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The Potentiality of LHA Nanoparticle Reinforced AA2024 Composites: A Focus on Microstructure, Mechanical Properties

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ABSTRACT

The study centered on producing and analyzing composites comprising aluminum 2024 alloy and Lanthanum Hexaluminate (LHA) nanoparticles. The study aimed at aerospace and automobile applications including thermal barrier coatings and high temperature applications. LHA particles were synthesized through chemical precipitation and filtration method and underwent characterization via X-ray analysis and FESEM/EDS analysis. Subsequently, the potential of Lanthanum Hexaluminate powders (LHA) as reinforcement material for integration into AA-2024 alloy was explored. The stir casting method was utilized to fabricate composites of Aluminum 2024 alloy with LHA. The Vickers hardness and mechanical behaviour of the proposed composites is investigated as per the ASTM standards and the effects of varying the weight fraction of reinforcement on particle distribution, particle-matrix interfacial interactions, and both physical and mechanical properties were evaluated. SEM and EDS examinations confirmed the presence of components and an even dispersal of particles within the composite. Mechanical properties such as hardness and tensile strength were evaluated, revealing a substantial enhancement of 22.31% in microhardness and 14.38% in tensile strength for the composites.

1. INTRODUCTION

Energy efficiency now ranks as a primary focus in both national and international policies. Historical overconsumption of energy has driven a notable increase in CO_2 levels, leading to substantial climate alterations [1]. Additionally, escalating fuel costs have heightened concerns within the aviation industry regarding energy consumption. Consequently, aircraft manufacturers are compelled to tackle both governmental ecological objectives and economic pressures by developing more fuel-efficient aircraft. In this dynamic economic landscape, aircraft manufacturers persistently seek avenues to diminish aircraft fuel consumption. The most effective approach involves integrating lighter materials, which directly reduces aircraft weight and subsequently curtails fuel usage [2]. The ongoing aging of both civil and military aircraft generates a need for these aircraft to operate beyond their initially intended lifespans. This presents various challenges, one of which is ensuring that aircraft materials can maintain their damage tolerance over extended periods [3]. Among the available options, aluminum stands out as a superior choice due to its familiar mechanical properties, ease of design, manufacturability, and established inspection methodologies [4, 5]. Throughout the history of the aircraft industry, aluminum alloys have consistently stood out as the preferred material. This is due to their adaptable nature, boasting exceptional strength-to-weight ratios, impressive resistance to corrosion, efficient thermal conductivity, and cost-effectiveness.

Among all, 2000 series alloys are renowned for their exceptional damage tolerance and resistance to fatigue crack propagation. The 2024 aluminum alloy, particularly in its T3 aged condition stands out for its remarkable damage tolerance and strong resistance to fatigue crack propagation, making it a crucial material for aircraft structures [6]. Nevertheless, its application is restricted to highly stressed areas due to its lower yield stress level and relatively modest fracture toughness [7]. To overcome these limitations researchers disseminated hard ceramic particles into the 2024 alloy to enhance the tribo-mechanical properties. Researchers reported that the integration of reinforcement into the matrix showed improvement in the mechanical properties of these composites compared to monolithic alloys [8, 9]. The various reinforcements used are Silicon Carbide, Aluminium Oxide, Titanium carbide, Boron Carbide, etc. Al_2O_3 reinforcement has good compressive strength and wear resistance [10]. The interfacial reaction between the materials is also important because if the load carrying transferred to the interface, it will affect the mechanical properties of the composite material. So, the properties of the material also depend on the other characteristics like micro structure, shape, processing time of technique and the way of reinforcement etc.

Out of all Particle-reinforced aluminum matrix composites (AMCs) are emerging as highly promising materials for aerospace and automotive applications, owing to their exceptional combination of high specific strength. However, the addition of ceramic particles tends to decrease the ductility of AMCs, thus constraining their potential for certain applications. Rare earth elements (REEs) exhibit unique properties and are utilized in diverse fields like electronics and aerospace. Lanthanum hexa-aluminate ($LaAl_{11}O_{18}$), abbreviated as LHA, is a key compound in the rare-earth aluminate family, used in thermal barrier coatings (TBCs) [11, 12]. Despite extensive research on ceramic materials like SiC [13, 14], B_4C [15], and Al_2O_3 [16, 17], LHA's potential as reinforcement in AA2024 remains unexplored. So the novelty of this study aims to take a step forward in investigation of mechanical properties for AA2024/LHA composites across various weight fractions. Section 2 outlines the preparation of LHA nanoparticles and associated procedures, including XRD and FESEM experiments, as well as composite preparation. Section 3 presents the findings and discussions on the microstructure and physical/mechanical behavior of AA2024/LHA composites. Conclusions are drawn based on the comprehensive analysis provided.

2. MATERIALS AND METHODS

2.1 Preparation of powders and their characterization

2.1.1 Preparation of powders

In the present work, alumina powder, lanthanum oxide, aluminium nitrate, ammonium carbonate and citric acid are used. LHA powders were produced via chemical precipitation and filtration method and prepared powders are showed in Figure 1. Eqs. (1) and (2) were employed in the preparation process to synthesize LHA nanoparticles through stoichiometric reactions.

Figure 1. Prepared LHA powders

$$La_2O_3 + 6\ HNO_3 + 9\ H_2O \rightarrow 2\ [La(NO_3)_3 \cdot 6\ H_2O] \quad (1)$$

$$2\ [La(NO_3)_3 \cdot 6\ H_2O] + 22\ [Al(NO_3)_3 \cdot 9\ H_2O] \rightarrow 2\ LaAl_{11}O_{18} + 72\ HNO_3 + 174\ H_2O \quad (2)$$

Initially, 7.93 grams of Al_2O_3 were combined with distilled water in a beaker. A stable solution was achieved by adding 0.05 wt.% of citric acid, known to effectively disperse alumina solutions. Ultrasonication for 15 minutes prevented agglomeration formation. In a separate beaker, 0.47 grams of lanthanum oxide powder were mixed with 0.56 grams of nitric acid in deionized water, blended for 20 minutes, followed by heating which lead to form a component named as lanthanum nitrate. Beaker 2 contained 11.809 grams of weighed aluminum nitrate, which was mixed with distilled water. Both solutions were titrated, heated for 15 minutes, and ultrasonicated for an additional 15 minutes. Precipitation was initiated by the dropwise addition of 0.2 M $(NH_4)_2CO_3$ solution to the combined solutions. Filtration was performed overnight and the resulting powder Al_2O_3-x $LaAl_{11}O_{18}$ was collected.

2.1.2 XRD analysis of powders

The XRD data for the prepared powders was obtained using the MINIFLEX 600 machine, with a step size of 0.02° in a specified scanning range i.e. 3 to 90 degrees. Increasing distinctness in phases between Lanthanum and Alumina suggests effective blending of the powders. Analysis of Eq. (2) revealed the synthesis resulting in Lanthanum Aluminate ($AlLaO_3$) and Lanthanum hexa-aluminate ($Al_{11.5}La_{0.85}O_{18.5}$), as confirmed by XRD peak analysis (Figure 2) are matched with the literature [18, 19]. The XRD pattern shows broadened peaks, indicating small-sized crystals, with strong diffraction peaks suggesting high crystallinity of the LHA particles.

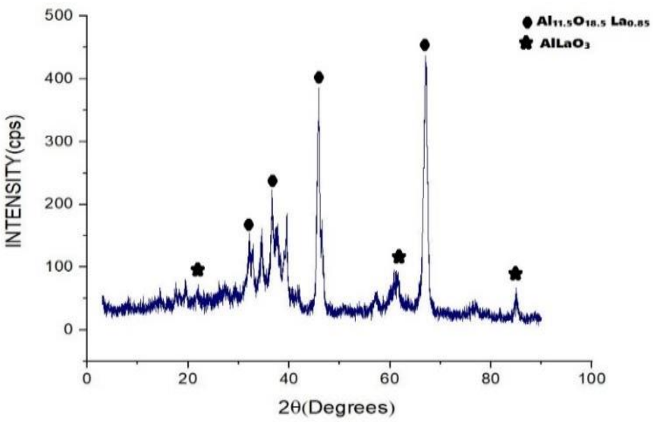

Figure 2. XRD analysis of prepared LHA powders

2.1.3 SEM analysis of powders

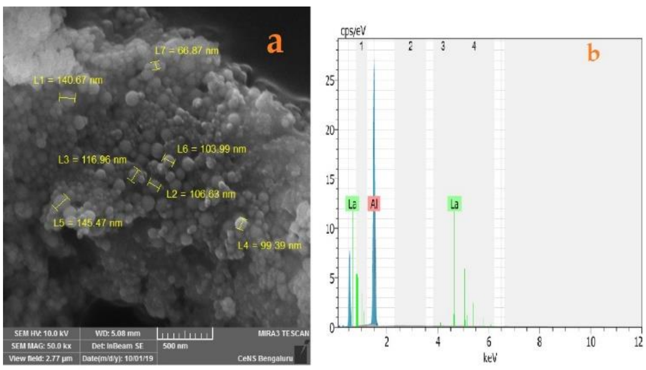

Figure 3. a) SEM of LHA powder b) EDX of LHA powders

In Figure 3a, it is evident that the method employed has effectively yielded nanoparticles with remarkable uniformity in both size and morphology. Through FESEM analysis, it was determined that LHA nanoparticles exhibit sizes ranging from 65 to 150 nm, with an average size of 111 nm. Meanwhile, Figure 3b shows the results of EDS analysis conducted on the manufactured powder. The analysis indicates that the predominant elements present in the sample are aluminium and lanthanum, affirming that these materials constitute the primary constituents of the synthesis process.

2.2 Fabrication of composites

For this study, AA 2024 is selected as the matrix material. This alloy stands as one of the most commonly employed aluminium-copper alloys in aircraft industry applications, utilized extensively in forging and rivets. The selection of this matrix alloy was driven by its exceptional blend of damage tolerance and strength at room temperature and under elevated conditions. The chemical composition of AA2024 aluminium alloy is shown in Table 1.

Table 1. AA2024 aluminum alloy composition (wt.%)

Element	Mg	Zn	Si	Mn	Cu	Ti	Fe	Al
Weight%	1.50	0.14	0.1	0.47	4.40	0.04	0.11	Bal

Ram et al. [20] and Srividya et al. [21] in their papers reported the optimized powder conditions for LHA preparation and best Conditions were considered and experimented. The AA2024 alloy was combined with the appropriate weight percentage of LHA powders (average size 111 nm) enclosed in aluminum foil and heated to 750℃ in a graphite crucible. Additionally, 1 wt.% of Mg was introduced to enhance the wettability of reinforcements within the matrix [22]. Following this, the mixture underwent agitation for 5 minutes at 350 rpm using a three-blade stirrer before being poured into a mild steel mold measuring Ø15 mm × 150 mm in height. Fabrication is carried out under argon gas atmosphere to avoid oxidation. The current investigation centers on augmenting the strength of AA2024 alloy by incorporating LHA ceramic powder, with concentrations varying from 3% to 9%wt LHA, at 3%wt increments. Figure 4 depicts the stir casting set up used for fabrication of composites. In further sections pure AA2024, AA2024 +3 % LHA, AA2024 + 6 % LHA, AA2024 + 9 % LHA are designated with terms as letters AI, AII, AIII and AIV.

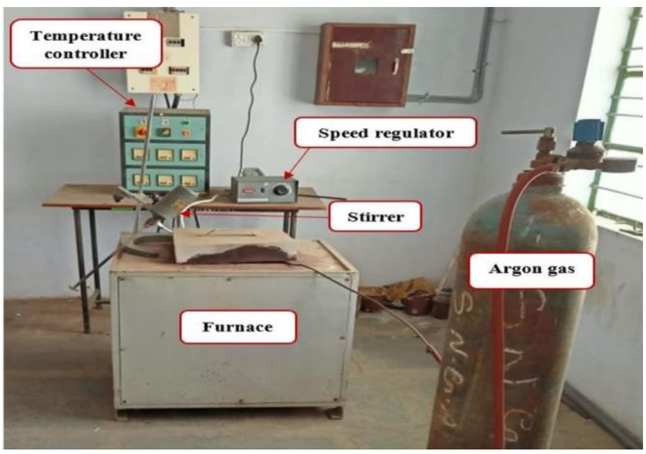

Figure 4. Stir casting setup

2.3 Porosity measurement

Eq. (3) was applied to determine porosity based on analytical and investigational densities [23], with the theoretical density of LHA particles recorded at 4.01 g/cc [24]. Using deionized water for immersion, composite densities were evaluated using the Archimedes principle, following surface finishing and weighing with an electronic balance. Six density measurements were obtained for each sample, and their average was computed.

$$\%porosity = 1 - \left(\frac{measure\ density}{theoretical\ density}\right) \times 100\% \quad (3)$$

2.4 Mechanical behaviour of composites

The microhardness and tensile characteristics of composites containing varied weight percentages of reinforcements were analyzed to gain insights into their mechanical performance. The impact of reinforcement on hardness was assessed through Vickers hardness tests using a 100-gf load and a 15-second loading time. For each sample eight readings were taken and the average value was recorded as the hardness. Tensile tests were conducted according to ASTM_B557 standards on a UTN 40 machine, with three readings recorded for each composition.

3. RESULTS AND DISCUSSION

3.1 Density and porosity

The discrepancy between analytical and investigational densities allowed for the estimation of porosity using Eq. (3), as shown in Table 3. The low porosity indicates a successful casting process. It's noted that an increase in the weight percentage of reinforcements correlates with increased porosity. Kumar et al. [25] found the similar tendency for 2024 Aluminium - High Entropy Alloy Composites. Table 3 reveals that a higher percentage of reinforcement resulted in increased porosity, likely due to the presence of clusters. Factors like irregular stirring and pouring into molds might have contributed to gas entrapment, resulting in high porosity values at increased percentages of reinforcements.

Table 3. Porosity calculations of AA2024 alloy and prepared composite

Sample	Theoretical Density ($g\ cm^{-3}$)	Experimental Density ($g\ cm^{-3}$)	% Porosity
AI	2.78	2.77	0.359
AII	2.818	2.821	1.063
AIII	2.856	2.868	0.418
AIV	2.929	3.01	2.691

3.2 Microscopy results

EDM technique was employed to cut 2 cm ×2 cm ×0.2 cm specimens from AA2024/LHA composites for microstructural analysis. Prior to examination, the samples underwent thorough polishing to avert scratches and were etched with Keller reagent for 4 seconds. Microstructure analysis was conducted using SEM coupled with EDX to illustrate the spreading of lanthanum hexa-aluminate particles within the AA2024 matrix. Figure 5 displays the micrograph of pure

AA2024 alloy, while Figure 6 illustrates the uniform distribution of LHA in the AA2024 matrix at 6 wt.% LHA. Subsequent reinforcement additions led to agglomeration, as observed in Figure 7.

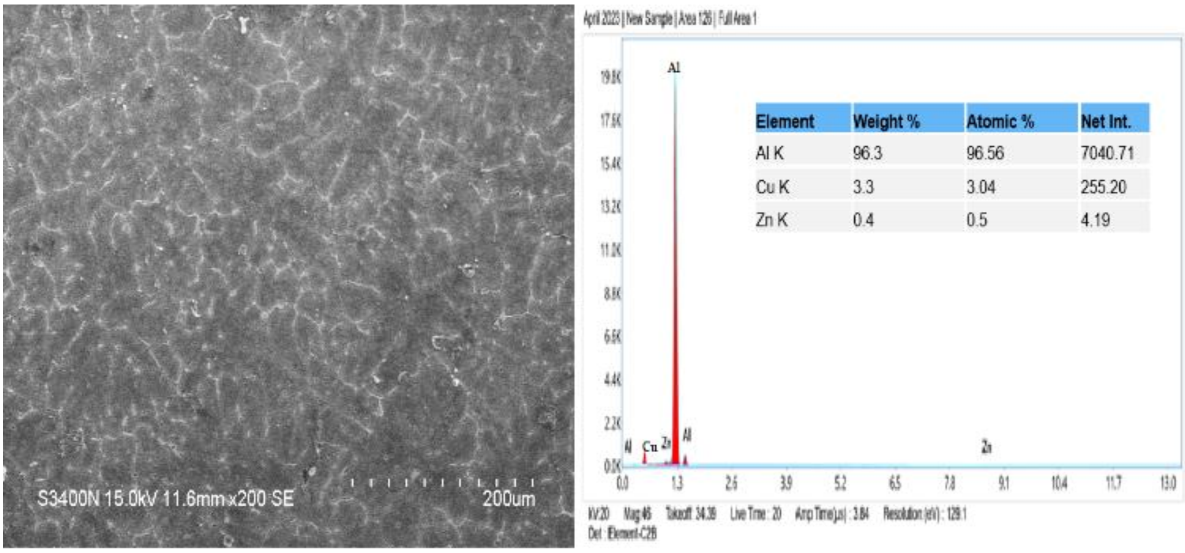

Figure 5. SEM/EDX analysis of AI

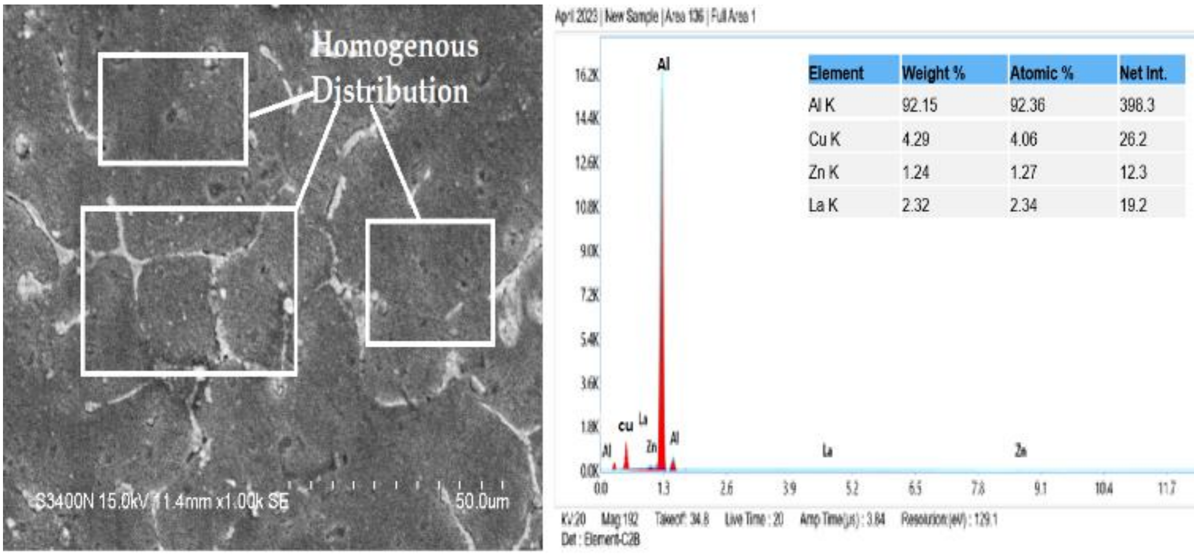

Figure 6. SEM/EDX analysis of AIII

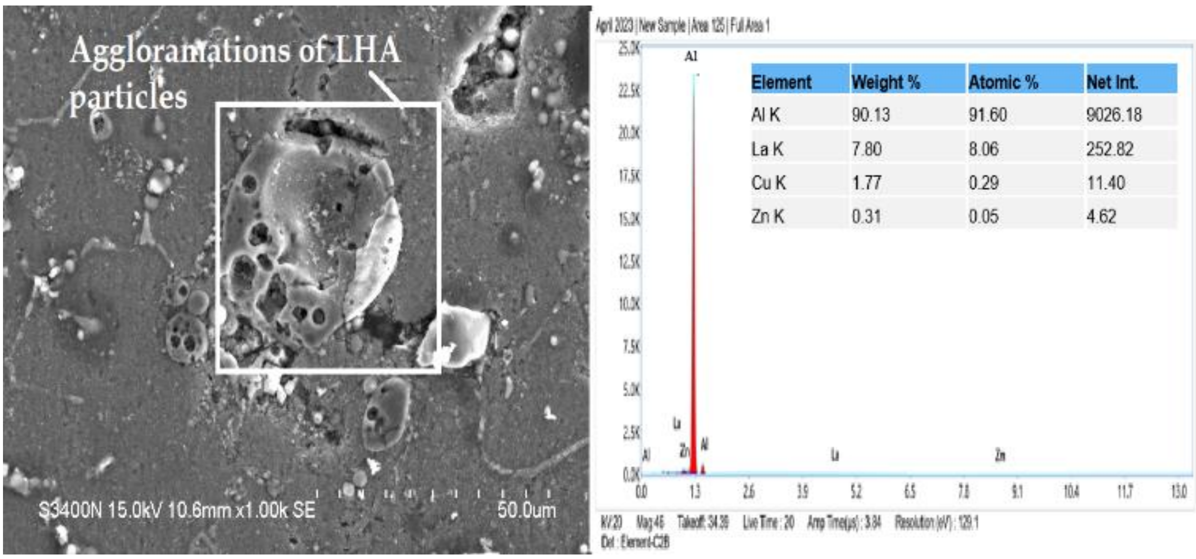

Figure 7. SEM/EDX analysis of AIV

3.3 Mechanical properties

Alloys of aluminum are constrained by their relatively low strength and stiffness, which limits their utility across various domains. However, the incorporation of aluminum alloys with composites boasting enhanced mechanical properties offers a solution to these limitations. This section aims to investigate and present findings on the hardness and tensile properties of AA2024 and its processed composites. This examination encompasses parameters such as ductility, elastic modulus, ultimate tensile strength and yield strength which collectively provide insight into the mechanical performance of these materials.

3.3.1 Hardness

Table 4 presents microhardness data, while Figure 8 illustrates the hardness variation across different reinforcement percentages. It's observed that as the reinforcement percentage increases, microhardness levels also exhibit a corresponding rise compared to the base alloy. The AI alloy has a hardness value of 72.34±3 VHN. For AII sample the lowest hardness is obtained among the composites (87.34±3VHN), whereas sample AIII has the highest (93.12±4 VHN). It is observed that in comparison to AI sample composites have a maximum rise of 22.31 % in hardness.

Table 4. Hardness results

Sample	Micro Hardness (HV)
AI	72.34
AII	87.34
AIII	93.12
AIV	80.17

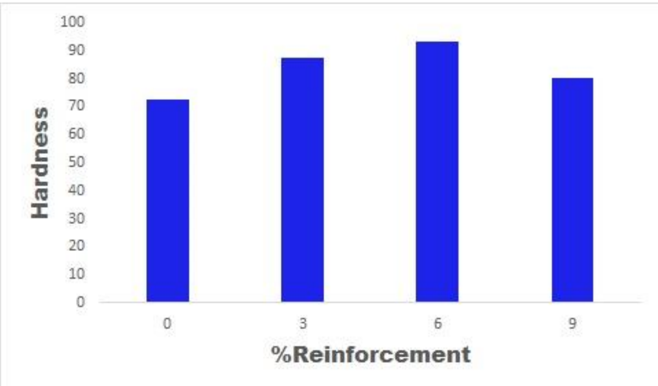

Figure 8. Variation of hardness with reinforcement

The hardness rise is due to the existence of rigid LHA particles, enhancing the material's load-bearing capacity while restricting matrix deformation by hindering the movement. However, due to the inadequate dispersion of LHA nanoparticles, the reinforcement effect diminishes, resulting in a decline in hardness beyond a 6% weight fraction. It is noteworthy that the observed hardness trend in this study aligns with findings reported by Reddy et al. [26].

3.3.2 Yield strength

With the increase in volume fraction of LHA particles composite yield strength is also increased. The interfacial bond between the reinforcement and matrix, influenced by the size and shape of the particles, contributes significantly to the composite's strength. When the bonding between the matrix and reinforcement is sufficiently robust, the load can transfer from the aluminum alloy to the hard LHA particles. This enhanced strength of the LHA particles serves to fortify the comparatively weaker aluminum alloy matrix. The base material exhibits a yield strength (0.2% offset) of 192 MPa, while the maximum logged yield strength for the 6 wt.% fraction reaches 246 MPa. It is evident that as the volume percentage increases, more loads can be borne by the reinforcement, ensuing in improved yield strength.

3.3.3 Tensile strength

Composite materials are engineered to achieve exceptional strength, crucial for withstanding tensile loads and preventing localized failure caused by internal stresses exceeding material strength. Reinforcing particles play a pivotal role in evenly distributing stress, delaying localized damage. Increasing the volume percentage of LHA shifts stress distribution towards the hard phase, boosting tensile strength. Figure 9 displays variations in YTS for both the alloy and composite. As-cast AA2024 exhibits tensile strengths of 256 MPa, 290 MPa, and 270 MPa for 3%, 6%, and 9% reinforcement, respectively.

Comparing with cast AA2024 alloy, it's evident that composites exhibit higher tensile strength. However, increased LHA content in the composites may lead to defects around the particles, potentially causing delamination of the interface and a subsequent decrease in ultimate tensile strength. When the reinforcement percentage reaches a certain level, the reaction between AA2024 and LHA ceramic powders results in the formation of hard layers. In previous works, they reported an improvement of 17.43% tensile strength on AZ91E/LHA composite materials. The primary challenge with magnesium lies in preparing composites because of its high reactivity. However, the AA2024 alloy, which contains copper, becomes suitable for high-temperature applications once it is reinforced with LHA.

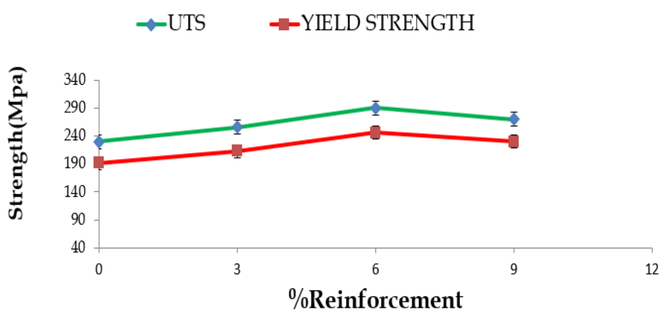

Figure 9. Variation of yield and tensile strength with reinforcement

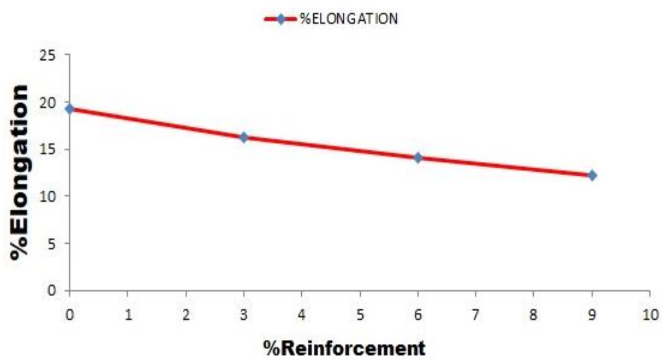

Figure 10. Variation of elongation with reinforcement

3.3.4 Ductility

Figure 10 demonstrates a decrease in elongation percentage from 19.3 to 12.2. The increase in reinforcement percentage may contribute to reduced ductility, likely due to void nucleation. Elevated stress concentration at cracked particle tips could further diminish composite ductility. Additionally, LHA powder which is a derivative from alumina may act as stress concentrators, promoting particle agglomeration. This agglomeration significantly elevates stress levels, potentially causing detachment between matrix and reinforcement materials.

3.3.5 Modulus of elasticity

The stiffness of a composite, expressed by the modulus of elasticity, reflects its rigidity. Incorporating LHA particles into the AA2024 alloy boosts its modulus as expected. These resilient particles, dispersed within the matrix, impede dislocation motion, strengthening the composite. Various factors, such as reinforcement percentage, matrix distribution, and particle characteristics, influence Young's modulus. The determination of modulus employs both the Halpin–Tsai equation and the rule of mixtures, with the former suited for discontinuous reinforcements and the latter for continuous ones.

Using Halpin–Tsai equation:

$$E_c = \frac{E_m(1 + 2S * q * V_r)}{(1 - q * V_r)} \tag{4}$$

where,

$$q = \left[\frac{\frac{E_p}{E_m - 1}}{\frac{E_p}{E_m}}\right] + 2S.$$

Using ROM equation:

$$E_c = E_m V_m + E_r V_r \tag{5}$$

The rule of mixtures is straightforward, accounting solely for volume fraction and elastic modulus, whereas the Halpin–Tsai method also incorporates the aspect ratio of the reinforcement particle. Both methods were employed to calculate the modulus of elasticity for all reinforcements. Figure 11 presents the experimental and calculated data for the modulus of elasticity concerning the proposed reinforcements. Table 5 depicts the Comparison table of Elastic modulus for samples using H-Tsai, ROM and experimental values.

Figure 11. Illustration of modulus of elasticity using experimental, ROM and Halpin–Tsai model

Table 5. Comparison table of Elastic modulus

% Reinforcement	H-Tsai Model	Rule of Mixtures	Experimental Model
0	77.56	78.14	76.3
3	81.8	86	83.12
6	86	91.33	88.2
9	92.1	97.92	95

Discrepancies between practical and calculated values are ascribed to uncertainties in determining the appropriate modulus value for the particle reinforcement. Using rule of mixtures AII, AIII and AIV values are 86 GPa, 91.33 GPa, 97.92 GPa whereas for H-Tsai equation it is 81.8Gpa, 86 GPa, 94.1 GPa respectively. It is noteworthy that experimental value error is in acceptable range in comparison to ROM, H-TSAI equation.

4. CONCLUSIONS

From experimental results, the following conclusions are drawn:

- Lanthanum hexa aluminate powder was successfully prepared by using chemical precipitation and filtration technique
- The micro structural studies revealed the uniform particle size of the powders and the average size of the particles were found to be 111 nm.
- XRD analysis we confirmed that our synthesis procedure for preparation of powders are accurate by identifying different compounds related to lanthanum Hexa-aluminate.
- Stir-casting technique was used to fabricate AA 2024 composites with LHA ceramic powder additions rising from 3 to 9 wt.% at 3wt.% interval.
- Through meticulous stirring, a uniform dispersion of LHA nanoparticles within the base material was achieved at 6 % LHA whereas thereafter addition of further reinforcement caused aggloromations.
- Mechanical behaviour (hardness, yield and tensile strengths) of the composites was found to increase with increased nano LHA up to 6%. However, beyond this threshold value further addition of nanoparticles led to a decline in mechanical performance because of aggloromations.
- Based on the findings, the composites reinforced with 6 wt.% LHA exhibited superior mechanical properties compared to other reinforcements due to their uniform distribution within the matrix. Notably, there was a significant increase of 22.31% in microhardness and 14.38% in tensile strength.
- LHA is an emerging material with significant potential in high-temperature applications and thermal barrier coatings. The preparation of LHA-based composites is expected to be of considerable industrial importance. This work can also be extended to investigate the tribological properties using statistical methodologies like design of experiments.

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