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Investigating the Effect of Fly Ash Addition on the Metallurgical and Mechanical Behavior of Al-Si-Mg-Cu Alloy for Engine Cylinder Head Application

Karthik Venkitraman Shankar ^{1,*} , Jan Jezierski ^{2,*} , Vaira Vignesh Ramalingam ³ , Devaprasad Padmakumar ¹, Midun Raj Leena ¹, Amal ¹, Gokul Reghunath ¹ and Rakesh Krishnan ¹

¹ Department of Mechanical Engineering, Amrita Vishwa Vidyapeetham, Amritapuri, Kollam 690525, India

² Department of Foundry Engineering, Silesian University of Technology, Towarowa 7, 44-100 Gliwice, Poland

³ Department of Mechanical Engineering, Amrita School of Engineering, Amrita Vishwa Vidyapeetham, Coimbatore 641112, India

* Correspondence: karthikvs@am.amrita.edu (K.V.S.); jan.jezierski@polsl.pl (J.J.); Tel.: +91-995-253-2461 (K.V.S.); +48-691-544-485 (J.J.)



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Abstract: The authors researched the physical, metallurgical, and mechanical characteristics of A354 alloy (Al-Si-Mg-Cu) reinforced with 5, 10, and 15 wt% of fly ash metal matrix composites. A baseline alloy and three composites were fabricated by a liquid metallurgy route and poured into a permanent mold to obtain cast rods of dimension $\Phi 32 \text{ mm} \times 156 \text{ mm}$. The metallurgical characterization of the developed alloy and metal matrix composites was conducted using energy-dispersive spectroscopy (EDS), field-emission scanning electron microscopy (FESEM), and X-ray diffraction. All the developed composites showed a pore-free nature, but only A354 alloy reinforced with 5 wt% of fly ash (AF₅) possessed a homogeneous distribution and perfect bonding of the fly ash with the A354 matrix. Therefore, transmission electron microscopy (TEM) analysis was performed on the sample AF₅. All developed alloys and metal matrix composites were subjected to hardness and mechanical property tests. It was observed that the AF₅ sample had $170 \pm 5.6 \text{ HV}$ and tensile strength of $216 \pm 2.3 \text{ MPa}$, 18.8% and 24.8% higher than the A354 matrix, but the ductility ($6.5 \pm 0.43\%$) was reduced by 23% from the baseline alloy. Finally, the fractography analysis was conducted on all the samples using FESEM to analyze the fracture mode. The fabricated 5 wt% fly ash-based metal matrix composite showed better mechanical performance than other samples. Hence, sample AF₅ is suggested for manufacturing components in automotive and structural parts.

Keywords: A354; fly ash; metal matrix composite; hardness; mechanical properties; automotive application; Al-Si-Mg-Cu-fly ash; A354—fly ash; stir casting/liquid metallurgy route; fractography

1. Introduction

Aluminum and its alloys are utilized in numerous applications in the automotive industry. Many manufacturers use these alloys to cast various parts of passenger cars, substituting cast iron in manufacturing cylinder blocks and cylinder heads to reduce weight. Approximately 20% of CO₂ and other harmful human emissions come from vehicles that affect the world's ecosystem. The increase in the emission of harmful pollutants, extreme consumption of energy and resources, and ineffective recycling practices significantly affect the environment [1]. In this regard, using Al and its alloys or composites has become essential [2]. Due to financial pressure to reduce harmful CO₂ emissions and fuel consumption, researchers conduct investigations to minimize the weight of the cars' structures, and the design has been considerably improved [3,4]. The manufacture of molded automotive parts (particularly cylinder heads) comprises alloys from the 3xx.x series (Al-Si-Mg/A356, Al-Si-Cu, Al-Si-Mg-Cu/A354) [5–9]. These alloys are considered to possess good castability [10,11], relatively low weight, and moderate mechanical properties [5–9]. Approximately

100% of engine pistons, about 80–85% of exhaust manifolds, 70–75% of combustion engine cylinder heads and gearbox housings, and other power transmission components such as drive shafts, rear axles, and differential casings are manufactured in the form of metal casting [2,5,12]. Generally, the cylinder heads are manufactured using permanent mold casting/gravity die casting [6,9,13]. As these Al alloys possess only moderate hardness, strength, and ductility and feeble wear resistance, studies have focused on implementing a single technique or a combination of multiple techniques such as T₆ heat treatment, surface modification, addition of alloying elements, and reinforcing ceramic particles in the matrix (Metal Matrix Composites) [14].

Metal matrix composites (MMCs) acquire considerably better properties, including high mechanical properties, specific modulus, specific strength, damping capacity, and reduced specific wear rate and low density [15–18]. The reinforcement used in the metal matrix affects the mechanical strength and durability of the developed material compared to the parent material/unreinforced alloy [18]. There has been an emerging trend among the industries in procuring composite materials with low-cost reinforcements and low density [19]. Among several ceramic reinforcements used by different researchers, fly ash is an inexpensive suspended particulate matter obtained as a solid waste by-product during combustion in coal-fired thermal power plants and cement kilns in enormous quantities [20–22]. Hence, MMCs with fly ash as reinforcement gain more importance and are expected to overcome the cost obstacle for the extensive application of composite materials in the automotive and aerospace industries. In addition, as part of industrial symbiosis, waste products that cause air pollution can be safely recycled and disposed of.

MMCs are fabricated by stir casting [18], friction stir processing [23], ball milling [24,25], powder metallurgy, and vacuum hot pressing. Among these techniques, casting is the most popular manufacturing method due to its low cost and high production rate [18]. Research has been conducted on reinforcing various ceramic particles into Al and its alloys to enhance their mechanical and tribological properties. Several authors have investigated the reinforcing of fly ash in the Al-Si-Mg alloy and observed the variation in mechanical and tribological properties [26–28]. He et al. [29] developed a composite material to reinforce fly ash nanoparticles to the Al-Si-Mg-Cu alloy by ultrasonic vibration. It was observed that A354/SiC/0.5p-T6 with an additional 15 min of ultrasonic treatment had the highest mechanical properties. Du et al. [30] investigated the effect of SiC on Al-Si-Mg-Cu-Ni through an in situ process. It was observed that the developed composites might be suitable for high-temperature applications. Aybarc et al. [31] presented an overview of reinforcements such as SiC, Al₂O₃, and graphene introduced into the aluminum alloy matrix. The research confirmed that the stir casting method is the best way to introduce solid particles into a liquid aluminum alloy matrix. With its outstanding properties, graphene appears to have the potential to become a perfect reinforcement for aluminum alloys. This combination can produce higher toughness with minimal additions compared to ceramic particle additions [31]. Some authors conducted experiments with the carbon nanotube-reinforced aluminum matrix composites, as described in [32]. There has been significant progress in the field of CNT-reinforced aluminum composites. However, due to the problems regarding CNT dispersion and a weak bonding interface, CNT–Al composites are still in the early stage of exploration, and practical applications are still minimal [32]. Another approach was presented in [33], where AMC reinforced with boron nitride nanotubes (BNNT) was described. Again, some problems appear when the particles are to be introduced into the liquid metal matrix. Therefore, different methods were investigated, and it was found that UTS increased by 20% and elongation at fracture was increased by 170% for some combination of treatment parameters [33]. To examine different properties of modern aluminum matrix composites, new methods have been developed, as in [34], where the free vibration analysis was used to investigate the A357 alloy reinforced with dual-particle-size silicon carbide metal matrix composite.

The above discussion shows that most studies are conducted to reinforce fly ash to Al-Si-Mg alloys. No research has been undertaken to strengthen these particles to the A354

alloy. Since Al-Si-Mg-Cu alloy is a promising material for manufacturing cylinder heads in the automobile industry, and fly ash particles in the air harm humanity, recycling these fly ash particles as a reinforcement is worthwhile. Therefore, this current investigation is conducted to enhance the properties of the Al-Si-Mg-Cu alloy by reinforcing varying weight percentages of fly ash and to determine the morphological and mechanical behavior of the developed metal matrix composites.

2. Materials and Methods

2.1. Fabrication of Metal Matrix Composite

The Al-7Si-3Cu-0.4Mg alloy that belongs to the Al-Si-Mg-Cu group of alloys is used to manufacture cylinder heads and other automotive components [9,30,35,36]. The chemical composition of the alloy investigated in this research is presented in Table 1. The casting manufacturer supplied the alloy melted in an electric furnace with an overheat level of 760–780 °C. The electric furnace was equipped with a mechanical stirrer to obtain a homogeneous distribution of the reinforcement in the matrix. Fly ash with an average particle size of 5 µm (analyzed with Image J software, V 1.8.0. 4.1. (827)), as shown in Figure 1, was preheated in an electric furnace at 300 °C and gradually added to the melt with the support of a hopper. Table 2 depicts the chemical composition of the fly ash particle used in this present investigation. A schematic image of the electric furnace equipped with a mechanical stirrer and hopper is shown in Figure 2. The melting of the composite was conducted in an inert Ar atmosphere to prevent the oxidation of the molten metal. The stirrer was rotated at 200 rpm for 10 min to obtain a uniform distribution of fly ash in the Al matrix. To enhance the wettability of the reinforcement with the matrix, 1 wt% Mg was added to the melt. After proper mixing, the melt was poured into a preheated Lubrikote-coated permanent mold to obtain cast rods of dimensions $\Phi 32 \text{ mm} \times 156 \text{ mm}$, as shown in Figure 3. The procedure mentioned above was followed to obtain MMCs of varying wt% of fly-ash (0, 5, 10, 15). Table 3 shows the details of the materials used in the investigation. As per ASTM standards, the cast specimens were machined to suitable dimensions for morphological and mechanical property testing. Advanced instrumental characterization (FESEM, EDS, TEM, XRD) was used to characterize the specimens.

Table 1. Chemical composition of the base alloy used in the study.

Elements	Al	Si	Cu	Mg
wt%	Rem.	6.9	2.8	0.39

Table 2. Chemical composition of the fly-ash particle.

Elements	Al	Si	O	C	Fe	Others (Na, Mg, K, P, Ca)
wt%	9.7	17.8	34.8	2.8	3.3	Rem.

Table 3. Details of the samples used in the investigation.

Sample Code	Matrix	Reinforcement	Wt% Reinforcement
A	A354	-	-
AF ₅	A354	Fly ash	5
AF ₁₀	A354	Fly ash	10
AF ₁₅	A354	Fly ash	15

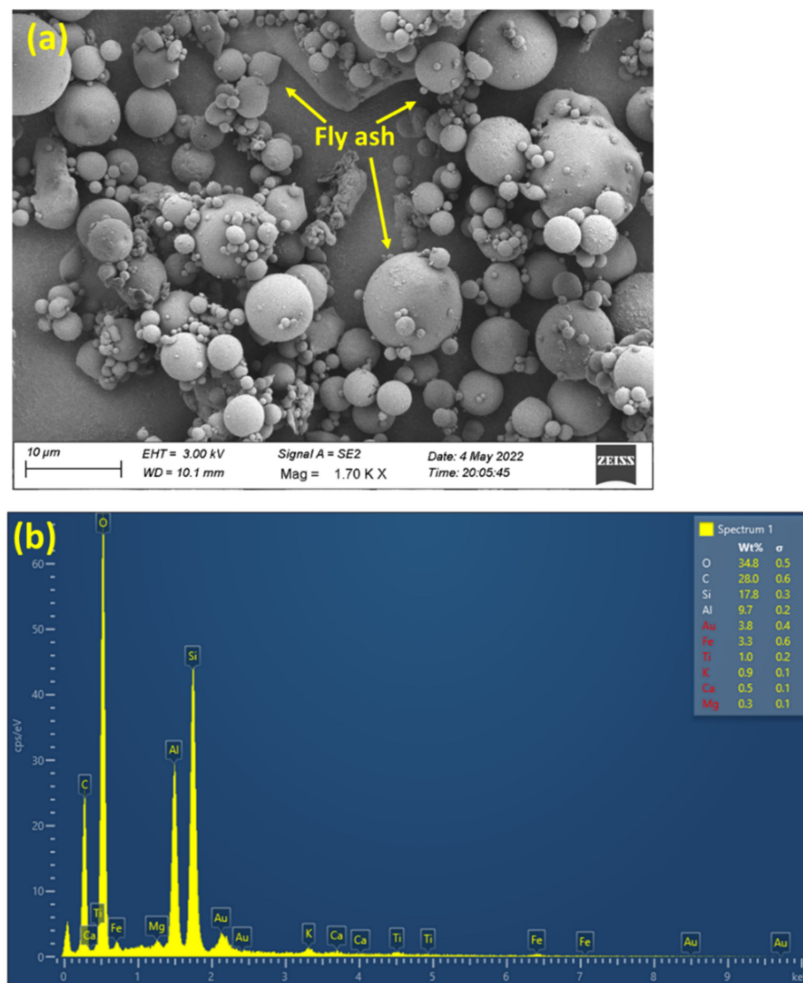


Figure 1. Image showing (a) fly ash particles at higher magnification and (b) EDS analysis of fly ash.

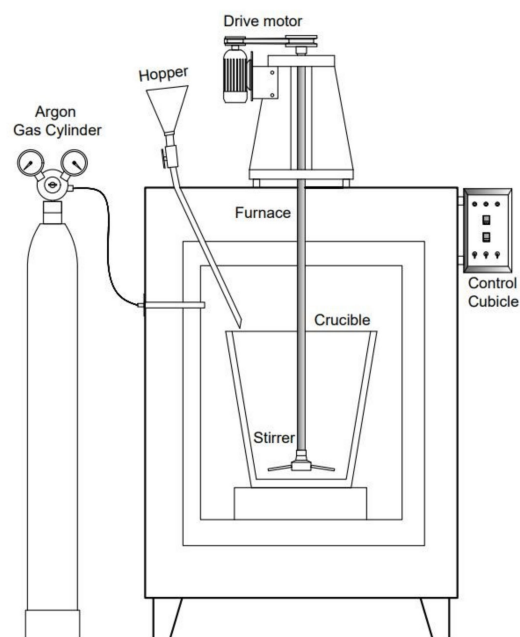


Figure 2. A melting furnace equipped with a mechanical stirrer.



Figure 3. Cast sample rods.

2.2. Density

Using the Archimedes principle, the experimental density values for all samples depicted in Table 4 were calculated. The test was carried out according to the ASTM B962-13 standard. The theoretical density for all samples was calculated using the rule of mixtures. Table 3 shows the values of the experimental and theoretical density. Table 3 shows that the value of the experimental density was found to be lower than the theoretically obtained density. The theoretical density of the fabricated composites (AMMC) was calculated using Equation (1), where $\rho_{(AMMC)}$ is the density of aluminum metal matrix composite, ρ_m is the matrix density, ρ_p is the particle density, and $V_{f(m)}$, and $V_{f(p)}$ are the volume fraction of matrix and reinforced ceramic particles. Before calculating the theoretical density using the mixtures rule, the alloying elements' weight percentage was converted into volume fractions.

$$\rho_{(AMMC)} = \rho_m \times V_{f(m)} + \rho_p \times V_{f(p)} \quad (1)$$

Table 4. Density value of A354 and its composites.

Sl No	Samples	Density Values in g/cm ³	
		Experimental	Theoretical
1	A	2.75 ± 0.014	2.79
2	AF ₅	2.45 ± 0.013	2.53
3	AF ₁₀	2.42 ± 0.012	2.48
4	AF ₁₅	2.39 ± 0.011	2.41

2.3. Microstructure and Hardness Test

Scanning electron microscope (SEM) and energy-dispersive spectroscopy (EDS) were used to analyze the metallurgical behavior and composition of the developed alloy and MMCs used in this investigation. Before SEM analysis, the specimens for microstructure and microhardness were prepared as per the standard (American Society for Testing and Materials) ASTM E3-01. The specimens were cut to dimensions of 10 mm × 10 mm. The specimens were mounted using a cold-setting compound. The mounted samples were polished in automatic disc-polishing units with various abrasive sheets (400 grit to 2000 grit). The specimens were then polished in a slurry of diamond particles to achieve a mirror-polished surface. Subsequently, the samples were etched using the standard etchant prescribed in the ASM handbook. Vickers microhardness tester (Mitutoyo, Kawasaki, Japan, Model: HM-210 A) was used to ascertain the microhardness value of the fabricated base alloy (A354) and MMC according to the ASTM E-384 standard. Before testing the hardness value, standard silicon carbide paper grit (emery sheet) was used to polish the surface of

the specimens. A load of 1N was applied to the sample's surface for approximately 30 s. The indentation was applied at five different locations, and an average hardness value for the material was calculated. The above procedure was repeated for all samples shown in Table 2.

2.4. Tensile Test

Tensile tests for the base alloy and fabricated MMCs were conducted using a universal testing machine (Tinius Olsen). The samples were machined according to the ASTM E8-04 standard. All samples were machined per the dimensions depicted in Figure 4. Fractography analysis was performed using FESEM analysis to determine the failure mode.

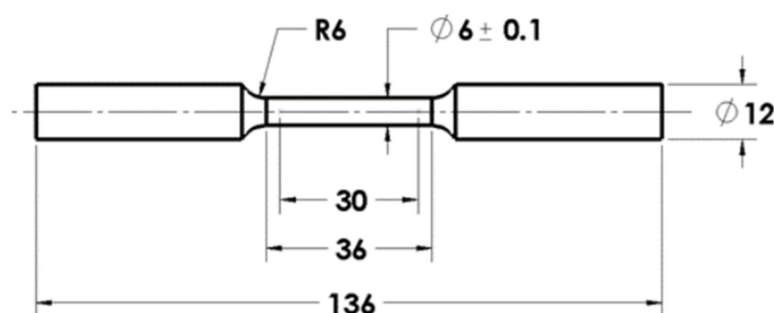


Figure 4. ASTM standard used for machining tensile samples (All dimensions are in mm).

3. Results and Discussions

3.1. Metallurgical Analysis

Figure 5a shows the microstructure of A354 alloy in the as-cast condition. The microstructure of A354 in the as-cast state comprises α -Al enriched and eutectic Si phase. In addition, several intermetallic phases were observed in the grain boundaries of the alloy in the as-cast condition. Figure 5b–d show the microstructure of the samples AF₅, AF₁₀, and AF₁₅, respectively. The addition of fly ash to A354 alloy modified the morphology of the eutectic Si phase, as observed in Figure 5b–d. EDS analysis and the elemental mapping of the as-cast A354 alloy are depicted in Figure 6a,b. Figure 5b,c shows the SEM micrograph of the A354 alloy reinforced with 5 wt% and 10 wt% of fly ash reinforcement. It can be seen from the figure that the proper distribution of fly ash is observed in the A354 matrix reinforced with 5 wt% of fly ash. It can also be seen from the figure that the distribution of fly ash particles is, in addition, the same for AF₅ and AF₁₀. These results show the successful fabrication of the metal matrix composite. However, Figure 5c depicts the SEM image of A354 alloy reinforced with 15 wt% fly ash particles (AF₁₅).

It can be observed from Figure 5d that the fly ash particles are in the state of an agglomerated condition within the matrix. Hence, non-homogeneously distributed fly ash particles in this developed MMC would be detrimental to the mechanical behavior [20]. The metal matrix composite can achieve increased mechanical properties when the reinforcement particles are embedded and homogeneously distributed within the matrix. Figure 7 shows the energy-dispersive spectroscopy (EDS) analysis and elemental map spectrum (Figure 8) of all the developed composites to confirm the existence of all elements associated with A354 and its composites. To ensure the presence of alloying elements in A354 alloy, optical arc spectrometry (Foundrymaster, Germany) was used. Five samples were tested, and the average value was calculated. The composition of the A354 alloy is depicted in Table 1. Furthermore, based on the morphological analysis, there was no sign of porosity or defect in the cast samples.

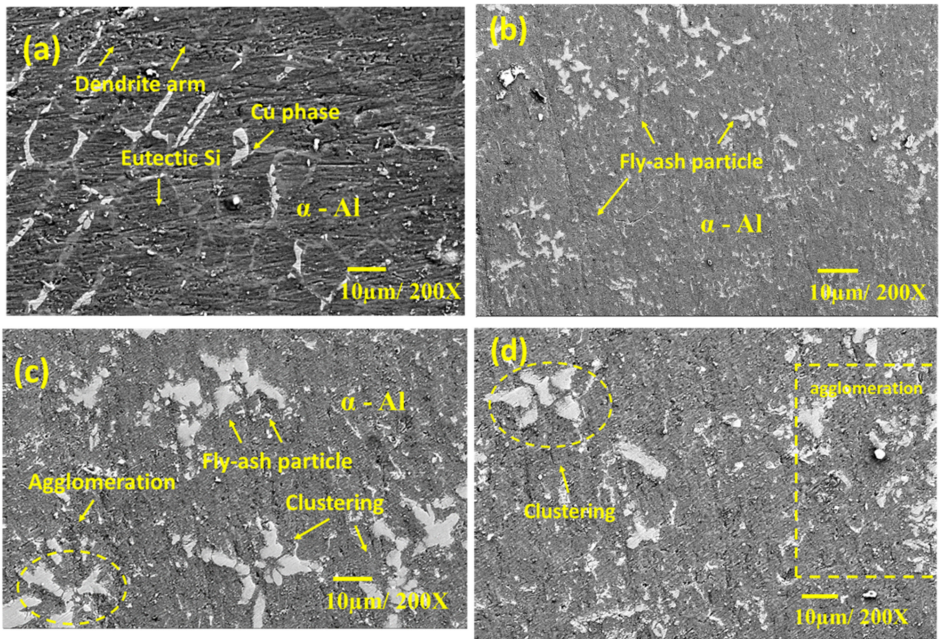


Figure 5. SEM image of the alloy A354 (a) as-cast condition (b) 5 wt% fly ash (c) 10 wt% fly ash (d) 15 wt% fly ash.

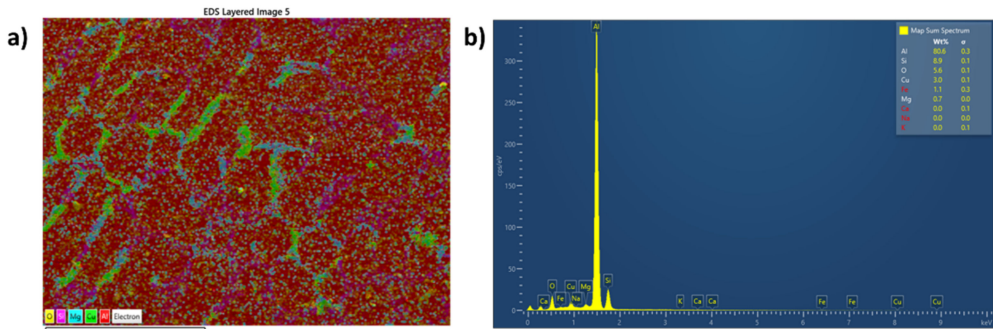


Figure 6. (a) Map spectrum (b) EDS of A354 alloy in the as-cast condition.

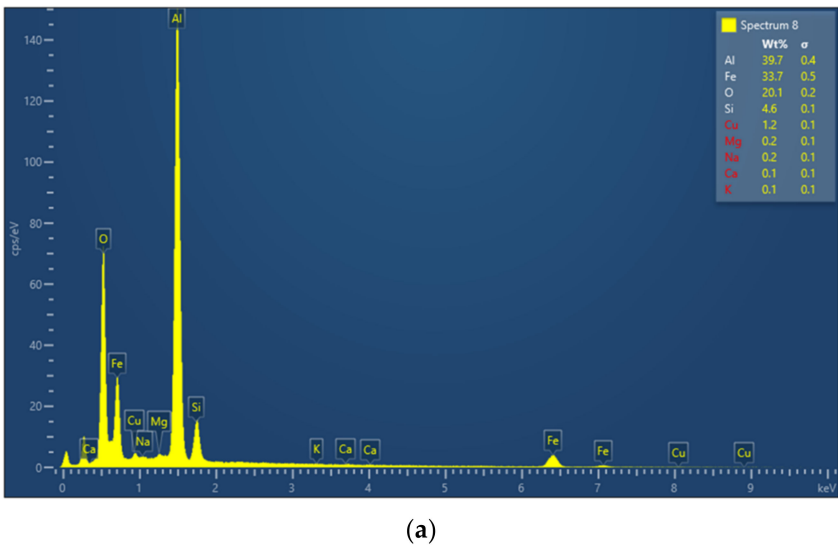
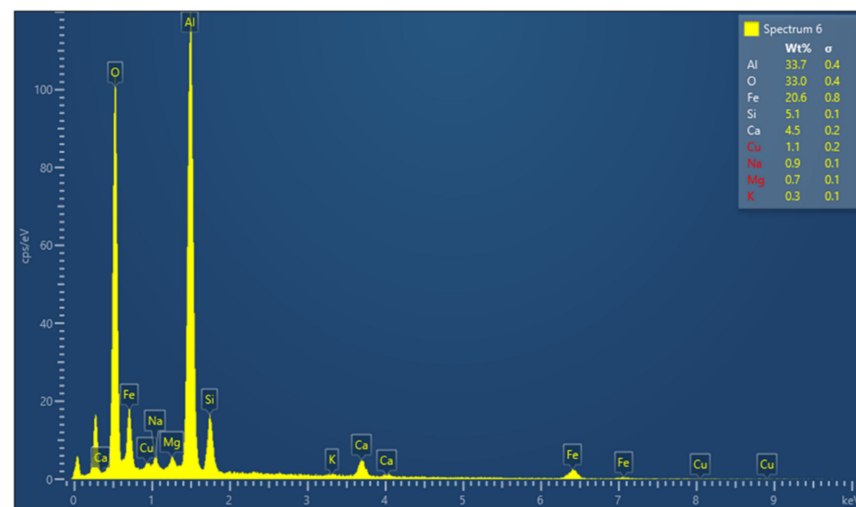
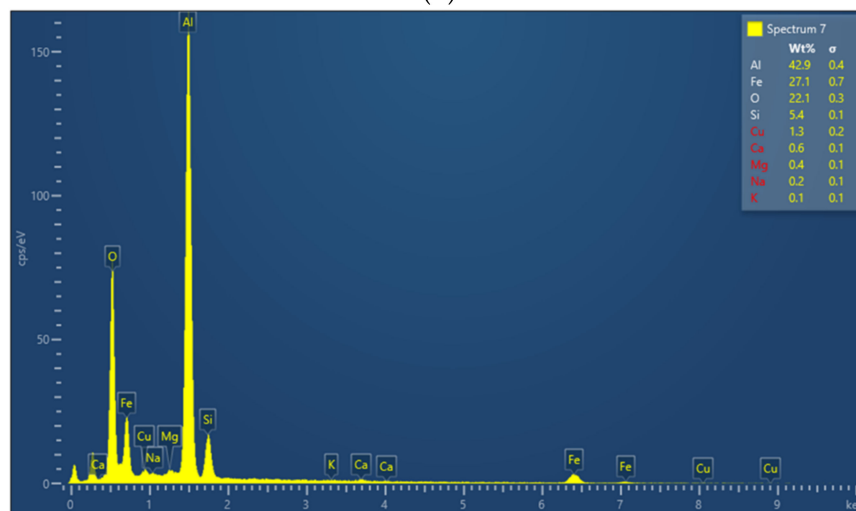


Figure 7. Cont.



(b)



(c)

Figure 7. EDS analysis of (a) AF₅; (b) AF₁₀; (c) AF₁₅.

3.1.1. XRD Analysis

X-ray diffraction on the A354 and its composite was conducted to determine the presence of fly ash and any intermediate phases. Figure 9 shows the XRD pattern of the fabricated samples in which the A354 alloy specimen showed α -Al and eutectic Si, whereas stir-cast A354/fly ash composite specimens exhibited α -Al, eutectic Si, and Quartz/mullite (fly ash) peaks. The formation of extra peaks is attributed to strengthening the fly ash content in the A354 matrix. The XRD results clearly illustrate the successful fabrication of A354 and its composites.

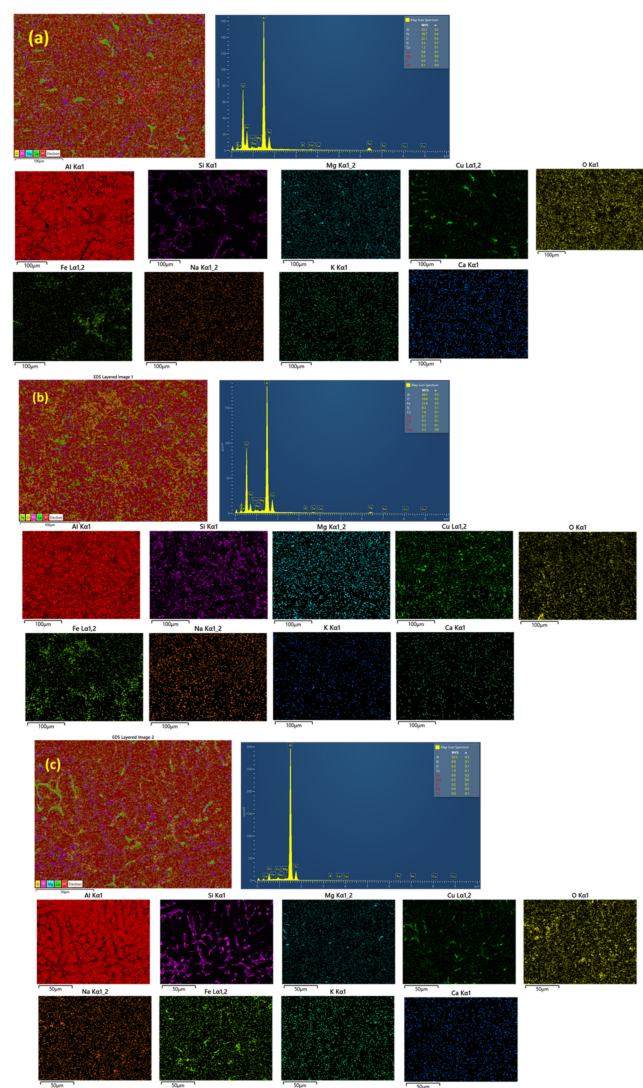


Figure 8. Elemental map spectrum of (a) AF₅; (b) AF₁₀; (c) AF₁₅.

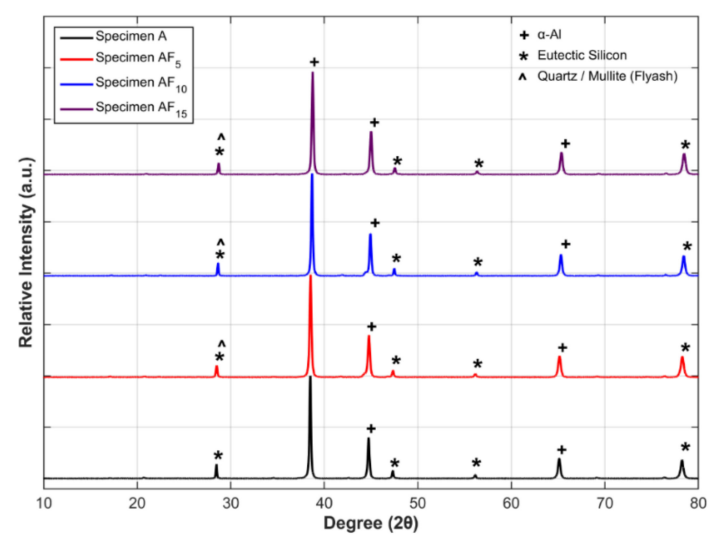


Figure 9. XRD pattern of the base alloy and metal matrix composite material developed.

3.1.2. TEM Analysis for the Sample AF₅

High-resolution transmission electron microscope images of the AF₅ composite specimen are shown in Figures 10 and 11. The bright-field image depicted the α -Al phase (bright region), eutectic Si (grey region), and submicron fly ash particle (dark region), as shown in Figure 10a. The dark-field image is shown in Figure 10b, contrasted with fly ash (bright region) and eutectic Si (grey region). TEM analysis confirmed the presence of submicron and nano-level eutectic Si in the fabricated composite. Figure 11a–d shows a few dislocations features adjacent to fly ash particles, apart from the fly ash particles and the eutectic Si phase. Furthermore, the authors observed characteristic partial dislocations in Figure 11c. The TEM observation corroborates ceramic particle clustering for the sample AF₅.

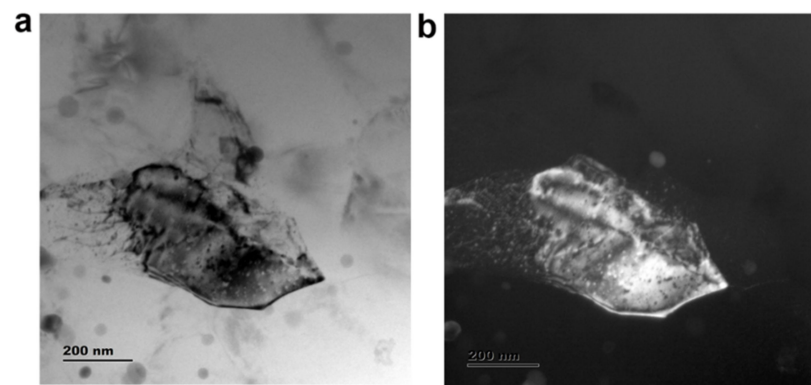


Figure 10. TEM image showing (a) bright-field image; (b) dark-field image of sample AF₅.

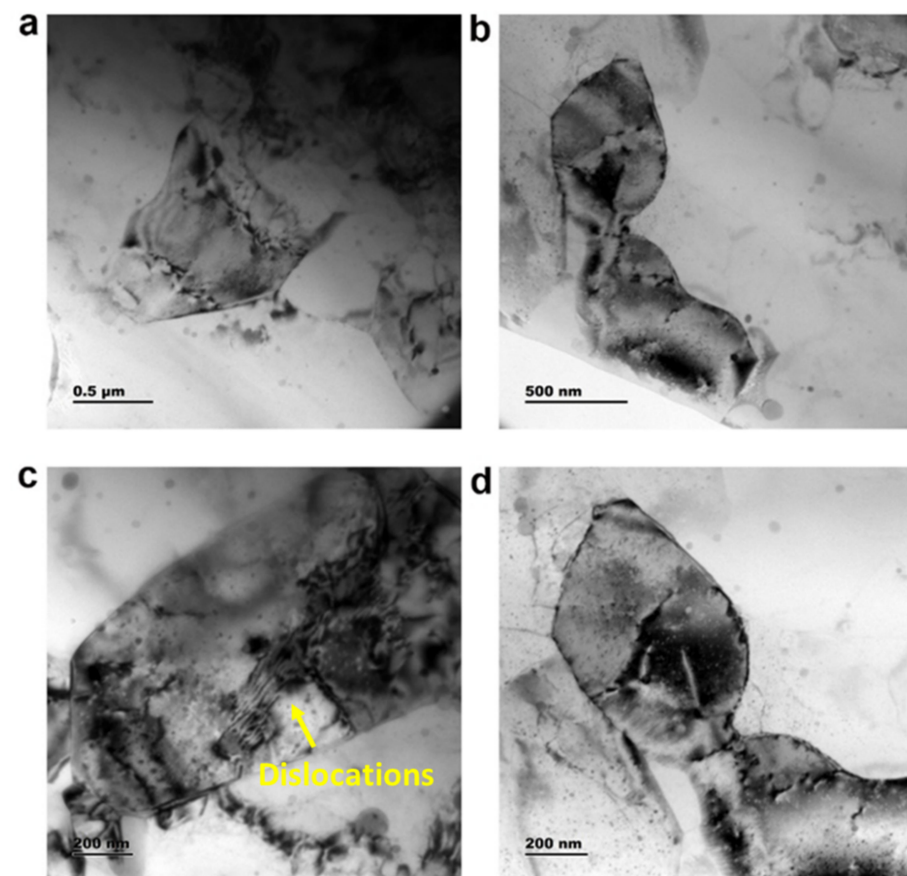


Figure 11. TEM image of the sample AF₅. (a) Refined grains; (b) Sub-micron intermetallic phase; (c) Dislocation features; (d) High magnification image of sub-micron intermetallic phase.

3.2. Mechanical Behavior of A354 Alloy and Stir Cast MMC

The mechanical characteristics were conducted using Vickers hardness, ultimate tensile strength, yield strength, and ductility (Table 5). With reinforcing varying wt% of fly ash particles, the resulting value of tensile strength, hardness, and yield strength increases from the baseline alloy to MMC reinforced with 5 wt% of fly ash and further decreases to 10, 15 wt% fly ash-reinforced MMC. Figure 11 shows the average Vickers hardness values for all samples with error bars. The resulting measured average hardness was found to be: sample A = 143 ± 5.1 HV, AF₅ = 170 ± 5.6 HV, AF₁₀ = 164 ± 5.5 HV, AF₁₅ = 128 ± 5.6 HV, respectively. Sample AF₅ was shown to be around 18.8%, 3.6%, and 32.8% higher than samples A, AF₁₀, and AF₁₅. Vickers hardness improved due to the proper mixing and embedding of fly ash particles with the A354 matrix (Figure 12) [1].

Table 5. Mechanical properties of baseline alloy and stir-cast A354 reinforced with fly ash particles.

Samples	Hardness (HV _{0.1})	Ultimate Tensile Strength (MPa)	Yield Strength (MPa)	Ductility (%)
A	143 ± 5.1	173 ± 2.1	104 ± 3.2	8.5 ± 0.25
AF ₅	170 ± 5.6	216 ± 2.3	130 ± 2.2	6.5 ± 0.43
AF ₁₀	164 ± 5.5	188 ± 2.2	113 ± 3.2	5.7 ± 0.38
AF ₁₅	128 ± 5.6	181 ± 2.1	109 ± 2.1	4.9 ± 0.27

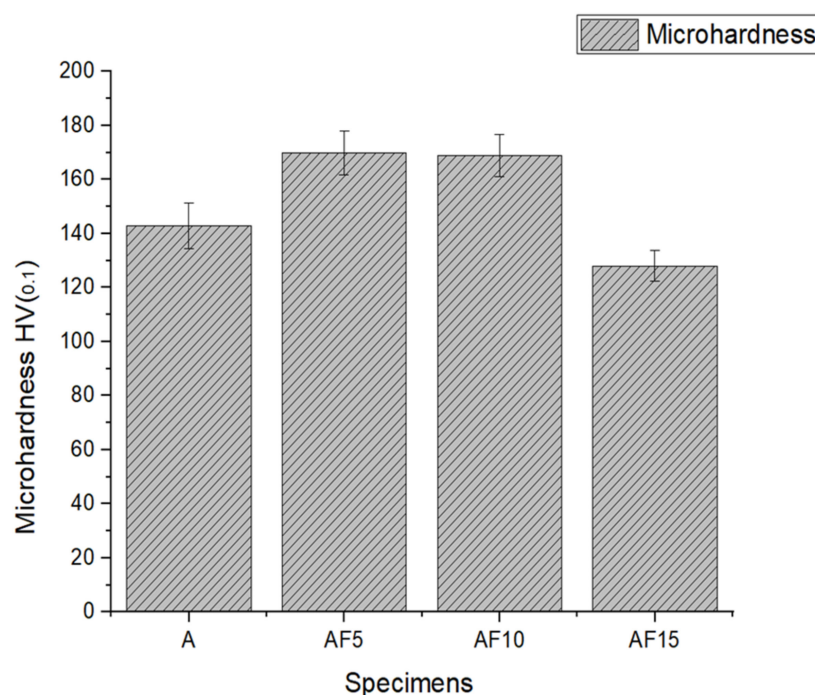


Figure 12. Microhardness of A354 and its composites.

Furthermore, a lower fly ash content could have led to more grain refinement with dislocations and efficient bonding of fly ash with the A354 matrix, which had resulted in an increase in the hardness value for sample AF₅ (Table 4). In addition, the applied load is transformed into the reinforcing particle [2,37–41]. Reinforcing particles withstand the load and geometrically constrain the plastic deformation, which generates the dislocation required to support the load. These dislocations promote the dislocation density of the MMC, causing dislocation hindering. Therefore, the hardness of the MMC increased from the baseline alloy (sample A) to sample AF₅. The hardness value improved due to the dispersion hardening and Orowan strengthening. In addition, more resistance against plastic deformation was anticipated from the fly ash particle because of the hard surface

area, resulting in the fabrication of pore-free surfaces. The presence of agglomeration caused by a nonuniform mix of the reinforcement particles on the A354 matrix led to a lower hardness value in 10 wt%, and 15 wt% fly ash reinforced MMC (Figure 5c,d).

Figure 13 shows the average tensile and yield strength for fabricated alloy and MMCs. The average UTS values, yield strength, and standard deviation for the baseline alloy and MMCs are depicted in Table 5. The A354 alloy showed an ultimate tensile strength of 173 MPa and yield strength of 104 MPa, and the alloy reinforced with varying wt% of fly ash possesses higher UTS and yield strength values than the base alloy. The highest UTS was shown by sample AF₅ (216 MPa), which is 24.8% higher than the value indicated by sample A (173 MPa), 14.9% higher than sample AF₁₀ (188 MPa), and 19.3% higher than sample AF₁₅ (181 MPa). Likewise, the higher yield strength was also shown by sample AF₅ (130 MPa), which is 25% more than the value indicated by sample A, 15% more than sample AF₁₀, and 19.26% more than the alloy reinforced with sample AF₁₅. Compared with sample A, the improved bonding strength of the reinforcement particle with the alloy improves both the composites' UTS and yield strength. Moreover, the increase in value of UTS and YS from sample A to sample AF₅, AF₁₀, and AF₁₅ is attributed to the dispersion-hardening mechanism. The transfer of tensile load from the alloy to reinforcement particles with higher load capacity delays fracture occurrence in the specimens. The high wettability of fly ash particles in sample AF₅ enables load transmission and distribution from matrix to reinforcement. Particulate reinforcements have a high elastic modulus, which decreases strain to failure. The increase in tensile strength is attributed to the rise in the volume fraction of reinforcements in the sample AF₅ compared to the unreinforced alloy.

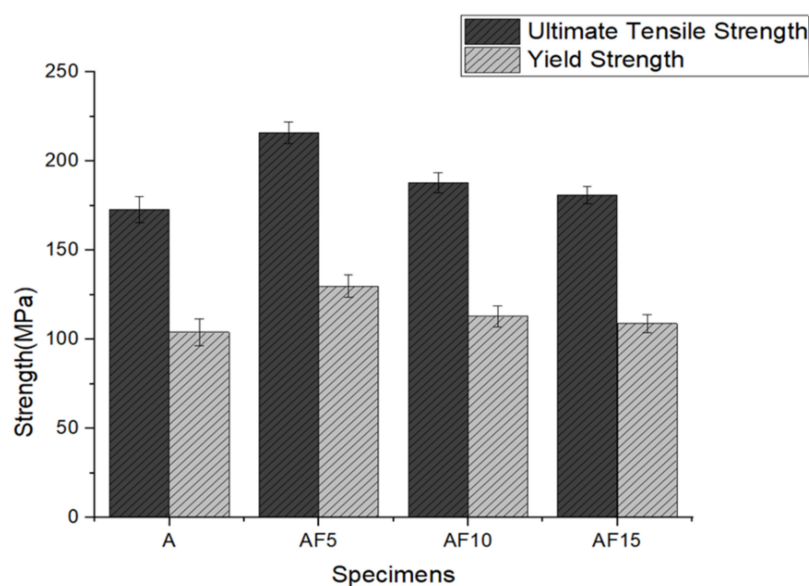


Figure 13. Tensile and yield strength of A354 and its composites.

Consequently, crack formation and propagation can be minimized. The grain size and particle-dispersion capacity determine the yield stress. The low values for the AF₁₀ and AF₁₅ are due to the agglomeration and clustering caused by the nonuniform mix of the reinforcement particles with the A354 matrix.

3.3. Ductility (Percentage Elongation)

Figure 14 shows the average ductility value of Al-Si-Mg-Cu and its composites. It can be noted from the figure that the average ductility of the base material was 8.5%. It was observed that an increase in the concentration of fly ash decreased the ductility of the specimens. The lowest ductility of 4.9% was observed in specimens with 15 wt% fly ash. The decrease in ductility could be correlated to the concentration of reinforcement particles. Though reinforcement particles augmented the hardness and strength of the material, their

inability to yield induced brittle tendency in the specimens. The clustered particles acted as crack initiation sites at a high concentration of fly ash particles and promoted crack growth. Hence, the samples with a high concentration of fly ash had characteristic ductile and brittle features (primary). The results align with the earlier research work [42–44].

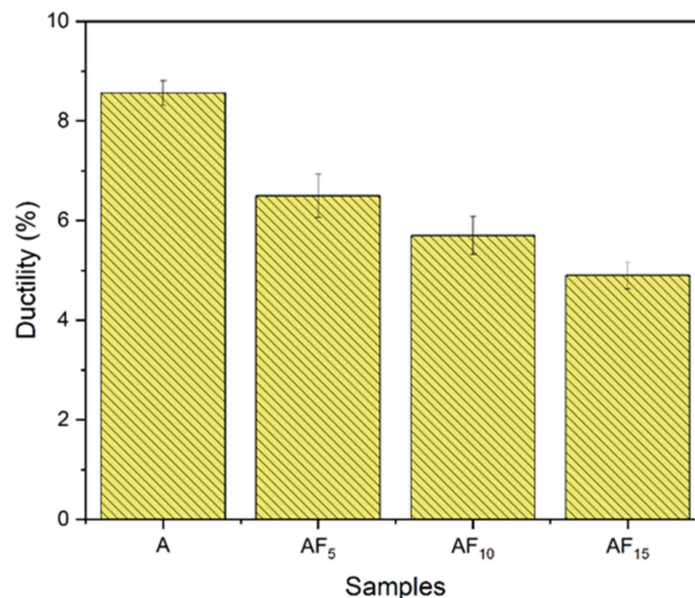


Figure 14. Percentage elongation value of A354 and its composites.

3.4. Fractography

The fractography of the A354 in the as-cast condition (Figure 15a) exhibited plastic deformation characteristics (marked as A) and cracks (marked as B). However, characteristic equiaxed dimples indicating the ductile nature of the specimen were not observed. The specimens exhibited more brittle fracture features with an increase in fly ash concentration. Typical fractography of the samples AF₅, AF₁₀, and AF₁₅ are shown in Figure 15b–d, respectively. The AF₅ sample exhibited cambers (marked C) and dimples (marked D), indicative of ductility. Moreover, a larger plastic deformation zone was observed in the specimen AF₅ than in the A354 alloy. In addition, shear dimples and a particle pullout region (large crater) were observed. The AF₁₀ specimen exhibited a higher fraction of facets (marked F) and cracks (marked as E) on the fractured surface. The grain facets appear dark, clean, and without any striation marks. The features confirm the intergranular rupture of the specimen during the tensile test. The fractograph of the specimen AF₁₅ exhibited typical transcrystalline cleavage, with an array of river patterns decorating the large facets. Furthermore, shear steps and cleavage steps were more prominent in the specimen. Therefore, the AF₁₅ specimen had fractured predominantly by a brittle fracture mechanism. The presence of a more significant fraction of hard-phase fly ash (FA) particles increased hardness (or decreased hardness attributed to FA agglomeration), decreased tensile strength, and promoted brittle fracture. The fractograph substantiates the fracture transition from a combined ductile-brittle mechanism towards a predominantly brittle mechanism, with an increase in FA composition.

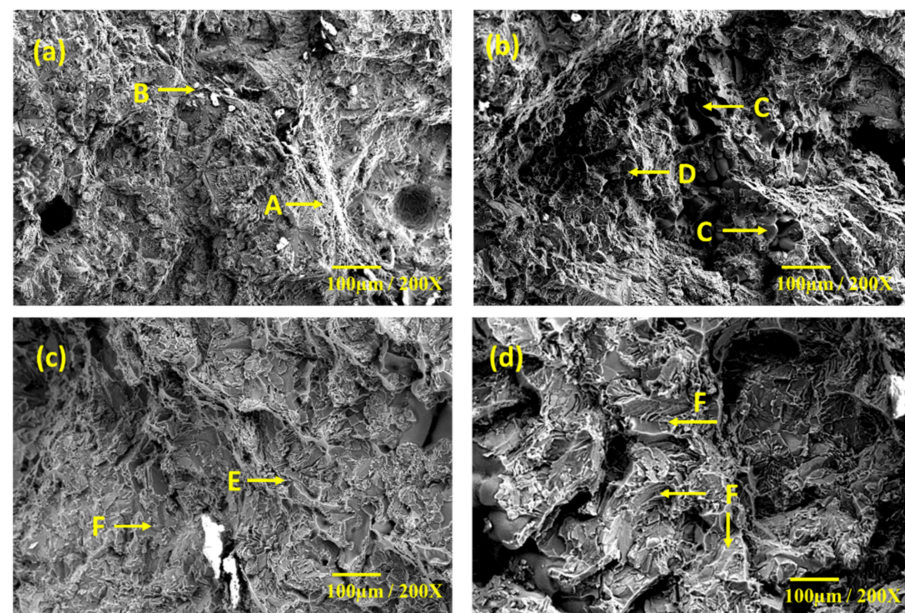


Figure 15. Fractography of A354 alloy. (a) As-cast condition; (b) 5 wt% fly ash; (c) 10 wt% fly ash; (d) 15 wt% fly ash.

4. Conclusions

A354 alloy-based composite was successfully fabricated via stir-casting technique with varied weight compositions of fly ash (5%, 10%, and 15%). The microstructural evolution, microhardness, tensile properties, and fracture mechanism of the as-cast and fabricated composite samples were tested. The results demonstrated the following:

1. The stir-casting technique fabricated sound castings of A354 alloy and composites.
2. The microstructure of the composite specimen AF₅ exhibited a homogeneous dispersion of FA particles and a fragmented eutectic Si phase. However, AF₁₀ and AF₁₅ specimens pronounced a greater agglomeration of FA particles. Furthermore, the AF₁₅ sample had preferential segregation of eutectic Si and FA particles along the grain boundaries.
3. The refined microstructure increased the microhardness (~20%), tensile strength (~25%), and yield strength (~25%) of the composite specimen AF₅ than the A354 alloy. The specimens AF₁₀ and AF₁₅ had diminished mechanical properties because of heterogeneous microstructure.
4. With an increase in the concentration of FA particles, the fracture mode transformed from a combined ductile-brittle mechanism to a predominantly brittle mechanism in the specimens.

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References

1. Kuchariková, L.; Tillová, E.; Bokuvka, O. Recycling and properties of recycled aluminium alloys used in the transportation industry. *Transp. Probl.* **2017**, *11*, 117–122. [\[CrossRef\]](#)
2. Hirsch, J.R. Recent development in aluminium for automotive applications. *Trans. Nonferr. Met. Soc. China* **2014**, *24*, 1995–2002. [\[CrossRef\]](#)
3. Henriksson, F.; Johansen, K. On Material Substitution in Automotive BIWs—From Steel to Aluminum Body Sides. *Procedia CIRP* **2016**, *50*, 683–688. [\[CrossRef\]](#)
4. Pana, G.M.; Grigorie, L.D. The Issue of Car Body Manufacture in Unibody Aluminum Alloy Design. *Appl. Mech. Mater.* **2018**, *880*, 183–188. [\[CrossRef\]](#)
5. Padmanaban, D.A.; Kurien, G. Silumins: The Automotive Alloys. *Adv. Mater. Process.* **2012**, *170*, 28–30.
6. Camicia, G.; Timelli, G. Grain refinement of gravity die cast secondary AlSi7Cu3Mg alloys for automotive cylinder heads. *Trans. Nonferr. Met. Soc. China* **2016**, *26*, 1211–1221. [\[CrossRef\]](#)
7. Molina, R.; Amalberto, P.; Rosso, M. Mechanical characterization of aluminium alloys for high temperature applications Part 2: Al-Cu, Al-Mg alloys. *Metall. Sci. Technol.* **2011**, *29*, 5–13.
8. Fan, K.L.; He, G.; Liu, X.; Liu, B.; She, M.; Yuan, Y.; Yang, Y.; Lu, Q. Tensile and fatigue properties of gravity casting aluminum alloys for engine cylinder heads. *Mater. Sci. Eng. A* **2013**, *586*, 78–85. [\[CrossRef\]](#)
9. Molina, R.; Amalberto, P.; Rosso, M. Mechanical characterization of aluminium alloys for high temperature applications Part 1: Al-Si-Cu alloys. *Metall. Sci. Technol.* **2011**, *29*, 5–15.
10. Kaufman, J.G.; Rooy, E.L. *Casting Properties, Processes, and Application*; ASM International: Materials Park, OH, USA, 2004; pp. 27–31.
11. Merchán, M.; Egizabal, P.; De Cortazar, M.G.; Irazustabarrena, A.; Galarraga, H. Development of an Innovative Low Pressure Die Casting Process for Aluminum Powertrain and Structural Components. *Adv. Eng. Mater.* **2018**, *21*, 1800105. [\[CrossRef\]](#)
12. Castella, C. Self Hardening Aluminum Alloys for Automotive Applications. Ph.D. Thesis, Politecnico di Torino, Turin, Italy, 2015.
13. Köhler, E.; Klimesch, C.; Bechtle, S.; Stanchev, S. Cylinder head production with gravity die casting. *MTZ Worldw.* **2010**, *71*, 38–41. [\[CrossRef\]](#)
14. Shankar, K.V.; Sellamuthu, R. Determination of optimum aging temperature and time, mechanical and wear properties for Cu-9Ni-6Sn spinodal bronze alloy cast using permanent mould. *Int. J. Mater. Eng. Innov.* **2017**, *8*, 27–38. [\[CrossRef\]](#)
15. Sathishkumar, P.; Deepakaravind, V.; Gopal, P.; Azhagiri, P. Analysis the mechanical properties and material characterization on magnesium metal matrix nano composites through stir casting process. *Mater. Today Proc.* **2021**, *46*, 7436–7441. [\[CrossRef\]](#)
16. Nanjan, S.; Muralia, J.G. Analysing the mechanical properties and corrosion phenomenon of reinforced metal matrix composite. *Materials Res.* **2020**, *23*, e20190681. [\[CrossRef\]](#)
17. Alizadeh, A.; Khayami, A.; Karamouz, M.; Hajizamani, M. Mechanical properties and wear behavior of Al5083 matrix composites reinforced with high amounts of SiC particles fabricated by combined stir casting and squeeze casting; A comparative study. *Ceramics Int.* **2022**, *48*, 179–189. [\[CrossRef\]](#)
18. Ramamoorthi, R.; Hillary, J.J.M.; Sundaramoorthy, R.; Joseph, J.D.J.; Kalidas, K.; Manickaraj, K. Influence of stir casting route process parameters in fabrication of aluminium matrix composites—A review. *Mater. Today Proc.* **2020**, *45*, 6660–6664. [\[CrossRef\]](#)
19. Mohana Rao, D.; Bandam, B.R. Preparation and Characterization of Al-Fly Ash Metal Matrix Composite by Stir Casting Method. *Int. J. Innov. Sci. Mod. Eng.* **2014**, *3*, 1–5.
20. De Oliveira, L.B.; de Azevedo, A.R.G.; Marvila, M.T.; Pereira, E.C.; Fediuk, R.; Vieira, C.M.F. Durability of geopolymers with industrial waste. *Case Stud. Constr. Mater.* **2022**, *16*, e00839. [\[CrossRef\]](#)
21. Zhou, H.; Bhattarai, R.; Li, Y.; Si, B.; Dong, X.; Wang, T.; Yao, Z. Towards sustainable coal industry: Turning coal bottom ash into wealth. *Sci. Total Environ.* **2022**, *804*, 149985. [\[CrossRef\]](#)
22. Zhao, H.; Zhou, F.; Evelina, L.M.A.; Liu, J.; Zhou, Y. A review on the industrial solid waste application in pelletizing additives: Composition, mechanism and process characteristics. *J. Hazard. Mater.* **2022**, *423*, 127056. [\[CrossRef\]](#)
23. Sharma, D.K.; Badheka, V.; Patel, V.; Upadhyay, G. Recent developments in hybrid surface metal matrix composites produced by friction stir processing: A review. *J. Tribol.* **2021**, *143*, 1–58. [\[CrossRef\]](#)
24. Ravikumar, K.S.; Ghanaraja, S.; Ramesh, M.R. Effect of milling on the hardness and wear behaviour of cast Al6061 reinforced with Al₂O₃ nanoparticles. *J. Bio Tribo Corros.* **2022**, *8*, 50801. [\[CrossRef\]](#)
25. Révész, Á.; Gajdics, M. Improved h-storage performance of novel mg-based nanocomposites prepared by high-energy ball milling: A review. *Energies* **2021**, *14*, 6400. [\[CrossRef\]](#)
26. Sathishkumar, A.; Soundararajan, R.; Siva Krishna, S.; Ranjan Prabakaran, B.R. *Assessment of Tribological Characteristics of Stir Cum Squeeze Cast AlSi7Mg with x wt % flyash and 3wt % MoS₂ Hybrid Composites*; SAE Technical Paper; SAE International: Warrendale, PA, USA, 2021; p. 173236. [\[CrossRef\]](#)
27. Sharma, V.K.; Singh, R.C.; Chaudhary, R. Effect of fly ash and graphite addition on the tribological behavior of aluminium composites. *Surf. Topogr. Metrol. Prop.* **2021**, *9*, 25027. [\[CrossRef\]](#)
28. Kumar, A.; Goyal, K.K.; Bhardwaj, A.; Sharma, N. Development and characterization of AA2024/SiC/Gr/Fly ash hybrid composite. *J. Phys. Conf. Ser.* **2021**, *1854*, 12022. [\[CrossRef\]](#)
29. He, F.; Han, Q.; Chen, Y.C.; Xu, C.; Shao, L. Study on mechanical properties of Al metal matrix nanocomposites processed using ultrasonic vibration. *Proc. ASME Int. Manuf. Sci. Eng. Conf.* **2009**, *2*, 285–288. [\[CrossRef\]](#)

30. Javidani, M.; Larouche, D. Application of cast Al–Si alloys in internal combustion engine components. *Int. Mater. Rev.* **2014**, *59*, 132–158. [[CrossRef](#)]
31. Aybarc, U.; Dispinar, D.; Seydibeyoglu, M.O. Aluminum Metal Matrix Composites with SiC, Al₂O₃ and Graphene–Review. *Arch Foundry Eng.* **2018**, *2*, 5–10.
32. Li, R.; Pan, Z.; Zeng, Q.; Ye, X. Influence of the Interface of Carbon Nanotube-Reinforced Aluminum Matrix Composites on the Mechanical Properties—A Review. *Arch Foundry Eng.* **2022**, *1*, 23–36.
33. Nemutlu, B.; Kahraman, O.; Demirel, K.B.; Erkul, I.; Cicek, M.; Sahin, H.; Dizdar, K.C.; Dispinar, D. Processing of Boron Nitride Nanotubes Reinforced Aluminum Matrix Composite. *Arch Foundry Eng.* **2022**; *accepted*. [[CrossRef](#)]
34. Lakshmikanthan, A.; Mahesh, V.; Prabhu, R.T.; Patel, M.G.C.; Bontha, S. Free Vibration Analysis of A357 Alloy Reinforced with Dual Particle Size Silicon Carbide Metal Matrix Composite Plates Using Finite Element Method. *Arch. Foundry Eng.* **2021**, *1*, 101–112.
35. Chaudhury, S.K.; Apelian, D.; Meyer, P.; Massinon, D.; Morichon, J. Fatigue Performance of Fluidized Bed Heat Treated 319 Alloy Diesel Cylinder Heads. *Met. Mater. Trans. A* **2015**, *46*, 3015–3027. [[CrossRef](#)]
36. Torres, R.; Esparza, J.; Velasco, E.; Garcia-Luna, S.; Colás, R. Characterisation of an aluminium engine block. *Int. J. Microstruct. Mater. Prop.* **2006**, *1*, 129. [[CrossRef](#)]
37. Kumar, L.; Jang, J.C.; Yu, H.; Shin, K.S. Effects of Cr and Ti Addition on Mechanical Properties and Thermal Conductivity of Al–7Si–3Mg Die-Casting Alloys. *Met. Mater. Int.* **2022**, 1–11. [[CrossRef](#)]
38. Wang, S.; Cui, X.; Xiao, A.; Zhang, L.; Zhao, P. Mechanical Properties and Microstructure Evolution of Pre-stretched Aluminum Alloy After Induced Electro-Pulsing Treatment. *Met. Mater. Int.* **2022**, 1–11. [[CrossRef](#)]
39. Kim, S.-B.; Jung, J.-G.; Cho, Y.-H.; Kim, S.-H.; Euh, K.; Lee, J.-M. Effect of Ultrasonic Melt Treatment on Solidification Microstructure of Al–5Ti–1B Alloy Containing Numerous Inoculant Particles. *Met. Mater. Int.* **2022**, *28*, 1549–1560. [[CrossRef](#)]
40. Kim, S.B.; Kim, D.H.; Kang, J.W. Role of Ultrasonic Melt Treatment in the Improvement of Corrosion Resistance of Al–7Si–0.4Mg Cast Alloy. *Met. Mater. Int.* **2022**; *in press*. [[CrossRef](#)]
41. Biyikli, M.; Karagoz, T.; Calli, M.; Koo, T.M.; Lee, Y.S.; Lee, J.Y.; Cho, Y.H.; Lee, J.M. Single Track Geometry Prediction of Laser Metal Deposited 316L–Si Via Multi-Physics Modelling and Regression Analysis with Experimental Validation. *Met. Mater. Int.* **2022**; *in press*. [[CrossRef](#)]
42. Ganesh, M.R.S.; Reghunath, N.J.; Levin, M.; Prasad, A.; Doondi, S.; Shankar, K.V. Strontium in Al–Si–Mg Alloy: A Review. *Met. Mater. Int.* **2022**, *28*, 1–40. [[CrossRef](#)]
43. Shankar, K.V.; Manu, K.; Raj, A.N.; Mukund, A.; Nair, A.S.; Nived, S.; Premkumar, A. Solutionising Temperature Influence on the Morphological and Mechanical Characteristics of Al–Si–Mg–Ni Hypoeutectic Alloys. *J. Inst. Eng. India Ser. D* **2021**, *102*, 131–148. [[CrossRef](#)]
44. Shankar, K.V.; Ajay Ghosh, K.J.; Sudhin, C.B.; Pai, A.S.; Biju, A.; Sriram, K.R. Investigating the significance of SiC particle size on the mechanical properties of cu-6Sn–SiC composite. *Int. J. Mater. Eng. Innov.* **2020**, *11*, 323–337. [[CrossRef](#)]