**MICROSTRUCTURAL ANALYSIS, MICROHARDNESS AND COMPRESSIVE BEHAVIOUR OF DUAL REINFORCED PARTICLES ADC-12 ALLOY COMPOSITE**

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**Abstract:**
The aim of this study was to determine the compressive properties of silicon carbide (SiC) and zircon sand (ZrSiO₄) particulate reinforced with ADC-12 alloy, ADC-12 alloy composite. In this experimental study, SiC and ZrSiO₄ particulates reinforced with ADC-12 alloy composite were manufactured by stir casting methods. Compressive properties of these composite materials were investigated by different weight percentages of dual reinforcement combinations (9+3) %, (6+6) %, (3+9) % wt. silicon carbide (SiC) and zircon sand (ZrSiO₄) respectively. The compressive tests were conducted to determine compressive strength and young’s modulus to investigate the effects of reinforce materials on different combinations of weight percentages. The outcome of the investigations reveals that the tensile strength of composites reinforced by Zircon sand (ZrSiO₄) and silicon carbide particles with a total reinforcement 12% wt, and in this hybrid reinforcement the variations (9+3) %, (6+6) %, (3+9) % were taken in to account for investigating the properties such as density, compressive strength and hardness of the composites synthesized by Stir casting technique, also compared between each other’s. The mechanical properties evaluation reveals variations in hardness and the compressive strength values with the composite combinations. From the experimental studies, the optimum volume fraction of hybrid reinforcement in ADC-12 alloy on the basis of microstructure and mechanical properties it is found that the (6+6) wt.% combination.

**Keywords:**
ADC-12 alloy, reinforcements, stirrs casting, Microstructure, Mechanical properties, SiC and ZrSiO₄ particles.

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1. **INTRODUCTION**

Aluminum alloy matrix composites (MMCs) have emerged as a good quality of materials capable for advanced structural, automotive, electronic, thermal management, and wear applications. MMCs compare to conventional materials provide the specific mechanical properties necessary for elevated and ambient temperature applications. In many cases, the
performance of metal-matrix composites is superior in terms of improved physical, mechanical, and thermal properties (specific strength and modulus, elevated temperature stability, thermal conductivity, and controlled coefficient of thermal expansion). The performance advantages of metal matrix composites are their tailored mechanical, physical, and thermal properties that include low density, high specific strength, high specific modulus, high thermal conductivity, and good fatigue response, control of thermal expansion, and high abrasion and wear resistance. In general, the reduced weight and improved strength and stiffness of the MMCs are achieved by various monolithic matrix materials. However, material liabilities for continuous fiber systems include low transverse and inter-laminar shear strength, foreign object impact damage, mechanical/chemical property incompatibility, and high fiber and processing costs. The ability to transition a metal-matrix composite from an advanced composite material to a cost-effective application for the commercial market involves several factors, including a large material production capacity, reliable static and dynamic properties, cost-effective processing, and a change in design philosophy based on experience and extensive durability evaluation [1]. Low wear resistance of aluminum has caused reducing its tribological uses; the aluminum matrix composites reinforced with ceramic particulates have shown significant improvements [2-9]. Many studies have been made in fabrication of secondary phase particles reinforced materials [10-15], but there are few studies on aluminum alloy/ ZrSiO$_4$/ SiC. The aluminum alloy which has been used in present research has good casting and fluidity in aluminum matrix composites producing process (16). In the present study, the effects of ZrSiO$_4$ and SiC reinforcing particles on microstructure and mechanical properties of this alloy have been investigated and compared.

2. MATERIALS USED AND EXPERIMENT PROCEDURE

The present study, well-known aluminum alloy ADC-12 is used as matrix material and high purity zircon sand (ZrSiO$_4$) and silicon carbide (SiC) as reinforcement. ADC-12 alloy was funded in the form of ingots. The compositional analysis of the ADC-12 alloy was done by wet chemical analysis which is given in Table 1. The composite was prepared by two-step stir casting route. Required quantity of ADC-12 alloy was taken in a graphite crucible and melted in an electric furnace. The temperature of melt was raised to 750°C. This molten metal was stirred using a graphite impeller at a speed 630 rpm. At this 630 rpm vortex is created in the melt, which facilitate to suck the reinforced particles inside the melt. The ceramic particles used as reinforcement were taken in defined proportion and mixed properly by spatula. Particle was stirred before to preheat at 450°C for the moisture. Zircon sand and silicon carbide particles of fine grade were selected for present work. After mixing of particles the melt slurry is allowed to solidify in a graphite crucible at room temperature conditions. After solidification the mixture is again re-melted in a furnace to ensure that slurry is in fully liquid condition and then melt is stirred with impeller for 10–15 min. similar type of synthesis of composite was reported earlier by various researchers (17). During production of composite, the amount of ADC-12 alloy, stirring duration and position of stirrer in the crucible were kept constant to minimize the contribution of variables related to stirring on distribution of second phase particles. The other detail of it is given in Table 2. The past research shows that 12 % reinforcement of zircon sand reinforced composite has given better mechanical property, so we have restricted the reinforcement up to 15 % only. Moreover, this is also in accordance with Table 1 Composition of the ADC-12 alloy in wt%.
**Table 1:** Composition of the ADC-12 alloy in wt%

| ADC12 Alloy | Si  | Cu  | Mg  | Ni  | Fe  | Zn  | Ti  | Pb  | Sn  | Mn  | Al  |
|-------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Wt%         |     |     |     |     |     |     |     |     |     |     |     |
| 11.8        | 1.2 | 0.9 | 0.9 | 0.3 | 0.2 | 0.02| 0.02| 0.005| 0.4 | Balance |

*Fig 1:* Stir casting machine  
*Fig 2:* Control panel of stir casting machine  
*Fig 3:* Casting processes [18]

**Table 2:** List of processing parameter

| Parameter               | 1<sup>st</sup> step | 2<sup>nd</sup> step |
|-------------------------|----------------------|---------------------|
| Melting temperature     | 750°C                | 800°C               |
| Total stirring time      | 22-25 min            | 5 min               |
Mixing time | 8-10 min | -
---|---|---
Blade angle | $90^\circ$ | $90^\circ$
No of blades | 4 | 4
Position of stirrer | Up to 2/3 depth in the melt | Up to 2/3 depth in the melt

In order to compare and correlate the effect of dual particle reinforcement on mechanical and tribological properties, five different composites containing a total of 12 wt. % reinforcement in different proportion were fabricated and have been designated by alphabets.

**Table 3: 12Wt% Reinforcement combination in composites**

| Composite | ZrSiO$_4$ | SiC |
|---|---|---|
| A | 3 | 9 |
| B | 6 | 6 |
| C | 9 | 3 |

The reinforcement combinations are given in Table 3. The compressive strength of ADC-12 alloy/SiC/ZrSiO$_4$ composites are tested at Universal testing machine (model- MECH/UTE-40T).

**Fig 4:** Universal testing machine (model- MECH/UTE-40T).

The micro structural analysis has been done with the help of both optical (Eclipse MA-100 and Nikon) and scanning electron microscope (JEOL, JSM-6390A, Japan) at various magnifications. Before SEM observation the sample was mechanically polished and etched by Keller’s reagent for obtaining better contrast. Micro hardness of the different phases was measured using (Mitutoyo, Japan) micro hardness tester. Micro hardness measurement was done on each set of sample by taking minimum of five indentations per sample at 100 kgf load.
2.1. MECHANICAL PROPERTIES OF REINFORCEMENT PARTICLES

2.1.1. Zircon Sand

Zircon sand consists of mostly zirconium silicate (ZrSiO$_4$) and some hafnium in addition to some rare earth elements, titanium minerals, monazite, etc. Zirconium was found to be a promising candidate as reinforcement material for aluminum, zinc and lead based composites (19).

Table 4: Chemical composition of zircon sand

| Composition | ZrO$_2$ | SiO$_2$ | HfO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | MgO |
|-------------|---------|---------|---------|-------------|-------------|-----|
| Content (wt%) | 67.22 | 30.85 | 1.39 | 0.11 | 0.029 | 0.014 |

Table 5: Mechanical properties of zircon sand.

| Properties                                      | Zircon sand |
|------------------------------------------------|-------------|
| M.P. ($^°$C)                                    | 2500        |
| Limit of application ($^°$C)                    | 1870        |
| Hardness (Moh’s Scale)                          | 7.5         |
| Density (g/cm$^3$)                              | 4.5-4.7     |
| Linear coeff. of expansion ($10^{-6}$k)         | 4.5         |
| Fracture toughness (MPa-m$^{1/2}$)              | 5           |
| Crystal structure                               | Tetragonal  |

2.2.2. Silicon Carbide

Silicon Carbide is the only chemical compound of carbon and Silicon. It is used in abrasives, refractories, ceramics, and numerous high-performance applications. Silicon carbide crystal structure is tetrahedral.

Table 6: Mechanical properties of silicon carbide

| Properties                                      | Silicon carbide |
|------------------------------------------------|-----------------|
| M.P. ($^°$C)                                    | 2200-2700       |
| Limit of application ($^°$C)                    | 1400-1700       |
| Linear coeff. of expansion ($10^{-6}$k)         | 4.1-7.7         |
### 3. RESULTS AND DISCUSSION

#### 3.1. MICROSTRUCTURE ANALYSIS

The optical micrograph of the composites is shown in Fig. 5a–c. The micrograph clearly reveals the absence of dendritic morphology in all the composites under investigation. The dendritic structure can be modified during casting which is influenced by many factors such as dendritic fragmentation, restriction of dendritic growth by the particles, and thermal conductivity mismatch between the particles and melt. Dendritic fragmentation can be attributed to the shearing of initial dendritic arms by the stirring action. It was also found that the perturbation in the solute field due to the presence of particles can change the dendrite tip radius and the dendrite tip temperature. These effects give rise to a dendrite to cell transition as the density of particles is increased. Also the length of the dendrite is reduced in the presence of the particles. Ceramic particle also act as a barrier for dendritic growth and this phenomena is more pronounced if the cooling rate is high. In this work reported that the particle can be assumed to act as a barrier to the dendritic growth.

![Fig 5](image-url)

**Fig 5:** The SEM micrograph of composites **a** composite A, **b** composite B, **c** composite C

The SEM micrographs of composite ‘B’ reinforced with 12 % of SiC and zircon sand particles in the ratio of 3:1 are shown in Fig. 5a. Micrograph shows the refined microstructure and homogeneous distribution of particles in the alloy matrix. Refined microstructure and absence of dendritic morphology can be attributed to the two-step stir casting process adopted here in which prolonged time of mixing and stirring is bifurcated. Colonies of eutectic silicon are arranged in the vicinity of the particle. Moreover, near the particle, eutectic silicon having globular morphology or blunted morphology as compared to the matrix can be seen. Each and every particle is having a colony of eutectic silicon which is indicative of the role of particles in nucleating the eutectic silicon. SiC and zircon sand particles provide effective site for nucleation.
and also restricts the growth of dendrite and modifies the matrix with more refined structure leading to improvement in strength.

The SEM micrographs of composite ‘B’ containing 12 % of silicon carbide and zircon sand particles of the ratio of 1:1 in this work is shown good bonding in Fig. 5b. It depicts the refinement of microstructure and eutectic silicon. Eutectic silicon along with dispersed particles is densely arranged in such a way that they almost cover the entire matrix. The eutectic silicon refines to finer scale and nucleates near particles as colonies. Clustering of particles is observed at some places and some of the clustered particles have chipped out during polishing the samples. However, fine particles have the tendency of clustering though it is not much pronounced in the prepared composites.

The SEM micrograph of composite ‘C’ containing 12 % of SiC and zircon sand particles in the ratio 1:3 is shown in Fig. 5c. Finer to coarse distribution of eutectic silicon along with homogeneous distribution of particles can be seen. Eutectic silicon colonies around particle are more pronounced in the micrograph. Eutectic silicon distribution is more refined and morphology has changed from acicular to globular around the particles. Also in the matrix the eutectic silicon having blunted morphology as compared to long needle shape or acicular is seen. However, the fine particles and silicon form a network structure because of pushing interface from different nucleation sites. Moreover, the clustering of fine particles at some places is also observed.

Overall analysis of structure indicates that microstructure is refined whereas eutectic silicon are having blunted and globular morphological features. This refinement may lead to better tribological and mechanical properties in the composite. The colonization of eutectic silicon in the vicinity of the particles enhances particle capability of wear resistance. The reinforced particles are uniformly distributed in the alloy matrix. The good bonding between particles and alloy matrix is also revealed in the microstructural analysis. Moreover, porosity is at minimum level and not observed in the optical examination, although clustering is seen at same places in the composite. Microstructure analysis shows that addition of SiC has a pronounced effect on the microstructure and eutectic silicon refinement. The degree of microstructure and eutectic silicon refinement increases in accordance with the increase of SiC-reinforced particle percentage. The most prominent feature observed in all composite is the absence of dendritic growth which is accounted for two-step stir casting processing of the composites.

3.2. MICRO HARDNESS AND DENSITY OF COMPOSITE

Micro hardness measurement at different phases of composite has been carried out to know the effect of reinforced particulates on the alloy matrix. This is given in Table 4. Micro hardness measurement has been carried out on the embedded reinforced particles as well as in the vicinity of particles and matrix.
Table 7: Variation of micro hardness (H<sub>v</sub>) And Density

| Composite | Particle (H<sub>v</sub>) | Interface (H<sub>v</sub>) | Matrix (H<sub>v</sub>) | Density g/cm<sup>3</sup> |
|-----------|--------------------------|---------------------------|------------------------|--------------------------|
| A         | 228.03                   | 121.38                    | 73.06                  | 2.716                    |
| B         | 165.10                   | 109.81                    | 61.43                  | 2.735                    |
| C         | 180.43                   | 111.91                    | 63.98                  | 2.715                    |

3.3. COMPRESSIVE TEST

The room temperature tensile test was conducted using Universal testing machine (model-MECH/UTE-40T). The as-cast ADC-12 alloy composite samples for compressive test were prepared using lathe machine. The tests were conducted trice to get the average response value.

![Dimension of testing specimen](image)

**Fig 6**: Dimension of testing specimen

![Composites before testing](image)

**Fig 7**: composites before testing

![Composites after testing](image)

**Fig 8**: composites after testing

Table 8: Compressive strength of composite A

|                              |                  |
|------------------------------|------------------|
| Ultimate compressive load (KN)| 19.040           |
| Ultimate compressive strength (N/mm<sup>2</sup>) | 242.424          |
| Deflection at ultimate load (mm) | 1.600            |
| Maximum deflection (mm)      | 1.700            |
Table 9: Compressive strength of composite B

| Parameter                                | Value  |
|------------------------------------------|--------|
| Ultimate compressive load (KN)           | 34.320 |
| Ultimate compressive strength (N/mm²)    | 437.904|
| Deflection at ultimate load (mm)         | 3.400  |
| Maximum deflection (mm)                  | 3.800  |

Table 10: Compressive strength of composite C

| Parameter                                | Value  |
|------------------------------------------|--------|
| Ultimate compressive load (KN)           | 16.112 |
| Ultimate compressive strength (N/mm²)    | 238.850|
| Deflection at ultimate load (mm)         | 2.500  |
| Maximum deflection (mm)                  | 2.600  |
The compressive strength of composites A and C is nearly similar but C is higher for composite A. The composite with equal amount of dual reinforcement, i.e., composite B provides better strength as compared to composites C & A. The composite with 50% silicon carbide and 50% zircon sand shows better compressive strength as compared to composite B&C. Dual particles have different role in the matrix, silicon carbide particles refines the eutectic silicon whereas zircon sand particles provides good bonding characteristics to the matrix. The composite B is combination of 50% zircon sand and 50% silicon carbide particles shows better compressive strength as compared to composite C&A which is composite C having 75% zircon sand and 25% silicon carbide particles and composite A having 25% zircon sand and 75% silicon carbide particles and.

After analyzing micro hardness and compressive strength behavior of composites, it is clear that dual particle reinforcement mixed in definite proportion is only effective for enhancing micro hardness and compressive strength. Silicon carbide particles are better reinforcement for compressive behavior as compared to zircon sand particles. Composite B exhibits better compressive strength due to high micro hardness of the interface between particle and matrix.

4. CONCLUSIONS

In this study the effects of SiC and ZrSiO$_4$ ceramic grain sizes and mixing proportions on the microhardness and compressive strength characteristics of composite materials were studied. The following conclusions can be drawn from the results of the present study performed on dual particle sized reinforced composites. The microstructure and micro hardness of the micro size particle reinforced composites, which have positively affected the microhardness and compressive strength behavior. We found that as the size of the reinforcement increases the microstructure heterogeneity increases. It has been observed that, in dual particle reinforced composites, large particles prove useful in preventing agglomerations.

1. Dual particles have different functions in the composite. Silicon carbide refines the eutectic silicon where as zirconium silicate provides good internal bonding strength and micro hardness.
2. The morphology is not observed in the composites as can be seen in the microstructures of stir cast composites.
3. The combination of reinforcement in ADC-12 alloy composites 50% SiC and 50% zircon sand particles reinforced composite (Composite B) yields better compressive strength as compared to other combination.
4. The reinforcement up to 12Wt% yields better mechanical properties as compared to other combination.

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