

## MECHANICAL PROPERTIES OF ALUMINIUM METAL MATRIX COMPOSITES REINFORCED WITH MILLSCALE AND ALUMINA

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### ABSTRACT

The present investigation examines the mechanical properties of aluminium metal matrix composites (AMCs) that are reinforced with millscale and alumina. The composite materials were produced through a stir casting technique and analysed for density, porosity, hardness, tensile strength, yield strength, specific strength, percentage elongation and fracture toughness. The microstructure of the produced composites and the fractured surfaces of the composites were examined using a scanning electron microscope (SEM). The findings demonstrate that the addition of millscale and alumina improves the mechanical properties of the composites. The mechanical testing reveals a gradual increase in tensile strength with a higher weight ratio of alumina, resulting in a maximum ultimate tensile strength of 124.73 MPa for sample D. Moreover, hardness increases slightly with higher weight ratios of alumina, with sample D exhibiting the highest hardness value of 94.6 BHN. Fracture toughness varies with composition, and sample D displays a fracture toughness of 7.6 MPa m<sup>0.5</sup>. The incorporation of alumina enhances hardness, yield strength, and tensile strength. Whereas the presence of millscale leads to improved hardness and tensile strength. The incorporation of millscale and alumina markedly improves the specific strength of the composites. The analysis of the microstructure demonstrates the distribution and bonding properties of the reinforcement particles in the aluminium matrix. The findings indicate the potential of millscale and alumina as reinforcing materials for AMCs, providing a sustainable and cost-effective solution for diverse engineering applications.

**Keywords:** aluminium matrix composite, millscale, alumina, mechanical properties, composite materials.

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### INTRODUCTION

Researchers are becoming more interested in Aluminium Matrix Composites (AMCs), a group of metal matrix composites, because of its exceptional mix of characteristics. Owing to these materials' advantageous mechanical, physical and thermal characteristics, they are being used more in both established and cutting-edge technical applications, including those in the aerospace, defence, automotive

and marine industries [1, 2]. AMCs are desirable for a wide range of technical applications due to their many advantages, including high specific strength, low density, high specific stiffness, great wear resistance and enhanced elastic modulus [3]. However, these qualities can be enhanced by selecting reinforcing materials and manufacturing settings carefully, enabling AMCs to meet demanding performance standards across a variety of sectors. Many industrial applications depend on the development of high-performance AMCs, which has

spurred extensive study into the application of ceramic reinforcements to improve their mechanical and physical properties [4]. Ceramic-based reinforcements like silicon carbide (SiC) and alumina ( $Al_2O_3$ ) can enhance the unique mechanical capabilities of aluminium matrices. These ceramic-based reinforcements were major components of the first wave of AMC research [5]. These materials offer superior reinforcing activities by improving the overall strength and hardness of the matrix metal. However, one of the main disadvantages of using typical ceramic reinforcements is their relatively high cost, particularly in developing countries where the cost of producing AMCs might become prohibitively high due to the importation of such materials [6, 7]. For this, researchers in underdeveloped nations are starting to investigate substitute reinforcing materials made from industrial and agricultural wastes to reduce cost without sacrificing the advantageous qualities of AMCs [8]. These reinforcements solve sustainability and waste disposal difficulties while also being more cost-effective and environmentally beneficial. Research has shown that materials such as periwinkle shell, rice husk ash, palm kernel shell ash, coconut shell, and so on, can effectively improve the mechanical properties of aluminium composites [9 - 11]. These derivatives of industrial and agricultural by-products are widely available in underdeveloped nations and provide an affordable substitute for synthetic reinforcements, hence aiding in waste management and environmental conservation initiatives [12]. Through various research, it has been established that the derivatives of industrial and agricultural by-products can be used as monolithic or hybrid reinforcements in a metal matrix [13, 14]. For instance, palm kernel shell ash (PKSA) was used as hybrid reinforcement with SiC in Al6063 to produce AMC at a weight percentage of 2 - 10 wt.%. It was observed that the mechanical properties of the developed AMCs improved with the addition of the reinforcements [5, 15]. Meanwhile, Edoziuno et al. developed AMCs using monolithic reinforcement of palm kernel shell (PKS) [16]. The physicochemical properties of the produced metal matrix composites were determined. Adeleke et al. also employed a monolithic reinforcement of sawdust ash (an agro-waste derivative), the findings in the study revealed improved investigated properties of the AMCs produced [17].

Nonetheless, there has not been much research done

on using millscale, an industrial by-product of hot rolling steel, as a reinforcement material for AMCs. Millscale having a flaky layer is primarily composed of iron oxides that is formed up during steel production process. After the production of steel, the generated mill scales are usually thrown away as waste, thereby, resulting into environmental issues. Despite this, because of its refractory character and the presence of advantageous oxides that may improve the mechanical properties of AMCs, millscale offers a great chance as a reinforcement material [18]. According to Bugdayci et al. millscale has a high concentration of iron oxides, which can be used as reinforcement to improve the wear resistance, hardness and strength of composite materials [19]. Despite these qualities, not much research has been done on its possible application in AMCs, especially when combined with more conventional reinforcements like alumina.

Known for its exceptional mechanical qualities, including high hardness, high wear resistance, and a high melting point, alumina ( $Al_2O_3$ ) is a popular ceramic reinforcing material. Its ability to reinforce AMCs has a strong body of research behind it, particularly when great strength and stiffness are needed [20]. Its relatively high density ( $3.9 \text{ g cm}^{-3}$ ) and expense, however, provide difficulties, especially for sectors looking to create lightweight composites [21]. Combining alumina with a lighter, less expensive reinforcement like mill scale, which has its own advantageous qualities, offers an interesting prospect for the development of hybrid aluminium composites. These hybrid composites may provide a cost-effective combination of superior density, strength, and exceptional resistance.

The scientific community has recently become more interested in the creation of hybrid composites made of aluminium that combine conventional and non-traditional reinforcements [22]. Cost and sustainability concerns can be addressed while simultaneously optimizing the mechanical properties of AMCs using reinforcements such as alumina and mill scale. This approach provides developing countries' industries with an affordable, high-performing solution while also reducing the environmental impact that industrial wastes like mill scale cause [23]. However, using locally accessible materials promotes the utilization of native resources and lessens reliance on pricey imported reinforcements, which is consistent with the objectives of sustainable development and industrial innovation.

In response to the increasing need for composite materials that are lightweight, affordable and high performing, this study focused on the development and assessment of aluminium metal matrix composites (AMCs) reinforced with alumina and millscale. The main goal is to evaluate how these reinforcements affect the produced composites' characteristics, such as density, porosity, hardness, tensile strength, yield strength, specific strength, percentage elongation and fracture toughness. To produce a hybrid composite with an ideal balance of attributes, the study specifically aims to investigate the potential of mill scale, which has been mainly underutilized as a reinforcing particulate and its combination with alumina as reinforcements

This study is a component of continuing investigations into novel, environmentally friendly materials that can improve the efficiency and affordability of AMCs for a range of engineering uses. This study aims to contribute to the field of materials science and engineering by examining the mechanical and microstructural properties of composite materials. In the process, it seeks to bring insight into the viability of using industrial by-products and ceramic materials as reinforcements.

**EXPERIMENTAL**

The materials used for this research are AA 6063 ingot, mill scale (MS), and Al<sub>2</sub>O<sub>3</sub> having 98.5 % purity. Particle sizes for millscale and alumina were 50 μm and

50 μm, respectively. The AA 6063 ingot was sourced locally from a vendor in Lagos State, Nigeria, the MS was obtained from the Nigerian Foundries Limited, Ota, Ogun, Nigeria. The procurement of alumina was also sourced from a reputable chemical vendor based in Lagos State, Nigeria. Tables 1 - 3 display the elemental compositions of AA 6063 alloy, mill scale, and alumina, respectively.

**Production of the Al hybrid composites**

The metal matrix composite was produced using a two-step stir-casting process, following the methodology outlined by Alaneme et al. [24]. The required amount of aluminium alloy (6063), millscale (MS) and alumina (Al<sub>2</sub>O<sub>3</sub>) particles was determined through charge calculations to achieve a reinforcement content of 8 wt.% in the aluminium matrix. The alumina and mill scale particles were heated at 250°C for 5 min to improve wettability of the particulates in the matrix. The AA 6063 ingots were then heated in a gas-fired crucible furnace to a temperature of around 750°C ± 30°C, which is above the alloy's liquidus temperature. The liquid alloy was allowed to cool in the furnace until it reached a semi-solid condition at roughly 600°C. At this temperature, the heated alumina particles were added, and the slurry was manually stirred for 5 min.

After the composite slurry had been heated to 720°C, a mechanical stirrer was used for a second stirring. For 10 min, swirling at a speed of 300 rpm helped to better distribute the alumina particles throughout the liquid

Table 1. Elemental composition of AA 6063 alloy.

Constituents	Si	Fe	Mn	Mg	Cu	Ti	Zn	Cr	Al
%	0.45	0.22	0.03	0.50	0.02	0.02	0.01	0.03	Bal

Table 2. Elemental composition of millscale.

Constituents	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	LOI (950°C)
%	0.64	0.34	97.96	0.82	0.29	0.09	0.67	0.02	0.01	0.00

Table 3. Elemental composition of alumina.

Constituents	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Others
%	1	98.5	Bal

AA 6063. The furnace's temperature was tracked using an external temperature probe (thermocouple). The liquid mixture was subsequently introduced into a pre-prepared metal mould. Casting was also used to make unreinforced AA 6063 for control experiments. Fig. 1a displays the produced metal matrix composites. Table 4 displays the varying wt. % of mill scale to alumina used in this study. The justification for using 8 wt. % reinforcement in the study may be attributed to several factors, including trade-offs between different material properties. While a higher reinforcement percentage could potentially enhance specific mechanical properties, such as strength and hardness, it might also lead to increased brittleness and reduced ductility. Moreover, a reinforcement percentage of 8 % could achieve a better balance, provide improved mechanical properties while maintain acceptable levels of ductility and toughness.

### Density measurement

The influence of different weight fractions of MS- $\text{Al}_2\text{O}_3$  on the densities of the composites was investigated using both theoretical and experimental approaches, following the methodology outlined by [25]. An accurate digital balance with a tolerance of 0.0001 g was used to weigh the samples, and the experimental density was calculated by dividing the result by the volume of the sample. The rule of mixing, as defined by Adediran et al, was used to determine the theoretical density of the composites [26]. The impact of varying weight fractions of MS- $\text{Al}_2\text{O}_3$  on the composite's density was evaluated by comparing the experimental and theoretical densities. The theoretical density was determined by using the rule of mixtures given by Eq. (1). However, Eq. (2) was used to calculate the composites' percentage porosity based on their theoretical and experimental densities.

$$d_c = d_m * V_m + d_f * V_f \quad (1)$$

where:  $d_c$ ,  $d_m$ ,  $d_f$  are densities of the composite, matrix and dispersed phase respectively;  $V_m$ ,  $V_f$  are volume fractions of the matrix and dispersed phase, respectively.

$$\% \text{ Porosity} = \frac{\rho_\tau - \rho_{ex}}{\rho_\tau} \times 100\% \quad (2)$$

where  $\rho_\tau$  is the theoretical density (in  $\text{g cm}^{-3}$ ),  $\rho_{ex}$  is the experimental density ( $\text{g cm}^{-3}$ )

Table 4. Sample designations for reinforcements:  $\text{Al}_2\text{O}_3$  and millscale (MS).

Sample designation	Compositions	
	Millscale (MS), wt. %	$\text{Al}_2\text{O}_3$ , wt. %
A	8	0
B	6	2
C	4	4
D	2	6
E	0	8
Control	0	0

### Mechanical testing

#### Hardness measurement

This was assessed using an FM-800 microhardness testing machine following ASTM E92-17 standards at the Materials Laboratory of Landmark University. A load of 0.1 kg was applied to each sample for a dwell time of 10 s. Three hardness indents were made on each sample, and the readings within a margin of  $\pm 2\%$  were recorded. The average of the three readings was then calculated as the hardness value for each composition. Figure 1b displays the produced composite samples being indented for the hardness test.

#### Tensile properties

This was assessed using an MTS STH tension meter, following the ASTM E8M-16a standard, at the material testing laboratory located at Obafemi Awolowo University, Ile-Ife, Osun State, Nigeria. Test specimens were machined according to the specifications outlined by Alaneme et al with a gauge length and diameter of 30 mm and 5 mm, respectively [27]. These specimens were securely mounted on the test platform and subjected to tension at a strain rate of  $10^{-3} \text{ s}^{-1}$ . The tests were conducted three times for each composition, and the average results from the three experimental tests were recorded. Key tensile properties, including UTS, yield strength and specific strength were evaluated.

#### Fracture toughness

This was determined utilising circumferential notch

tensile (CNT) specimens according to Adediran et al [9]. The samples were manufactured with a gauge length of 40 mm, a gauge diameter (D) of 6 mm, and a notch diameter (d) of 4.5 mm, resulting in a notch angle of 60°. Using a universal tensile testing machine, the specimens were subjected to tensile loading until rupture. At ambient temperature and a quasi-static strain rate of  $10^{-3} \text{ s}^{-1}$ , the test was conducted. Each designation examination was administered twice to ensure data comparability and accuracy. Based on the fracture load ( $pf$ ) obtained from the load-extension plots of the CNT specimens, the fracture toughness was calculated. In accordance with Dieter [28] and Alaneme and Ajayi [29], the data was then used to calculate the fracture toughness utilising the Eq. (3). Fig. 1c displays the samples for evaluating both the tensile properties and the fracture toughness.

$$K_{IC} = \frac{P_f}{D^{3/2}} \left[ 1.71 \left( \frac{D}{d} \right) - 1.27 \right] \quad (3)$$

where the notched section diameter and gauge diameter, respectively, are indicated by D and d. The accuracy of the calculated fracture toughness values was assessed using the relationship in Eq. (4) according to Nath and Das [30].

$$D \geq \left( \frac{K_{IC}}{\sigma_y} \right)^2 \quad (4)$$

where  $\sigma_y$  is the yield strength and  $K_{IC}$  is the fracture toughness determined.

### Scanning electron microscopy (SEM) characterization

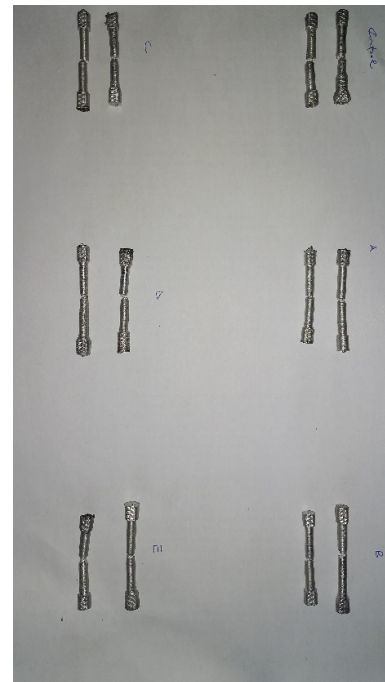
The morphological features of the reinforcements and the produced composites were examined using a Phenom proX scanning electron microscope (SEM), model MVE0224651193. The SEM was operated at an acceleration voltage of 15 keV. Elemental compositions were obtained using the energy-dispersive X-ray (EDX) attachment in the microscope. The SEM analysis was conducted at the research laboratory of Covenant University in Sango Ota, Ogun State, Nigeria and CSIR-NML India. Samples for the composites was prepared through grinding and polishing operations, using automatic grinding and polishing machines. They were then etched in 4 % initial for 8 to 10 s before the morphological examination.



a)



b)



c)

Fig. 1. (a) Developed MMC samples (b) hardness samples (c) fractured samples.

## RESULTS AND DISCUSSION

### Density and porosity

Fig. 2 illustrates the relationship between density, percentage porosity and the weight percentage of millscale and alumina in the produced AMMCs. Sample A exhibited the highest density among all compositions with a weight ratio of 8:0 of alumina to millscale. This can be attributed to the higher density of alumina ( $3.95 \text{ g cm}^{-3}$ ) compared to the base alloy ( $2.70 \text{ g cm}^{-3}$ ), resulting in a slight increase in the density of sample A. The introduction of millscale only as reinforcement in sample E (8 wt. % MS) resulted in increased density compared to the control sample. However, the density obtained when alumina was used as the only reinforcing particle in the matrix (sample A (8 wt.%  $\text{Al}_2\text{O}_3$ )) was higher than that of sample E. This can be attributed to the influence of the higher density of alumina over MS when used as reinforcements. Conversely, the inclusion of hybrid reinforcements of alumina and millscale in the matrix (samples B - D) showed better density compared to sample E and lower density compared with sample A. As the millscale percentage increased in the hybrid reinforcing particulates, the density of the samples decreased. This is because millscale possesses a lower density than the alumina content. Similar observations were reported by Prasad et al and Edoziuno et al in studies involving hybrid reinforcements and novel PKS reinforcement, respectively [16, 31]. The densities of samples B through E, however, were higher than those of the control sample. This is influenced by the presence of hybrid millscale and alumina reinforcing particles, which have densities higher than the base alloy.

The percentage porosities of the produced metal matrix composites (MMCs) were generally below 2.9 %, which is considered within acceptable limits (4 %) for cast MMCs. However, it should be noted that sample B exhibited a higher porosity of 4.51 %, which could be attributed to the instability of the weight ratio of 6:2 between millscale and alumina during the casting process, resulting in an uneven reinforcement distribution across the matrix alloy. Porosity could result from trapped gas bubbles and poor wettability of the reinforcement, as observed by Edoziuno et al [16]. The incorporation of magnesium and the double stir- method, improved wettability and reduced the tension on the surface between the base alloy and the reinforcement particles, which contributed to the reduction in porosity.

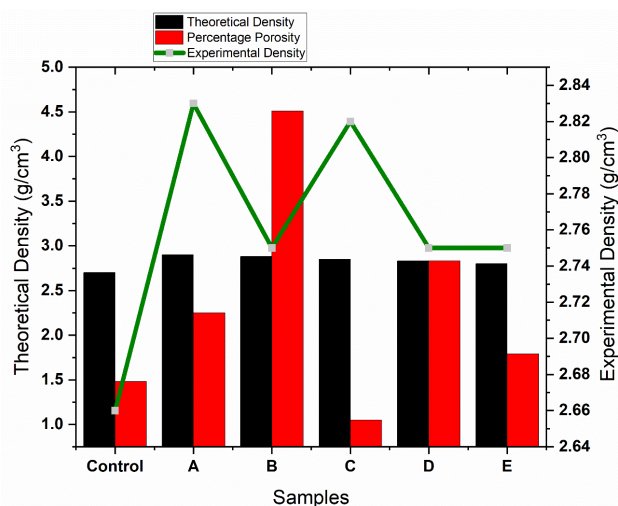


Fig.2. Density and porosity of AMCs samples.

### Mechanical properties

#### Hardness

The average hardness values of the composites are illustrated in Fig. 3 and it is commonly understood that the hardness of a material influences its toughness, strength and resistance to wear. In this study, it was observed that the hardness of the composites slightly increased as the weight ratio of alumina in the reinforcement mix increased. This increase in hardness can be attributed to the presence of hard phases from the millscale and other strengthening components in the aluminium alloy. It is worth noting that alumina is known to have relatively lower hardness compared to the millscale, which contributes to the observed hardness values [32].

The findings of this study, which demonstrate increased values for hardness in the fabricated MMCs, make them appropriate for lightweight applications requiring moderate hardness such as architectural applications, window and door frames. Fig. 3 demonstrates that all reinforced alloys have higher hardness values than the unreinforced alloy, with values spanning from 9.654 to 16.905 BHN. Compared to the control sample with a hardness value of 9.654 BHN, Sample D exhibited an increased hardness value of 13.2 BHN. This increase is attributable to the presence of minor volume fractions of refractory phases such as  $\text{MgO}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{CaO}$ ,  $\text{SO}_3$ ,  $\text{K}_2\text{O}$ ,  $\text{Na}_2\text{O}$ ,  $\text{FeO}_3$ , and  $\text{SiO}_3$  in millscale particulates. These findings are

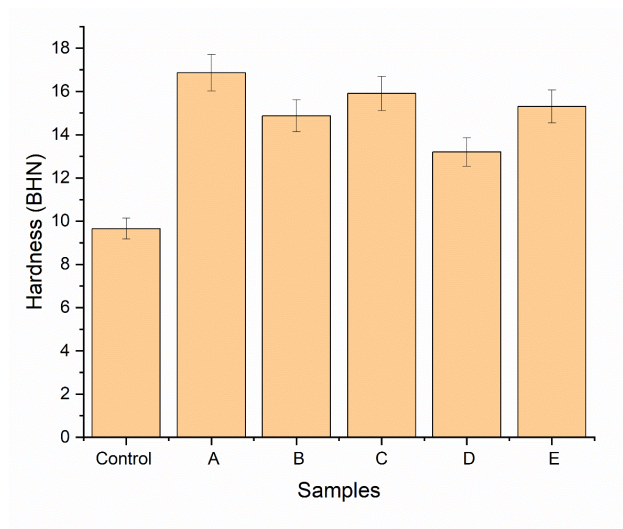


Fig. 3. Hardness of AMCs samples.

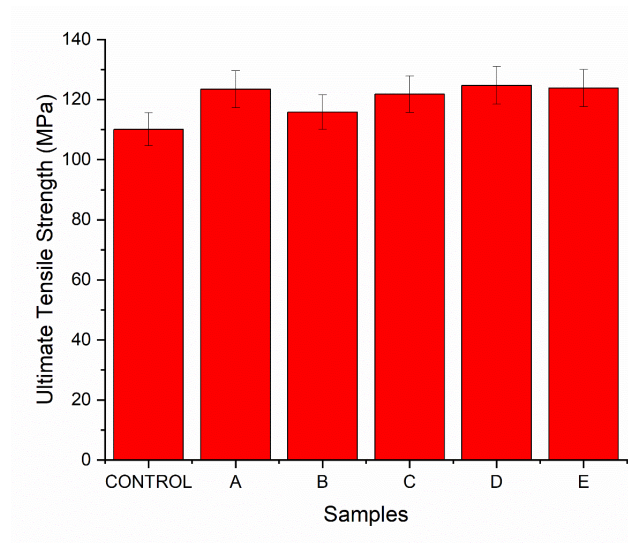


Fig. 4. Ultimate tensile strength and yield strength of AMCs Samples.

consistent with Alaneme et al [24]. Furthermore, the higher hardness of sample A compared to sample D could be attributed to the greater hardness and strength of alumina particulates present.

### Universal tensile strength (UTS)

The ultimate tensile strength (UTS) of the hybrid reinforced composites was assessed by analysing the stress-strain curves obtained from the universal testing machine. The UTS of the composites is shown in Fig. 4. The ultimate tensile strength (UTS) values of the samples varied between 110.11 MPa and 124.73 MPa. Sample A demonstrated a higher ultimate tensile strength (UTS) than the unreinforced control samples. The increase in UTS values can be partially attributed to the presence of millscale particulates. The incorporation of millscale and alumina particulates, within the range of 2 to 8 wt. %, led to an enhancement in the ultimate tensile strength (UTS) for samples B - D. Additionally, higher alumina content in the composites led to enhanced strength. The presence of millscale, which contains oxides such as  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$  and  $\text{MgO}$ , enhanced the tensile strength. The correlation between UTS and hardness aligns with prior research findings [27, 33].

The rise in ultimate tensile strength (UTS) in composites can be ascribed to three mechanisms: thermal mismatch due to differing coefficients of thermal expansion between reinforcement and matrix materials,

grain boundary strengthening and matrix material strengthening [34]. Atuanya et al. identified that various factors, such as grain boundaries, solid solutions, sub-structures and secondary phases, significantly influence the mechanical properties of metallic materials [35]. Grain boundaries impede dislocation movement and enhanced grain refinement results in increased strength of the composite [36].

The alumina reinforcement particles are critical for load transmission, thereby enhancing the strength of the composites. An increase in the volume proportion of reinforcements within the matrix alloy results in enhanced strength due to the transmission of greater loads. This outcome aligns with the findings of Atuanya et al [35]. A low volume proportion of particles leads to increased grain sizes in the matrix, potentially activating the Orowan bypass mechanism within the larger grains. Conversely, a higher proportion of reinforcing particulates diminishes grain sizes, resulting in grain refinement and enhanced tensile strength values through direct load-bearing transfer at the interface [37]. Furthermore, the UTS rose with an increase in the weight percentage of alumina in the reinforcement. The UTS values rose by 4.8 % with the incorporation of 2 wt. %  $\text{Al}_2\text{O}_3$  as reinforcement (Sample B), yet remained inferior to those of Sample A. The presence of hardeners in the millscale particles contributes to the strength increase observed in sample B relative to the control

sample. The increase in the weight percentage of alumina in the composites accounts for the rise in ultimate tensile strength (UTS) observed in samples C to D. This finding aligns with results from multiple studies conducted by Kumar et al. and Ikubanni et al [10, 33]. The noted increase in UTS in alumina-containing samples indicates a more robust bond between the matrix alloy and alumina particulates during fabrication, facilitated by continuous stirring to enhance the distribution of reinforcements throughout the matrix alloy [27].

### Yield strength

Fig. 4 illustrates the yield strength of the synthesized composites. The yield strength of composite materials is influenced by the weight ratio of millscale to alumina reinforcement. Sample A, which contained a millscale weight ratio of 2 %, demonstrated the highest yield strength at 118.8 MPa. The incorporation of millscale reinforcement positively influences the yield strength of the composites. In comparison to the control sample (93 MPa), the other samples (B, C, D, and E) exhibited enhanced yield strength values. Nevertheless, the yield strength values for these samples were slightly lower than those of sample A. The incorporation of millscale reinforcement contributes to an increase in yield strength; however, the increasing alumina content seems to have a more significant effect on yield strength enhancement due to its strong bonding characteristics. The results align with previous studies that investigated the impact of reinforcement combinations on the mechanical properties of composite materials. Babu et al observed analogous trends in their investigation of aluminium-based composites reinforced with alumina and millscale, indicating that increased alumina content resulted in enhanced yield strength [38].

### Specific strength

Fig. 5 illustrates the specific strength of the synthesised composites. The specific strength values vary from 41.4 MPa g cm<sup>-3</sup> for the control sample to 45.4 MPa g cm<sup>-3</sup> for sample D. The specific strength values demonstrate that incorporating alumina as a reinforcement enhances the specific strength of the composites relative to the control sample. This improvement is due to the advantageous properties of alumina, including its high strength and rigidity. Prior research by Rajak et al. and Patel et al. indicated

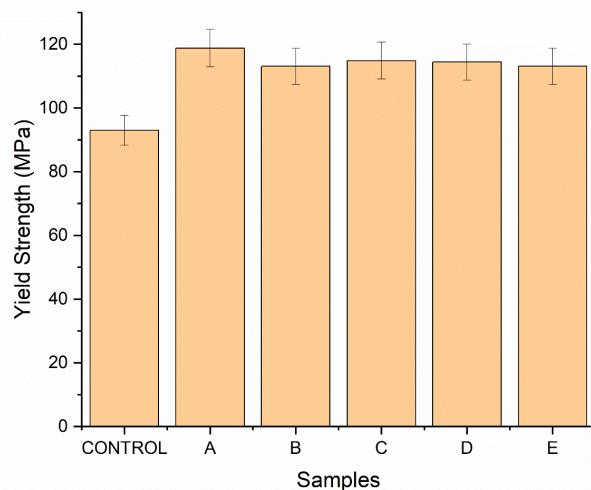


Fig. 5. Specific Strength of AMCs Samples.

increasing trends, highlighting the beneficial effect of alumina reinforcement on the specific strength of composites [39, 40]. Additionally, an analysis of the specific strength values across various samples (A, B, C, D, and E) reveals a discernible trend. Sample D, containing the highest weight percentage of alumina at 6 %, demonstrates the greatest specific strength compared to all other samples. An increased concentration of alumina reinforcement enhances the specific strength of the composites. Parveez et al. reported findings, noting an increase in specific strength associated with elevated alumina content in composites [41]. It is important to observe that the specific strength enhancement attains a plateau after a certain threshold. Samples B and C, which contain 2 % and 4 % alumina, respectively, exhibit marginally lower specific strength values than sample D. This suggests an optimal range of reinforcement composition that provides the best balance between strength and weight.

### Percentage elongation (strain to fracture)

The percentage elongation (% Elongation) is a measure of a material's ability to stretch or the percentage elongation (% Elongation) quantifies a material's capacity to stretch or deform before fracture under tensile stress. This analysis offers insights into the ductility and toughness of a material. Fig. 6 illustrates results indicating a nearly uniform trend in which the

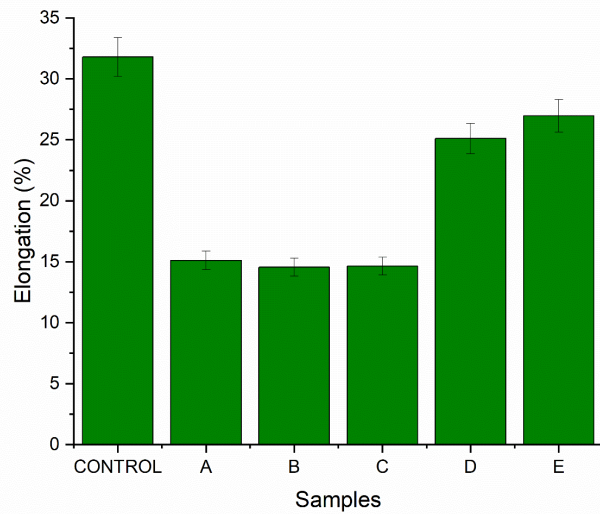


Fig. 6. Percentage elongation of AMCs samples.

incorporation of millscale and alumina reinforcement results in a reduction in percentage elongation relative to the control sample. The observed reduction in elongation suggests that reinforcement limits the material's capacity for plastic deformation before fracture. The reinforcing particles impede dislocation movement, leading to a reduction in ductility [32]. Singh and Goyal investigated the influence of various reinforcement materials on the mechanical properties of metal matrix composites [42]. Their research indicated a comparable reduction in percentage elongation as reinforcement content increased. The inclusion of reinforcing particles hindered the plastic deformation process, resulting in decreased elongation of the composite materials. Studies by Bhoi et al examined alumina-reinforced composites, demonstrating a gradual decrease in percentage elongation with an increase in the weight fraction of alumina reinforcement [43]. The relationship between reinforcement content and decreased elongation is due to the enhanced stiffness and diminished plasticity resulting from the reinforcement particles. Samples D (25.1 %) and E (27.0 %), which contained greater weight ratios of alumina reinforcement, demonstrated marginally higher percentage elongation values than samples A (15.1 %), B (14.6 %), and C (14.6 %). The inclusion of alumina reinforcement enhances toughness and localised plastic deformation, resulting in greater elongation relative to samples with increased millscale content.

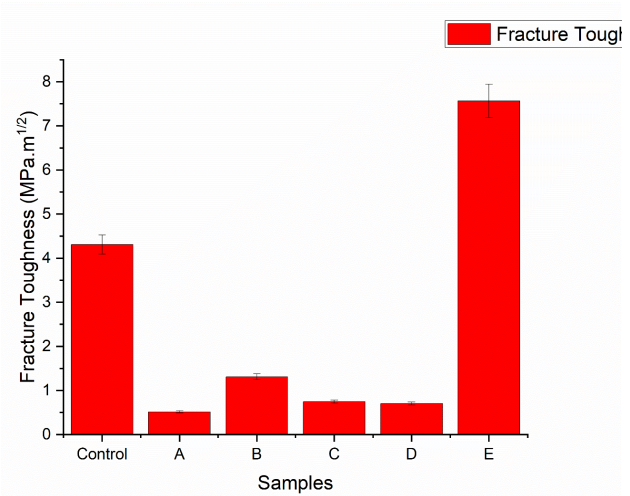


Fig. 6. Fracture toughness of AMCs samples.

### Fracture toughness

Fig. 6 provides the results for the fracture toughness of the composites, indicating their resistance to crack propagation. The investigation revealed that an increase in the weight ratio of alumina corresponded with a decrease in fracture toughness values, which ranged from 0.5 to 7.6 MPa•m<sup>0.5</sup>. This outcome aligns with the findings of Alaneme et al., which indicated that fracture toughness values decrease with increasing alumina content [27]. The unreinforced matrix exhibited the highest fracture toughness, recorded at 121.26 MPa•m<sup>0.5</sup>. Deng et al. identify fracture mechanisms, including particle cracking, interfacial cracking, and particle debonding, in ceramic-reinforced metal matrix composites [44]. Brittle alumina reinforcement particles, characterized by their susceptibility to cracking due to reduced resistance to crack propagation, may enhance crack nucleation and elucidate the observed trend, as noted by Alaneme et al [27]. Additionally, Sample A, devoid of alumina reinforcement, demonstrated a fracture toughness value of 0.52 MPa•m<sup>0.5</sup>. Sample E, incorporating a monolithic alumina reinforcement, exhibited superior fracture toughness relative to the other composite samples produced. Alumina, as a ceramic material, exhibits intrinsic brittleness, which results in swift crack propagation and the possibility of catastrophic failure under certain conditions, as noted by [45]. Bagatini et al. investigated aluminium

composites reinforced with alumina and millscale [46]. The findings indicated that alumina, owing to its brittle characteristics, demonstrated a greater propensity for crack initiation and propagation relative to millscale particles. This behaviour is attributed to the increased hardness and decreased toughness of alumina, leading to the accelerated formation and propagation of cracks within the material.

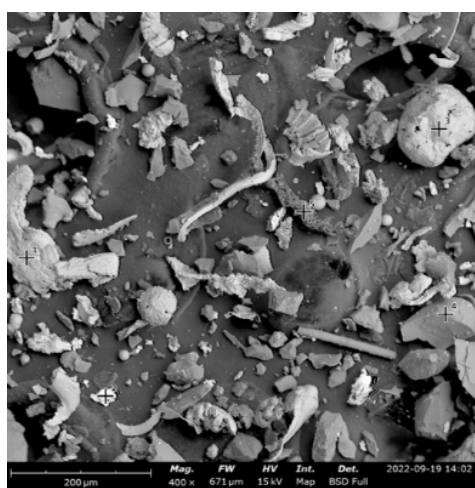
The computed fracture toughness values offer important insights into the mechanical behaviour of the produced composites. The observed trend highlights the influence of alumina content on fracture toughness, suggesting that a balanced relationship between reinforcement content and material properties is crucial for optimising the fracture toughness of composites. The findings enhance the understanding of fracture behaviour in similar composite systems and align with prior studies [9].

### **Microstructure analysis and characterization of millscale and alumina**

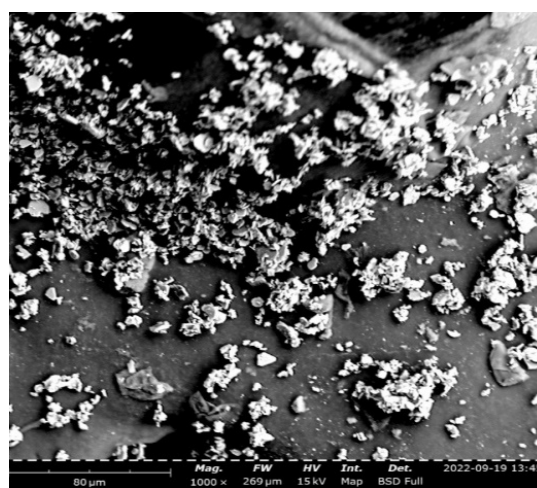
Fig. 7 (a) and (b) provide micrographs illustrating the microstructure of millscale and alumina, respectively. The SEM analysis of the millscale reinforcement material revealed significant findings related to its surface morphology, composition, distribution and bonding characteristics. The millscale particles displayed a rough

surface texture, suggesting the existence of irregularities and rough features, primarily due to their composition of iron oxides. The SEM images confirmed the presence of iron oxides, thereby validating the composition of the millscale particles. SEM analysis indicated the scattering of millscale particles within the alloy matrix, implying effective dispersion. The dispersion is essential for facilitating uniform load transfer and improving mechanical performance.

The SEM analysis revealed visible defects, notably weak bonding between the millscale particles and the matrix, suggesting suboptimal interfacial bonding. The weak bonding adversely affects mechanical properties, leading to compromised load transfer efficiency and reduced overall strength. The SEM observations yield important insights into the surface morphology and microstructural characteristics of millscale as a reinforcement material. They highlight the importance of factors such as roughness, iron oxide content, distribution and bonding strength in the assessment of millscale-reinforced composites [47]. The results of this SEM analysis are consistent with prior research in the discipline. Jjagwe et al. investigated findings concerning the rough surface morphology and the occurrence of iron oxides in millscale particles [48]. Durowaye et al. examined the dispersed distribution of millscale particles and their effect on mechanical properties, highlighting

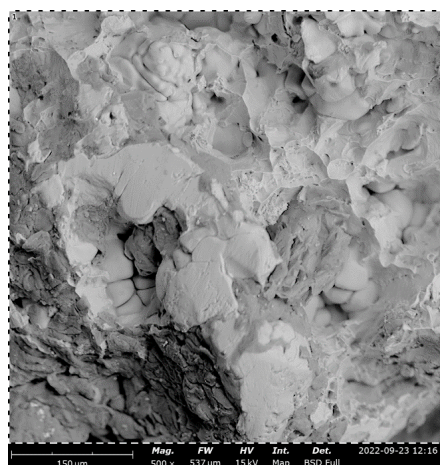


a)

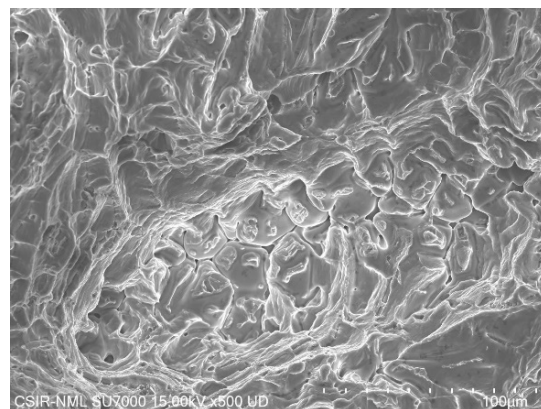


b)

Fig. 7. (a) SEM of millscale; (b) SEM of alumina.



a)



b)

Fig. 8. SEM of the fractured surface of (a) sample D reinforced with 2 % MS and 6 %  $\text{Al}_2\text{O}_3$ , and (b) the control sample.

the significance of interfacial bonding in millscale-reinforced composites and its influence on strength and durability [49].

The SEM analysis of the alumina reinforcement material revealed significant observations. The alumina particles exhibited a rough texture with irregularities, aligning with the typical surface morphology of ceramic alumina. Alumina particles exhibited a non-uniform distribution within the matrix, resulting in variations in load transfer and stress concentration. Despite the non-uniform distribution, the SEM analysis revealed well-bonded clusters of alumina particles, indicating robust interfacial bonding and enhanced load transfer efficiency. Minimal visible defects were noted, suggesting satisfactory structural integrity; however, the existence of pores may influence material density and additional properties.

The SEM observations yield important insights into the surface morphology, distribution, bonding, and defect characteristics of alumina as a reinforcement material, enhancing our understanding of its behaviour and suitability for composite applications. The results of this SEM analysis are consistent with earlier research on alumina-reinforced composites, including studies by Khaerudini et al. [50], Adesina et al. [51], and Touzi and Horchani-Naifer [52]. These studies similarly examined surface morphology, distribution, interfacial bonding, and defect analysis in alumina-reinforced composites.

### Characterization of fracture surfaces

Fig. 8a depicts a SEM investigation of the broken surface (Sample D), which offers substantial details about the material's fracture behaviour. The fracture mode was characterised as ductile, which implies the material underwent plastic deformation and absorbed energy before collapsing. The surface morphology revealed roughness, abnormalities, and different textures, indicating a complicated fracture mechanism involving numerous phases. Evidence of crack initiation was found, implying that pre-existing flaws or stress concentrations likely initiated crack propagation, contributing to the overall fracture. There were also indicators of interfacial debonding, which indicated weak bonding between phases or constituents in the composite. This debonding prevents load transfer, promotes crack development, and lowers mechanical performance. Voids were also found to behave as stress concentrators, amplifying localised stress and influencing fracture behaviour. The presence of these voids indicates porosity, which may undermine the material's strength and endurance.

Further analysis of the control sample fracture surface (Fig. 8b) at 500x magnification revealed surface pits, indicating localised material degradation, possibly because of corrosion or erosion. The overall fracture pattern was complex, with cracks spreading along several routes caused by contaminants, material imperfections, or mixed-mode fracture mechanisms. There was no

apparent fracture route, indicating that cracks formed randomly due to local stress concentrations or pollutants. The largely uniform surface roughness across the cracked area indicates that surface energy and abrasion did not play a substantial role in crack initiation or propagation. This behaviour is consistent with ductile fracture mechanisms observed in similar materials, in which fracture processes are governed by plastic deformation, crack initiation, and interfacial debonding. Balokhonov et al. and Halil et al. studied comparable composites and found that voids and interfacial debonding boost localised stress concentration and crack propagation, eventually impacting the material's fracture behaviour [53, 54].

## CONCLUSIONS

The physico-mechanical properties of hybrid reinforced (millscale and alumina) Al matrix composites were investigated. The analysis of the composites' physical properties indicated that the density and porosity of the developed materials were affected by the weight ratio of alumina and millscale utilised as reinforcements. Sample A with 2.83 g/cm<sup>3</sup> had the highest density amongst other developed composite samples which is attributed to the density of alumina as compare with millscale. The porosity of the composites, despite variations, remained within acceptable limits (1.05 % - 4.51 %), demonstrating the effectiveness of the fabrication process in producing solid composites.

The incorporation of millscale and alumina resulted in a significant enhancement of the mechanical properties of the composites. The addition of alumina resulted in increased hardness (16.905 BHN), yield strength (118.8 MPa) and tensile strength of the materials (124.73 MPa), significantly enhancing specific strength and improving the strength-to-weight ratio. The ductility of the composites, as measured by percentage elongation, diminished with an increase in reinforcement content. The observed reduction in ductility is likely attributable to the brittle characteristics of alumina particles, which also led to a decrease in fracture toughness. The stress-strain curves obtained during testing illustrated the materials' capacity to endure applied loads, indicating their suitability for lightweight applications that necessitate a balance between hardness and strength.

Scanning electron microscopy (SEM)

characterization of millscale and alumina offered insights into the morphology and distribution of reinforcement particles within the aluminium matrix. Millscale particles demonstrated a rough, irregular texture and exhibited a scattered distribution with reduced bonding to the matrix. In contrast, alumina particles exhibited a consistent rough texture characteristic of ceramic materials and formed well-bonded clusters, which contributed to the enhanced mechanical properties observed in the composites. The findings highlight the significance of appropriately selecting and combining reinforcement materials to attain the desired properties in aluminium matrix composites.

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## Authors' contributions

*A.A.A. designs the project, supervise the project, prepare the samples and analyse all the data; O.A.M contribute to the analysis of the data and revise the manuscript; P.P.I supervise the project and contribute to the analysis of the data; C.I.M contribute to the analysis of the data and R.U contribute to the analysis of the data.*

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