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ARTICLE

Study on Mechanical Behaviour of Al-15Si-10Zn/x ZrO₂ Composites Synthesized by Stir Cast Method

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ABSTRACT

In this research, the microstructure and mechanical properties of Al-15Si-10Zn-xZrO₂ (x = 5, 7 and 10 wt.%) composites synthesized by the stir-casting process have been investigated. The matrix and composite samples were characterized using an X-ray diffraction test (XRD) and their microstructures were studied using optical microscopy and scanning electron microscopy (SEM). The microstructure of different phases was analyzed using energy-dispersive spectroscopy (EDS). The microstructure of the Al-15Si-10Zn/ZrO₂ composites consisted of coarse primary Si, needle-like eutectic Si, and Zn-rich phases, with ZrO₂ reinforcement particles distributed homogeneously in the composites. The micro-hardness and tensile properties of the matrix and composites were determined at room temperature. The results show significant improvements in the micro-hardness and tensile strength of the composites compared to the matrix alloy. The micro-hardness of the stir-cast composites increased by 24%, 44%, and 58% in the 5, 7, and 10 wt% ZrO₂ reinforcement composites, respectively. The ultimate tensile strength (UTS) of the 5, 7, and 10 wt% ZrO₂ composites increased by 17%, 30%, and 44%, respectively, compared to the matrix alloy. The increased content of ZrO₂ resulted in an increase in hardness, ultimate tensile and yield strengths and a decrease in ductility.

Keywords: Al-Si-Zn/ZrO₂ composites; Stir-casting; Microstructure; Hardness; Yield strength; Tensile strength; Ductility

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1. Introduction

The demand for high-strength and light materials is increasing due to rapid developments in the automobile and aerospace. Binary aluminum-silicon (Al-Si) alloys with more than 13% Si are the leading commercial cast alloys due to their high wear resistance, high strength-to-weight ratio, low thermal expansion coefficient, good machinability, and good castability^[1]. Many studies have been carried out on the effect of Si content on the mechanical properties of the hypereutectic Al-Si cast alloys^[2]. It was observed that the hypereutectic range of binary Al-Si alloy depicts poor mechanical properties. This is due to the microstructure of the hypereutectic Al-Si alloy consisting of coarse polygon with sharp-edged Si and needle eutectic Si particles causing matrix instability and stress concentration at the particle edges ^[3]. Aluminum matrix composites (AMCs), whose properties may be customized by the inclusion of particular reinforcements, are becoming more attractive materials for cutting-edge applications in aerospace and automotive^[4]. The alloying of a trace amount of zinc (Zn) to the Al-Si alloy results in a decrease in the eutectic needle size and an improvement in the homogeneity of the matrix ^[5]. Dhakar et al.^[6] reported that the alloying of Zn with the hypereutectic Al-Si alloy results in the formation of high-density precipitates in the microstructure, which significantly results in an increase in tensile strength, wear resistance, and hardness^[7,8].

Alloying Zn to the Al-Si alloy enhances strength but decreases elongation because of the accumulation of coarse eutectic phases in the alloy. Because of this, it is impracticable to only increase Zn concentration to improve the material's overall performance ^[9]. AMCs became a high-performance material for application in the aerospace, automotive, transportation, and aerospace industries due to their higher strength, high elastic modulus, and increased wear resistance over typical base metals ^[10]. The different types of carbides and oxides, such as Si₃N₄, Bn, SiC, TiC, BiC, Al₂O₃, TiB₂, B₄C, and ZrO₂, are being used as reinforcements in aluminum matrix composites ^[10].

Zirconium dioxide (ZrO_2), the crystalline oxide of zirconium, also known as zirconium oxide, is a

ceramic element and is widely used as a reinforcing particle for high-temperature applications because of its high melting point (1850 °C) ZrO₂ particles have extremely good tribological properties, high thermal (16.7 W/m-K) conductivity, good specific heat capacity (0.285 J/g-°C) and corrosion resistance ^[11]. Also, the high fracture toughness of ZrO₂ particles decreases in the catastrophic failure of composites ^[12]. It is worth noting that zirconium oxide does not react with aluminum at lower temperatures, which is important in preventing the growth of unwanted brittle reactions at interfaces. Additionally, using reinforced zirconium oxide in aluminum alloys can reduce the risk of catastrophic failure during service conditions. Zircon-reinforced composites are preferred as these composites exhibit relatively high refractoriness and excellent resistance to abrasion, thermal shock, and chemical attack compared to composites reinforced with other reinforcements ^[13]. Song Xiu-an et al. ^[14] studied the new silicon-reinforced Zn-Al alloy in a large composition range, as the Si phase is modified with sodium salt compounds and rare metal oxides. Results show that the effects of sodium salt compounds and rare metal oxides on the silicon phase promote each other, the mechanical properties of silicon-reinforced Zn-Al alloys are improved, and the selected alloy. K.B. Girisha et al.^[15] found that the composites containing ZrO₂ reinforcement showed improved wear properties and hardness strength. Salimi et al. [16] demonstrated that accumulative roll bonding resulted in excellent ZrO₂ particle distribution in the aluminum matrix, leading to enhanced tensile, hardness, and elongation properties. Hajizamani et al. [17] also observed increased yield strength, ultimate tensile strength, and compressive strength with the addition of Al₂O₃-10% ZrO₂ reinforcement. These findings collectively suggest that the incorporation of ZrO₂ reinforcement in an aluminum matrix composite can enhance its mechanical properties. Muralidharan et al. [18] found that the addition of ZrO₂ particles to AA6061 increased the hardness and tensile strength of the composites. Pandiyarajan^[19] also observed improved hardness and strength in Al6061-grade aluminum alloy composites reinforced with ZrO₂. Kavitha et al. ^[20] investigated

AA6081 composites reinforced with ZrO_2 and found enhanced mechanical properties, including hardness and compressive strength. Overall, these findings suggest that ZrO_2 reinforcement positively impacts the mechanical properties of aluminum matrix composites.

Rathaur et al. ^[21] focus on the mechanical and structural properties of an Al-7075/Si₂N₂/ZrO₂ hybrid composite, showing improved tensile strength, hardness, and wear properties with increasing zirconia reinforcement. Abd-Elaziem et al. [22] investigated Zn-Al matrix composites reinforced with alumina, carbon, or steel fibers, demonstrating reduced creep rate, thermal expansion coefficient, and improved impact toughness. Jin Wang et al. ^[23] investigated the silicon particle-reinforced zinc-based composites fabricated using a gravity casting technique with sodium salt as a modifier. The results show that the fabrication technique of Si particle-reinforced Znbased composites with fine silicon particles distributed uniformly can be obtained, and with the increase in Si content in the alloy, the hardness of the alloy increases, the impact toughness of the alloy decreases, and with the increase of Si content in the alloy, the flowability of the high Al-Zn zinc-based alloy decreases. The stir-casting process is a cost-effective and convenient method for producing Aluminium Matrix Composites $(AMC_s)^{[24]}$. Although there have been numerous studies on the wear behavior of Albased composites, there has been limited research on the mechanical properties of hypereutectic Al-Si-Znbased composites with ZrO2 reinforcement. In this research, the stir-casting process was used to synthesize Al-15Si-10Zn matrix composites reinforced with ZrO_2 particles of varying wt.% (5, 7, and 10%).

The main objective of this work was to study the microstructure and investigate the impact of ZrO_2 content on the hardness, yield strength, ultimate tensile strength and ductility of the Al-15Si-10Zn/xZrO₂ (*x*-5, 7 and 10 wt.%) stir-cast composites and compared with the matrix alloy.

Novelty statement: Numerous research publications were carried out on the mechanical and wear behavior of ZrO_2 reinforced hypoeutectic Al-Si alloy with small content of Zinc matrix composites. The present work aim is to study the mechanical behavior of hypereutectic Al-Si-Zinc based on different quantities of ZrO_2 reinforced stir-casted composites. The findings collectively suggest that hypereutectic Al-Si-Zn based matrix composites can be tailored to exhibit desirable mechanical and thermal properties through the addition of ZrO_2 reinforcement and is the potential material for automobile applications parts like piston, cylinder and piston rings etc.

2. Experimental procedure

2.1 Materials and methods

The base alloy Al-15Si-10Zn was prepared using master alloys in a graphite crucible using electric furnace (Figure 1a). The chemical composition of the matrix alloy is displayed in Table 1. The matrix alloy was then remelted in an electrical furnace at a temperature of 850 °C and treated with hexachloroethane degassifier tablets and scum powder to remove gases from the melt. After skimming, pre-heated ZrO₂ reinforcement powder with a size ranging between 20 to 50 µm and different wt.% (5, 7, and 10%) was added to the melt. The chemical composition of the ZrO₂ reinforcement and composite materiaals is shown in Tables 2 and 3. The mixture was stirred by a mechanical stirrer (Figure 1b) at a speed of 400 rpm for 15 minutes to ensure an even distribution of the ZrO₂ particles in the matrix alloy. The mixture was then poured into a pre-heated mold (Figure 1c) to prepare casting samples with a diameter of 50 mm and a length of 250 mm (Figure 1d). The standard size and image of the tensile test sample (as per ASTM standards) are shown in Figure 2.

2.2 Metallographic examination

In order to examine the microstructure of the matrix and composites, a sample was prepared using standard grinding and polishing techniques. The surface of the sample was then treated with Keller's reagent. The microstructure was analyzed using an optical microscope (Zeiss) and a scanning electron

Table 1. Chemical composition of Al-5Si-10Zn alloy Metal matrix.									
Elements	Si	Zn	Fe		Mn	Mg	Sn		Al
Wt.%	15	10	0.1	12	0.009	0.007	0.008		Bal
Table 2. Chemical composition of ZrO2 reinforced powder (wt %).									
Constituent		SiO ₂		TiO ₂		Fe ₂ O ₃		ZrO ₂	
Wt.%		10		0.112		0.009		Bal	
Table 3. Chemical composition of Al-5Si-10Zn-x (x-5, 7, 10) ZrO ₂ composites.									
Composite Elements wt.%									
		Si	Zn	ZrO_2	Fe	Mn	Mg	Sn	Al
Al-15Si-10Zi	n-5ZrO ₂	14.28	10.1	4.95	0.112	0.002	0.004	0.002	Bal
Al-15Si-10Zi	n-7ZrO ₂	15.12	9.93	7.23	0.141	0.001	0.006	0.002	Bal
Al-15Si-10Zi	n-10ZrO ₂	14.92			0.163	0.002	0.003	0.002	Bal

microscope (Model: JEOL JSM-6480LV). The chemical compositions of the constituents present in different phases were determined using an energy-dispersive X-ray (EDX) micro-analyzer. The SEM was operated at an acceleration voltage of 10-30 kV. The X-ray diffraction analysis was carried out on a pan analytical Philips system (Model: MCRS 2000-3). This system has a Cu-K α anode ($\lambda = 0.154065$ nm) and 482 kW sealed tube operated at 160 kV and 20 mA. The step size and step time are 0.01 and 1° respectively. The observed d-values were compared with corresponding peaks given in JCPDS files.

2.3 Hardness test

The micro hardness of the matrix alloy and composites were measured using a Vickers hardness testing machine (Model: HV-5, Meta-tech) under the load of 250 g and dwell time of 15 s, in accordance with the standard ASTM E92-17, and the average of 5 readings values was reported.

2.4 Tensile test

The standard tensile specimens (Figure 2) of the matrix and composites were tested at room temperature by following the ASTME-8 standard (ASTM, 2000). The typical configuration of a tensile specimen is shown in Figure 2. The tensile test was carried out using universal testing machine (Model: Instron, Strain gauge type, 2630-102) at a cross-head speed of 0.0083 mms⁻¹ and strain rate of 0.05 min⁻¹. The yield strength (YS) was calculated according to the standard 0.2% offset strain. The samples were mounted on two grips and aligned with the load axis. The test data reported was the average of three tests.



Figure 1. (a) Melting furnace, (b) mechanical stirrer, (c) steel mold and cylindrical castings.



Figure 2. (a) Schematic of the tensile test specimen (ASTME8), (b) tensile test specimen.

3. Results and discussion

3.1 XRD analysis

Figure 3 shows the X-ray diffraction pattern of the ZrO₂ reinforcement particulates and composite samples. The XRD pattern Of ZrO₂ powder displayed in **Figure 4a** shows clear diffraction peaks at 20 values of 28.2°, 31.5°, 38.5°, 50.1°, and 59.8°, which correspond to the [-111], [111], [120], [022], and [131] planes, respectively, of monoclinic ZrO₂. On the other hand, **Figure 3b** presents the qualitative X-ray diffraction analysis of the composite material. It can be observed that the peaks correspond to reflections of atomic planes belonging to Al, and Si and reflections of crystalline planes of Zn-rich and ZrO₂ phases. The composite exhibits high-intensity peaks of α -Al and Si and low-intensity peaks of Zn and ZrO₂ phase.



Figure 3. Qualitative analysis of XRD pattern (a) matrix and (b) composite material.

3.2 Microstructure study

Figure 4a displays an optical micrograph of the Al-15Si-10Z matrix alloy. It contains coarse polygonal primary Si, needle eutectic Si particles, and dendrite-shaped Zn-rich phases, which are non-uniformly dispersed in the dendrite Al matrix. The formation of blocks of primary Si and needle-like eutectic Si phase is due to the solidification rate of the Al-Si alloy. It is also observed that dendritic Zn-rich phase clusters exist between the eutectic Si and Al-rich phases. Figures 4b-4d show the optical microstructure of stir cast composites. Its microstructure mainly consists of primarily consists of coarse and nearly spheroidized primary Si, small size needle form of eutectic Si, Zn-rich phases, and uniformly dispersed ZrO₂ particles in the matrix. It is clearly observed that the distributions of the ZrO₂ are uniform over the entire sample.



Figure 4. Optical micrographs (a) Al-15Si-10Zn alloy, (b) Al-15%Si-10%Zn/5ZrO₂, (c) Al-15%Si-%10 Zn-7%ZrO₂, and (d) Al-15%Si-%10 Zn-10%ZrO₂ composites.

The SEM microstructure of the matrix and composites is shown in **Figure 5**. The SEM microstructure of matrix alloy (**Figure 5a**) mainly consists of Al-rich dendrites, interdendritic Zn-rich phase, needle-like eutectic Si, and block-like Si particles. The addition of Zn causes an increase in coarse Si particles and their segregation in the matrix. Furthermore, the shape of eutectic Si particles changed from needle-like to rounded-like form. The SEM micrographs of Al-Al-15Si-10Zn composites with 5, 7, and 10 % ZrO_2 particles are shown in **Figures 5b-5d**. It is observed that the ZrO_2 particles are uniform distribution of ZrO_2 particles throughout the matrix. The agglomerated reinforcement (white contrast) particles can be noticed in the composites having a η -AlZn₂ high content of ZrO_2 . From the microscopic image (**Figure 5d**), it is observed that with increasing ZrO_2 content, the agglomeration of ZrO_2 particles in the matrix.



Figure 5. SEM microstructure of matrix and stir casted composites (a) Al-15i-10Znmatrix, (b)Al-15Si-10Zn 5wt.% ZrO₂, (c) Al-15Si-10Zn-7wt.%ZrO₂, and (d) Al-15Si-10Zn/10wt.% ZrO₂ composite.

SEM micrograph displaying the point of EDS analysis and the corresponding EDS spectrum of the

Al-15Si-10Zn-7wt.%ZrO₂ composite is presented in **Figure 6**. EDS spots 1, 2, and 3 correspond to the primary Si phase, ZrO_2 , α -Al, and Zn-rich phases, respectively. **Table 4** shows the composition of phases that correspond to the points indicated in **Figure 5**.

3.3 Micro-hardness

Table 5 shows the micro-hardness of the base and composites. The composite materials show high hardness compared to the matrix alloy. The hardness of the composites increased with an increase in ZrO_2 content. The composites have 5, 7 and 10 wt.% ZrO₂ reinforcement shows nearly 29%, 35%, and 66.0% respectively. The high hardness values compared to the matrix alloy. The high hardness of composites is due to increasing ZrO₂ reinforcements, which increase the fraction of hard volume of Si and ZrO₂ phases. This restricts localized deformation of the softer matrix under indentation load. Due to the stir-casting method, the composite exhibits a homogeneous distribution of fine, hard Si and Zn-rich phases. This distribution helps to reduce stress concentration and prevent plastic deformation ^[24]. The addition of Zn with Al-Si alloy also helps to retain more solute during solidification, leading to solid



Figure 6. (a) SEM/EDS micrograph and (a) EDS spectrum of Al-15Si-10Zn-7wt.%ZrO₂ composite.

Mass (%)					
EDS Spot	0	Al	Si	Zr	Zn
1	0.32	3.50	92.86	3.33	0.00
2	20.46	0.28	0.35	78.91	0.00
3	0.77	92.75	0.37	0.00	6.11
4	0.12	5.67	0.18	0.00	94.12

Table 4. EDS analysis of Al-15Si-10Zn-7wt.%ZrO₂ composite.

solution strengthening in the matrix. These factors contribute to the superior hardness of the composite. The high hardness in the composite is also due to the large difference in the thermal expansion coefficients of the ZrO_2 particle and the base alloy. As a result, during the solidification process numerous formation of dislocations at the interface between the ZrO_2 particles and the base. This leads to an increase in resistance to plastic deformation, which is a significant resistance to indentation deformation ^[25].

A WOLC OF THE OTHER OF THE OTHE	Table 5.	Vickers	micro-	-hardness	values	of matr	ix and	composites.
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Alloy/composite	Micro-hardness H _v
Matrix	65 ± 3
5% ZrO ₂ composite	88 ± 5
7% ZrO ₂ composite	92 ± 4
10% ZrO ₂ composite	88 ± 5

3.4 Tensile properties

Table 6 displays the UTS, YS, and ductility of the composites. The results reveal that the UTS and YS of the composite increase with the rise in ZrO_2 wt.%. Compared to the base alloy, the UTS of the 5, 7, and 10 wt% ZrO₂ composites rose by 17%, 30%, and 44% respectively. Increasing the ZrO₂ content results in improved UTS and YS, but ductility decreases with an increase in ZrO₂ wt.%. Hard ZrO₂ and Si particles act as load-bearing elements and lead to high UTS. During tensile loading, the Al-15Si-10Zn matrix transfers the load to hard ZrO₂ and Si particles, increasing the fracture resistance in the matrix. The improved tensile strength is also attributed to the strong interfacial bonding between reinforcement particles and the matrix alloy, which is due to the presence of the Zn-rich phase ^[26]. The yield strength of composites increases noticeably and ranges from 128 to 144 MPa with an increase in ZrO₂ content from 5 to 10 wt.%. The low tensile strength in the matrix owing to the presence of the block-like primary Si with sharp edges, eutectic Si in the form of needles, and flake-like Zn-rich intermetallic on create stress concentrations and become the sites where cracks start to form ^[27]. When a fracture occurs in an alloy, it can spread quickly along the eutectic and Zn rich secondary phases. The stir casted composites have a more even distribution of hard ZrO_2 particles, nearly spheroidized primary Si, eutectic Si and Zn rich secondary phases, resulting in higher hardness, UTS and YTS. The presence of the Zn-rich strengthening phase, primary Si eutectic phase, and the uniform dispersed ZrO_2 particles have an obstruction caused by the progress of dislocations when the material is subjected to an external load. The increase in the volume percentage of ZrO_2 particles, which act as stress concentrators, results in a reduction in composite ductility.

Base alloy/Composite	YS (MPa)	UTS (MPa)	Elongation (%)
Base alloy	115 ± 6	144 ± 5	8 ± 1.2
Al-15Si- 10Zn/5wt.%ZrO ₂	127 ± 4	165 ± 4	4.5 ± 0.7
Al-10Zn -5Si/7wt.%ZrO ₂	139 ± 4	187 ± 5	4.1 ± 0.4
Al010Zn- 5Si/10wt.%ZrO ₂	144 ± 7	207 ± 7	3.1 ± 0.34

4. Conclusions

In this study, the hypereutectic Al-15Si-10Zn-ZrO₂ composite with reinforcement varying of (5, 7 and 10 wt.%) ZrO₂ material using stir-cast method has been developed. The effect of Zn influence and variation of ZrO₂ quantity on the hardness and mechanical properties of stir-cast hypereutectic Al-Si-Zn composites were studied and compared to the matrix alloy. The study results are summarized below.

- The Al-15%Si-10Zn matrix alloy has a microstructure that consists of primary Si in blocklike shapes, eutectic Si in needle-like shapes, and non-uniformly distributed Zn-rich phases in the dendrite α-Al matrix. The stir cast composites have a homogeneous distribution of ZrO₂ reinforcement particles in the Al matrix.
- The addition of ZrO₂ to the composite resulted in a significant increase in micro-hardness. Specifically, the composite containing 10 wt.%

ZrO₂ displayed approximately 66% higher hardness compared to the matrix alloy.

- The combination of an Al-Si-Zn alloy and reinforced zirconium oxide produces a material with superior mechanical properties. The ultimate tensile strength of composites containing 5%, 7%, and 10% ZrO₂ increased by 17%, 30%, and 44%, respectively, when compared with the matrix alloy. The increase in ZrO₂ content led to a higher hardness and yield strength, while a decrease in ductility.
- The higher hardness and higher mechanical strength of the hypereutectic Al-Si-Zn/ZrO₂ composite can be useful materials for automobile and aerospace applications.

Author Contributions

Vijaykumar Chavan: Experimental data analysis and drafting.

Dayanand M.Goudar: Literature search, editing, review.

Vinay V. Kuppast: Conceptualization, data curtain.

Rajashekar V.Kurahatti: Editing and supervision. Mangalppa M. Naik: Methodology.

Conflict of Interest

No potential conflict of interest was reported by the author(s).

Data Availability

Not applicable.

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