly throughout the matrix for both samples, which is in agreement with the FESEM image displayed in figure 1(a).

3.2. Material characterisation
XRD analysis was performed to identify the phases formed during the casting process. Figure 3 displays the XRD plot for the E8 composite. The highest intensity peak was obtained for the Al matrix (38.38°) and alumina reinforcements (44.63°), which confirmed the distribution of an alumina reinforcement in the Al matrix. The other alloying elements present in the matrix in considerable amounts, namely silicon, magnesium, and iron, exhibited low-intensity peaks. The high-intensity peaks indicate polycrystalline materials.

3.3. Physical and mechanical properties of the composites
Table 3 summarises the mechanical properties of the AHMMCs, including the control sample (E10). The mechanical properties of the composite samples were affected by their physical properties, such as the porosity,
grain size, and distribution of reinforcements. With an increase in the proportion of nanoparticles, agglomeration occurred. Consequently, it became difficult to achieve uniform particle dispersion. The relative density of the composites decreased because of the agglomeration, and the pores acted as crack initiation sites. When agglomeration occurs, an increase in the interparticle distance results in a decrease in the strength as per the Orowan mechanism [12]. Therefore, the concentration (wt%) of alumina nanoparticles should be selected such that the agglomeration tendency is minimal. The reinforcement particle distribution in the matrix and their bonding considerably influence the properties of MMCs as well as the volume fraction of reinforcement. For the E8 composite, a nano-alumina particle concentration of 1 wt% minimised agglomeration, decreased porosity, and increased density.

The E8 composite had the highest hardness (60 HRB), followed by the E9 (52 HRB) and E2 (48 HRB) composites. The high hardness value of the E8 composite was due to the small size of its alumina particles. The small average grain size of the E8 composite also contributed to its high hardness value because the grain boundaries acted as barriers for the propagation of dislocations.

The addition of reinforcement significantly improved the UCS and UTS of the composites compared with those of the control sample (E10), as presented in table 3. Some inconsistencies were observed in the UCS, which may be attributed to the porosity of the composites. In general, a higher concentration (wt%) of reinforcement results in higher porosity, which results in lower composite strength. The E8 composite exhibited the highest UCS. Moreover, the E2 composite exhibited the highest UTS, followed by the E8 composite, which may be attributed to the marginally higher concentration of nano-alumina in the E2 composite than in the E8 composite. During deformation, the dislocation slip distance decreased with an increase in the mass fraction, which resulted in an increase in the composite strength. No significant improvement was achieved in the mechanical properties of nanosized reinforcements for nanoparticle concentrations of over 2 wt% due to inhomogeneous particle distribution and agglomeration [13].

Figure 4 displays a typical tensile stress–strain curve obtained for the E8 composite. The UTS of the composite was approximately 186 MPa. The presence of micro- and nano-Al2O3 particles in the composite led to significant improvement in the tensile strength; however, the strain decreased. The decrease in strain indicated the brittle nature of the composite, which is in agreement with the fracture analysis results. The presence of Al2O3 particles in the composites induced brittleness, which increased the composite material strength. The hard nature of the Al2O3 particles did not allow crack propagation, which led to dislocation in the composite material. This phenomenon is also known as the Orowan mechanism [14]. The increase in the matrix dislocation density may be attributed to the increase in the composite strength. In the casting process, the preheating of Al2O3 particles and ultrasonic-assisted stirring can improve the interface strength and enable uniform dispersion of reinforcements in the aluminium matrix [15]. The preheating of reinforcements results in the production of thermal stress, which may affect the strength of composite materials. The thermal mismatch between reinforcements and the matrix contributes to a decrease in the UTS. When the nanocomposites were subjected to squeeze casting, increased grain refinement and porosity reduction were achieved [6]. In the
squeeze casting process, the squeezing pressure reduced the space between the dendrites, which resulted in the formation of fine grains and a homogeneous microstructure.

Figure 5 displays the FESEM image of the fracture surface of the E8 composite after the tensile test. The image indicates the formation of several cleavage plane fractures and secondary cracks in the composite, which clearly indicate a brittle failure mode. The tensile fracture surface also comprised small dimples due to the refinement of eutectic silicon grains. The interfaces of the matrix and reinforcement did not exhibit a large number of voids and considerable amount of debonding. This result can be attributed to the ultrasonic impulses and squeezing action during the solidification of the composite.

All the compressed E8 composite samples displayed in figure 6(a) exhibited a barrel shape and a clear cleavage line inclined at 40°–45°, which indicated brittle failure. When the UCS was exceeded, the material underwent shearing mostly at an angle of 30°–45°, as indicated by the graphical model in figure 6(b). The friction between the flat top and bottom contacting surfaces of the sample generated a nonuniform plastic flow. The nonuniform plastic flow weakened the material and created a shear deformation in the diagonal plane [8]. Figure 7 displays a typical compressive stress–strain plot for the E8 composite. The reinforcement particles in the composite strengthened it; therefore, the yield stress of the composite increased, as indicated by the ultimate strength point in the curve. The E8 composite exhibited the highest UCS (539 MPa) among all the composites. The E8 composite exhibited the highest UCS because of its low porosity and high density. The compressive strength is significantly influenced by the reinforcement type and particle size [16]. The high strength of composites can be attributed to the high silicon weight percentage in the matrix. Morphological analysis indicates that the mechanical properties of composites can be significantly improved through three methods: (a) alpha-Al grain refinement, (b) uniform distribution of nano-alumina ((Al2O3)n), and (c) refinement of the eutectic silicon phase. Moreover, the mechanical properties are enhanced by increasing the
grain size of the matrix, especially with alumina reinforcement. Additional nucleation sites are created for the formation of the silicon eutectic phase by using a small amount of alumina reinforcement.

3.4. Wear analysis

Wear analysis was performed only for the top three ranked composites (i.e., E8, E9, and E2) by using a pin-on-disc wear machine (table 3). To determine the wear mechanism process, the surface of the E8 composite subjected to wear testing was examined through FESEM, as displayed in figure 8(a). The worn-out surface was relatively smooth and mostly dark with many irregular pits (shown in the magnified image as an inset), which were caused by the removal and sliding of the hard reinforcement particles. The presence of irregular pits is a clear sign of an abrasive type of wear mechanism [17]. When the pin (composite) slid against the hard disc (made of EN31 steel), the abrasive action of the disc removed material microscopically from the pin through the mechanism of microcutting and microploughing [19], as depicted in figure 8(a). Due to the sliding action and subsequent temperature increase, an oxide layer was formed on the pin surface. This layer experienced a shearing force due to the sliding effect and appeared as a displaced material, as illustrated in figures 8(a) and (c). The formed oxide layer contained both the pin and disc materials. The abrasive reinforcement particles eroded the steel surface, which resulted in the transfer of iron and production of a mechanically mixed layer (MML) [18]. The elemental mapping image (figure 8(b)) displays the iron particles formed, and figures 8(c)–(f) displays the EDS spectra of the dislodged alumina reinforcement (spectrum 2) as well as the iron- and oxygen-rich MMLs (spectra 3 and 4, respectively).

Figure 9 displays the variation in the cumulative depth of wear with the interaction time for the E8 composite. Initially, the wear rate was high, which was attributed to the high load between the surface asperities of the sliding surfaces that failed rapidly [19]. The wear progressed gradually after this stage because the wear mode changed from two-body wear to three-body wear due to the wear debris [19]. The change in the wear mode resulted in the formation of the MML, which reduced the wear rate. The MML formed between the pin, and the disc surface played a vital role in the reduction of wear. Due to the sliding action, the formed MML layer was subjected to distortion, spalling, and detachment [18]. This cycle was repeated and was responsible for increases in the wear rate, as displayed in figure 9.

Figure 10 illustrates the relationship between the wear loss and hardness for the top three ranked composites, namely E8, E9, and E2. As the hardness increased, the wear loss decreased. The abrasive wear volume was proportional to the normal load and sliding distance and inversely proportional to the hardness. Therefore, the trends obtained were in line with the observations.

4. Conclusions

In this study, AHMMCs were successfully produced through a novel approach that involved using the stir–squeeze casting process with ultrasonic stirring. This approach is economically viable, and it can be used to produce environment-friendly composite material from scrap Al matrix material. The conclusions of this study are as follows:
1. According to the MOORA optimisation technique, the E8 composite had the best properties among all the composites, followed by the E9 and E2 composites.

2. Optical microscopy images revealed that among all the composites produced, the E8 composite exhibited the most uniform dispersion of alumina particles in the microstructure and had the lowest porosity.

3. XRD examination confirmed that no significant phases apart from eutectic silicon-alumina formed in the AHMMCs regardless of the reinforcement used.

4. The E8 composite had the highest hardness (60 HRB), highest UCS (539 MPa), and second-highest UTS (186 MPa).

Figure 8. (a) FESEM image of the worn-out surface exhibiting the wear mechanism, (b) EDS layered image illustrating the wear debris, (c) FESEM image displaying the wear surface at high magnification as well as the points at which EDS spectra were recorded, and (d)–(f) EDS analysis of spectra 2–4, respectively.
5. The fracture surfaces of the composites obtained after tensile testing indicated that failures predominantly occurred under a brittle mode of fracture. The fracture surfaces did not display any evidence of plastic deformation. However, a limited ductile failure mode was observed.

6. The E8 composite had a lower wear loss than the E9 and E2 composites after the wear test. The aforementioned composites exhibited a wear morphology of irregular pits and grooves on the worn-out surface, which indicated an abrasive type of wear mode.

7. An MML formed between the sliding surfaces and helped in reducing the wear after the initial steep rise in the wear rate.

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