

# INFLUENCE OF ZrB<sub>2</sub> ON THE MICROSTRUCTURAL CHARACTERISTICS OF AA6082/ZrB<sub>2</sub> COMPOSITES

## VPLIV ZrB<sub>2</sub> NA MIKROSTRUKTURNE KARAKTERISTIKE AA6082/ZrB<sub>2</sub> KOMPOZITOV

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In a recent scenario, particulate-reinforced aluminium-matrix composites (AMCs) are used for plenty of applications in aerospace, non-structural, structural, transportation and automotive industries. This study concentrates on the manufacturing of AA6082/ZrB<sub>2</sub> aluminium-matrix composites (AMCs) using a liquid metallurgy process. The zirconium diboride particles of three different weight percentage, i.e., (0, 3, 6 and 9) %, are reinforced with aluminium alloy (AA6082) using the stir casting technique. Hardness, tensile and compression tests were conducted to evaluate the mechanical behaviour. The microstructures of the composites were examined using a scanning electron microscope (SEM). The SEM microphotographs proved the successful dispersion of ZrB<sub>2</sub> particles into the aluminium matrix. The tensile fracture surface of the prepared composites and the plain aluminium were examined with the SEM to understand the tensile fracture mechanism. Tensile fracture morphology reveals different modes of fractures, like brittle and ductile. The unreinforced AA6082 plain matrix alloys are subjected to a ductile mode of fracture, but with an increase in zirconium diboride the mode of failure gradually transforms to brittle fracture. The mechanical properties of the composites are improved after the dispersion of ZrB<sub>2</sub> particles. A tremendous improvement in the mechanical properties of the AMCs was found for 9 w/% ZrB<sub>2</sub> particles in the matrix.

Keywords: tensile strength, fracture morphology, AA6082 alloy, compression strength

Dandanes se kompoziti s kovinsko osnovo iz Al zlitin, ki so ojačani z drobnimi karbidnimi delci (AMCs), uporabljajo za mnoge aplikacije v letalski, vesoljski, konstrukcijski (gradbeni), nekonstrukcijski, transportni in avtomobilski industriji. Ta študija se je osredotočila na izdelavo AMCs na osnovi AA6082/ZrB<sub>2</sub> z uporabo metalurgije raztaljene kovine. Avtorji so cirkonijeve diboridne delce v treh različnih masnih deležih, to je (0, 3, 6 in 9) % vmešavali v Al zlitino (AA6082) s t.i. tehniko vmešavanja delcev v raztaljeno kovino in njenega intenzivnega premešavanja (angl.: stir casting technique). Na preizkušanjih iz izdelanih kompozitov so določili trdoto, natezno in tlačno trdnost. Mikrostrukturo kompozitov so opazovali pod vrstičnim elektronskim mikroskopom (SEM). SEM mikrosnetki so potrdili uspešno disperzijo (enakomerno porazdelitev) ZrB<sub>2</sub> delcev v matrici Al zlitine. Za razumevanje mehanizmov loma nateznih preizkušancev čiste Al zlitine in izdelanih kompozitov so uporabili SEM mikrosnetke. Morfologija preloma nateznih preizkušancev je pokazala različne tipe prelomov, od krhkega do žilavega (duktilnega). Neojačana, čista Al zlitina AA6082 je kazala popolnoma žilav prelom. Z naraščajočim deležem dodanih Zr diboridnih delcev pa je prelom postopoma prehajal v krhki način. Mehanske lastnosti so se izboljševale s povečevanjem disperzije ZrB<sub>2</sub> delcev. Avtorji ugotavljajo, da je bistveno izboljšanje mehanskih lastnosti AMCs nastopilo pri dodatku 9 w/% ZrB<sub>2</sub> delcev.

Ključne besede: natezna trdnost, morfologija preloma, zlitina AA6082, tlačna trdnost

## 1 INTRODUCTION

Aluminium-matrix composites (AMCs) are extensively used in the aircraft, marine, automobile and defence sectors because of their outstanding strength-to-weight ratio, low thermal expansion, high wear resistance, etc. These are largely used in the fabrication of automobile components like drive shafts, pistons, cylinder liners and connecting rods.<sup>1,2</sup> There are different processing routes available for the production of AMCs. The processing routes can be divided into three groups, namely, liquid-state processing, semi-solid-state processing and solid-state processing.<sup>3</sup> The fabrication of AMCs by the liquid metallurgy route is inexpensive, simple, flexible and it is also applicable for mass production.<sup>4</sup>

The liquid-state processing method contains either the incorporation of ceramic particles externally or formed inside the molten metal. The former is known as ex-situ (stir casting technique), while the latter is called an in-situ technique.<sup>5</sup>

There are a massive number of techniques employed to produce the composites. Among them, stir casting is tremendously popular owing to its applicability for large volume production and low production costs. In the stir casting method, the matrix material was melted above the re-crystallization temperature in the furnace and the reinforcement particles were added into the molten metal to prepare the composite material. The properties of the composite material depend on the matrix material, reinforcement particle, shape and size of the reinforcement, the volume percentage of the reinforcement and the spatial distribution of the particles in the matrix. The stir

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casting technique has shown some incomparable advantages, like the uniform distribution of particles in the matrix and strong interfacial bonding between the matrix and the particle.<sup>6-8</sup> In the recent times, a huge number research works have been going on by attempting different reinforcement materials into aluminium matrix to improve and enrich the properties of the composites. Ex-situ ceramic particles, such as TiC, B<sub>4</sub>C, SiC, TiB<sub>2</sub>, ZrB<sub>2</sub>, Si<sub>3</sub>N<sub>4</sub>, TiO<sub>2</sub>, have been extensively used as reinforcements in AMCs.<sup>9-11</sup> Among these reinforcement particulates, (ZrB<sub>2</sub>) is a potentially attractive class of reinforcement material for AMCs as it possesses a desirable combination of physical and mechanical properties, including high strength, superior hardness, high young's modulus, low density and also better wear resistance. Also, ZrB<sub>2</sub> does not react with molten aluminium to form reaction products at the interface of the matrix and reinforcement.<sup>12-14</sup>

The fabrication of AA6082-alloy-based AMCs was reported in some literature as presented here.<sup>15-18</sup> Pardeep Sharma et al.<sup>15</sup> synthesized AA6082/Gr AMC by the stir casting technique and evaluated the mechanical and microstructure characteristics of the aluminium-matrix composites. A. Thangarasu et al.<sup>16</sup> fabricated AA6082/TiC composite AMCs using the stir casting route and investigated the influence of TiC particles on the mechanical and wear behaviour of the AMCs. They concluded that the TiC particles enhanced the hardness, tensile strength and wear resistance of the AMCs. Pardeep Sharma et al.<sup>17</sup> fabricated AA6082 aluminium reinforced Si<sub>3</sub>N<sub>4</sub> by the stir casting technique. They have analysed the role of Si<sub>3</sub>N<sub>4</sub> particulates on the microstructure and mechanical properties of the AMCs. K. Ravikumar et al.<sup>18</sup> worked on AA6082 aluminium alloy-WC AMCs and reported the tensile surface morphological and mechanical properties of the developed composite.

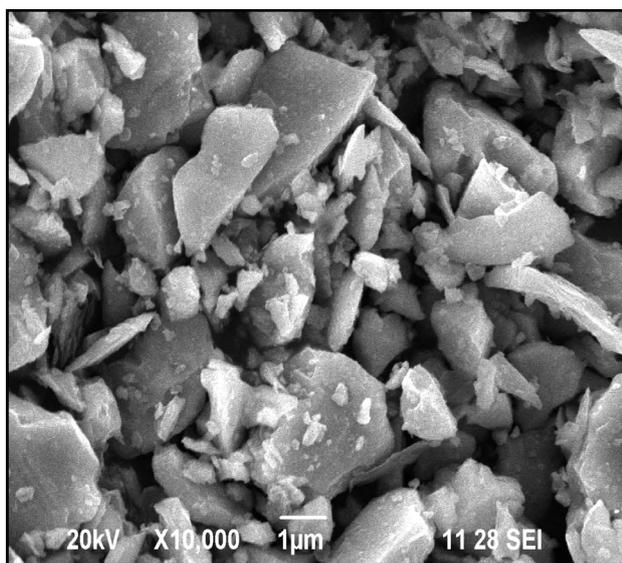


Figure 1: SEM images of ZrB<sub>2</sub> particles

An extensive review of several research articles showed that no detailed findings are available for Al-ZrB<sub>2</sub> composites prepared through a stir casting route. The present study focuses on the production, microstructure characterization and mechanical properties of ZrB<sub>2</sub> particulate-reinforced AA6082 alloy composites fabricated through the stir casting technique. The microstructures of the AA6082 parent alloy and manufactured composites were evaluated using scanning electron microscopy (SEM). The SEM morphologies of the fracture surfaces were examined to understand the tensile fracture mechanism.

## 2 EXPERIMENTAL PART

### 2.1. Processing of composites

In this work, aluminium alloy AA6082 (Si: 1.1 %, Fe: 0.2 %, Cu: 0.02 %, Mn: 0.8 %, Mg: 0.9 %, Zn: 0.1 %, Cr: 0.15 %, Ti: 0.08 % and Al: balance) was employed as the matrix material. Zirconium diboride (ZrB<sub>2</sub>) was chosen as reinforcement for the fabrication of the composite material in powder form. An SEM micrograph of ZrB<sub>2</sub> is shown in **Figure 1**. The SEM micrographs of the received ZrB<sub>2</sub> showed that the particles had a wide size distribution and irregular shape. The average size of the ZrB<sub>2</sub> particles was 40 µm.

The AA6082 rods were melted using an electrical furnace in a graphite crucible. In order to remove the moisture content and gases in the particulates preheating of the ZrB<sub>2</sub> was carried at 400 °C for an hour. The stirrer was lowered into the crucible and was maintained at a constant speed of 400 min<sup>-1</sup>. The temperature of the furnace was maintained at 850 °C and a calculated quantity of reinforcement particles was added to the molten aluminium. The melt was stirred erratically for the

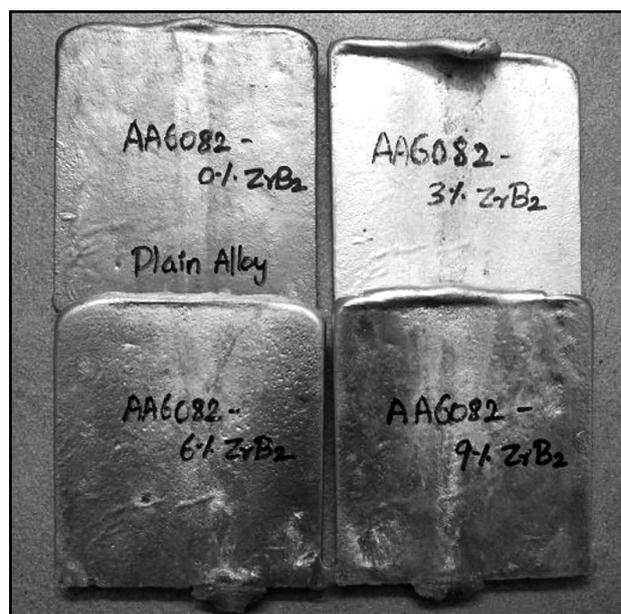


Figure 2: Photograph of a typical batch of castings

duration of 20 min. After the execution of the process, the melt was poured into a preheated die. Castings were obtained for different weight fractions of particles as per the same procedure. **Figure 2** displays a typical batch of castings.

## 2.2. Microstructure and mechanical testing

The stir-cast composite samples were machined, polished using a standard metallographic procedure and etched with Keller's reagent. The microstructure was observed using a scanning electron microscope attached with an energy-dispersive spectroscopy. The microhardness of the base alloy and the prepared composites were estimated by conducting Vickers hardness testing at 0.5 Kgf load, which is applied constantly for a dwell time of 15 s. The measurement of the hardness was taken at six locations on each specimen to obtain an average value of the hardness. Tensile tests were carried out on the computerised 100 KN Servo hydraulic universal testing machine (Instron 8801) with a strain rate of 1.0 mm/min at room temperature. The tensile specimens were prepared as per the ASTM E8 standard, having a gauge length of 40 mm, a gauge width of 7 mm and a thickness of 6 mm. For every combination, three tests were conducted and the average values are reported. The compression strength of the base matrix alloy and the prepared composites were determined using a fully computerised universal testing machine. The compression specimens were prepared as per the ASTM E9 standard.

## 3 RESULTS AND DISCUSSION

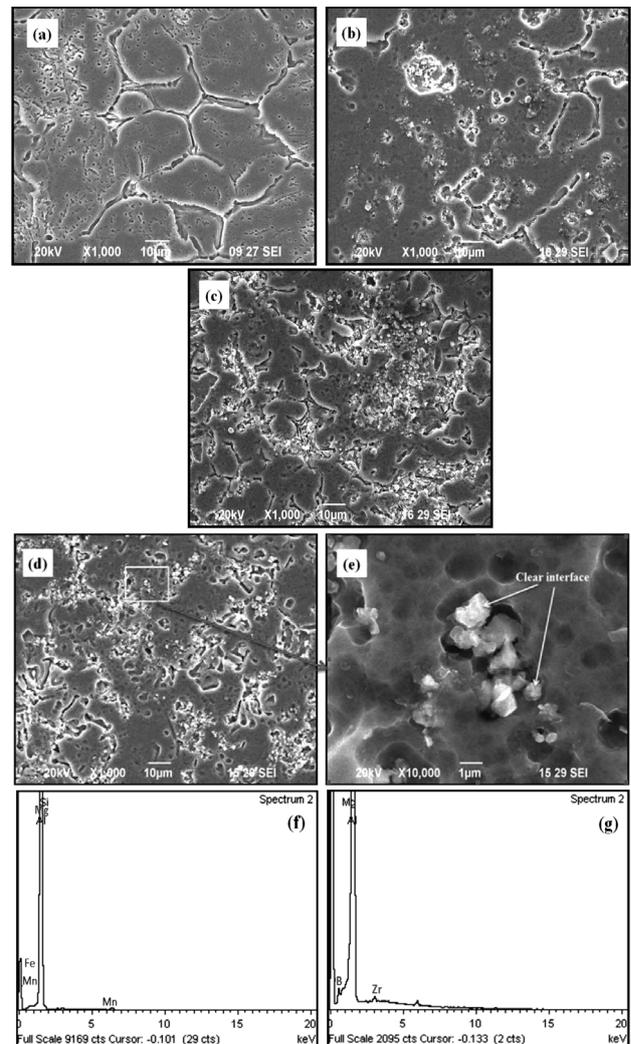
### 3.1. SEM and EDAX analysis of AA6082 alloy and AA6082/ZrB<sub>2</sub> composites

**Figure 3a** presents the SEM images of the as-cast matrix AA6082 alloy. **Figure 3b to 3e** records the SEM microphotographs of the AA6082/ZrB<sub>2</sub> composites. The microstructural examination proves the continuous dissemination of ZrB<sub>2</sub> particles in the matrix and also shows the sharp and clear interface between the ZrB<sub>2</sub> particle content and the AA6082 matrix. This kind of dissemination is desirable as it helps to enhance the properties of the composites. The SEM images clearly show a clean and finer interface and strong bonding between the reinforcement and the matrix. It could improve the load bearing capacity of the composites.<sup>5,14</sup> Moreover, the Al matrix-ZrB<sub>2</sub> particle interface plays a vital role in deciding the mechanical properties of the composites. These findings agree with earlier studies by several researchers.<sup>2,19,20</sup> **Figures 3f and 3g** depict the energy-dispersive X-ray analysis (EDAX) of the AA6082 alloy and AA6082/ZrB<sub>2</sub> AMCs. The magnesium (Mg) and silicon (Si) particles in AA6082 are confirmed by carrying out EDAX analysis depicted in **Figure 3f**. The element peaks of aluminium and the chemical composition identified with a high-intensity

peak were magnesium and silicon. The extra elements such as manganese and iron were identified with very low peaks in the AA6082 alloy.<sup>17</sup> **Figure 3g** depicts the EDAX pattern of 9 w/% ZrB<sub>2</sub> reinforced composite, it is noticed from the EDAX pattern that the main elements present are Al, Zr and B, which ensures that the elements are homogeneously dispersed throughout the matrix.

### 3.2. Effect of ZrB<sub>2</sub> in the AA6082 matrix on hardness

The microhardness of the AA6082/ZrB<sub>2</sub> composites is plotted in **Figure 4**. The hardness was recorded to 46 HV at 0 w/% of ZrB<sub>2</sub> to 81 HV at 9 w/% of ZrB<sub>2</sub>. The hardness of the casted composites is linearly increased with the increase in ZrB<sub>2</sub> particle content. The improvement in hardness could be attributed to the continuous distribution of ZrB<sub>2</sub> in the matrix and the particulate strengthening effect of the AA6082 alloy. Several investigators reported a similar trend that the incorporation of



**Figure 3:** SEM micrographs of AA6082/ZrB<sub>2</sub> composites containing ZrB<sub>2</sub> content; a) 0 w/% ZrB<sub>2</sub>, b) 3 w/% ZrB<sub>2</sub>, c) 6 w/% ZrB<sub>2</sub>, d and e) 9 w/% ZrB<sub>2</sub>, f) EDAX analysis of AA6082 alloy and g) EDAX analysis of AA6082/9 w/% ZrB<sub>2</sub>

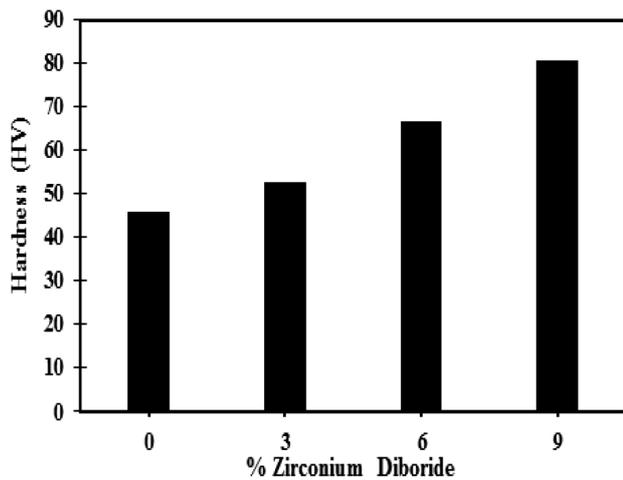


Figure 4: Effect of hardness on w/% of ZrB<sub>2</sub> particles

B<sub>4</sub>C, Al<sub>2</sub>O<sub>3</sub>, TiC, rice husk ash, Si<sub>3</sub>N<sub>4</sub> and AlN enhances the hardness of AMCs.<sup>8,11,21,22</sup>

Baradeswaran et al.<sup>23</sup> stated an opposite trend, that an increase in the incorporation of graphite drastically decreases the hardness of the AMCs owing to the lubricating nature of graphite. Rajmohan et al.<sup>24</sup> showed that the incorporation of mica decreased the hardness of AMCs, thus enhancing the machinability of composites. An increase in hardness is reasonable, owing to the resistance provided to the indentation by the hard reinforced particles. AA6082 with 9 w/% of ZrB<sub>2</sub> composites exhibited the greatest hardness. The accumulation of ZrB<sub>2</sub> particles in the AA6082 matrix enhances the surface area of the reinforcement and also reduces the matrix grain size. Therefore, the mechanical properties of the composites were notably increased with the increase in ZrB<sub>2</sub> content.

### 3.3. Effect of ZrB<sub>2</sub> in the AA6082 matrix on tensile strength

Figure 5 displays the role of ZrB<sub>2</sub> particle content on the tensile strength of the AA6082/ZrB<sub>2</sub> composites. The

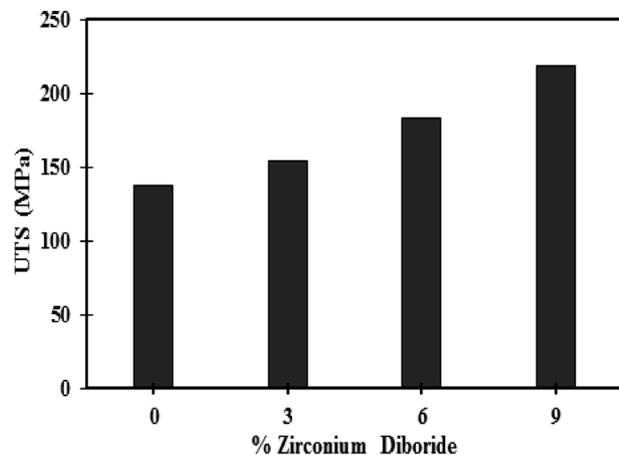


Figure 5: Effect of tensile strength on w/% of ZrB<sub>2</sub> particles

tensile strength of the AMCs was increased from 138 MPa to 219 MPa because of the increasing weight fraction of ZrB<sub>2</sub> particles. A remarkable augmentation in the tensile strength of the composite was found at 9 w/% ZrB<sub>2</sub> particles in the matrix. A similar kind of enhancement in tensile strength while incorporating particles such as TiB<sub>2</sub>, AlN, TiC, Si<sub>3</sub>N<sub>4</sub> and WC was reported by investigators.<sup>5,6,11,17,18,25</sup> However, the inclusion of Gr, mica, ZrSiO<sub>4</sub> in aluminium alloy decreased the mechanical behaviour of the composites.<sup>23,24,26</sup> The reason for the enhancement of tensile strength in the AA6082/ZrB<sub>2</sub> composite is the incorporation of the hard nature of ZrB<sub>2</sub> particles in the soft AA6082 matrix. Moreover, the escalation in tensile strength may be ascribed to the uniform dissemination of reinforcement particles in the matrix, resulting in the effectual transfer of applied tensile load from the matrix to the reinforcement and thereby increasing the load-bearing capacity of the composites.<sup>2,6,19</sup> AA6082/9 w/% ZrB<sub>2</sub> AMCs showed a 62.31 % higher tensile strength compared to the unreinforced AA6082 alloy. This can be credited to the presence of an improved surface area of the reinforcement particle that provides massive resistance to the plastic deformation, which leads to improving the tensile strength of composites.

### 3.4. Tensile fracture surface analysis of AA6082/ZrB<sub>2</sub> composites

The Figure 6 shows the tensile fracture morphology of the AA6082 alloy and the AA6082/ZrB<sub>2</sub> AMCs. The AA6082 alloy revealed ductile fracture behaviour with evenly disseminated, larger size voids noticed in the fracture surface, shown in Figure 6a. Figure 6b depicts how the incorporation of ZrB<sub>2</sub> particles extensively reduces the voids on the fracture surface. It can be ascribed to the grain refinement by the ZrB<sub>2</sub> particulates. Figure 6c re-

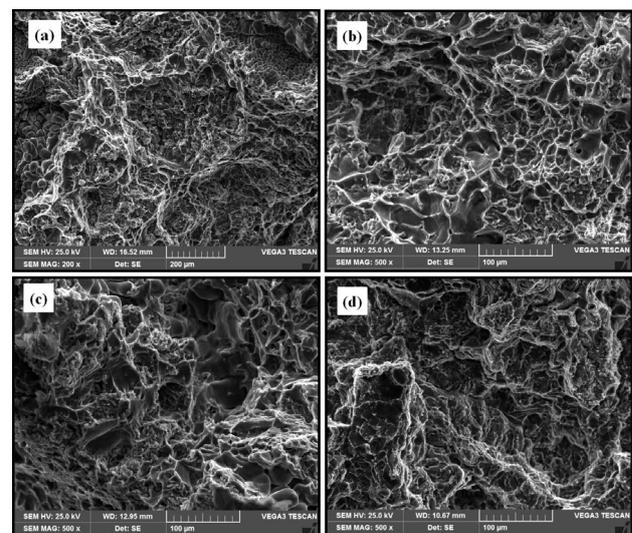


Figure 6: Fracture morphology of AA6082/ZrB<sub>2</sub> AMCs containing ZrB<sub>2</sub>: a) 0 w/%, b) 3 w/%, c) 6 w/% and d) 9 w/%

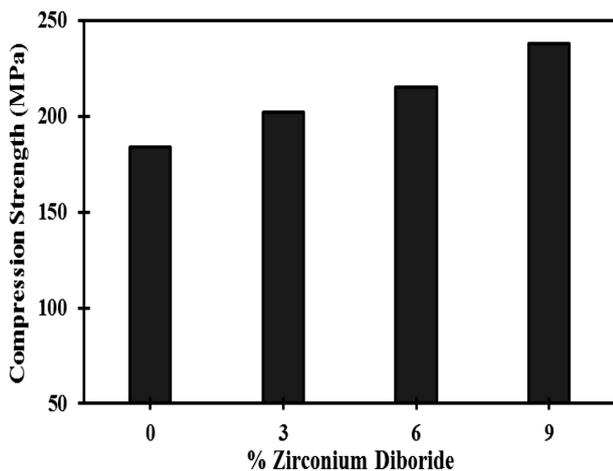


Figure 7: Effect of compression strength on w/% of ZrB<sub>2</sub> particles

veals the smaller size voids compared to that of the base alloy. Moreover, the ZrB<sub>2</sub> particles are intact at several places owing to superior bonding with the matrix. **Figure 6d** depicts how the flatness of the fractured surface increases with the enhanced weight fraction of ZrB<sub>2</sub> particle content. There are no voids around the particles, ensuring strong interfacial bonding between the matrix and the reinforcement.<sup>2,19</sup> The fracture surface analysis shows that the fabricated composite experienced a ductile nature of fracture at the microscopic level and brittle nature of fracture at the macroscopic level.

### 3.5. Effect of ZrB<sub>2</sub> in AA6082 matrix on compression strength

The effect of ZrB<sub>2</sub> content on the compression strength of AA6082/ZrB<sub>2</sub> AMCs is revealed in **Figure 7**. The compression strength was noticed to increase with the increase of ZrB<sub>2</sub> particle content and it is remarkably higher than the compression strength of the non-reinforced matrix alloy. The presence of hard ZrB<sub>2</sub> particles in the matrix serves as a barrier for the dislocation movement. This barrier leads to enhance the compression strength of the AA6082/ZrB<sub>2</sub> composites. The incorporation of ZrB<sub>2</sub> particles increases the mechanical bonding between the matrix and reinforcement. Therefore, the compression strength of AMCs increased to about 29.34 % while adding 9 w/% of ZrB<sub>2</sub> particle to the aluminium alloy.

## 4 CONCLUSIONS

In the present experimental study, AA6082-alloy-based composites were manufactured through the stir casting method with several weight proportions of ZrB<sub>2</sub> and the characterization was performed with SEM. The mechanical properties of the AA6082 parent alloy and AA6082/ZrB<sub>2</sub> developed composite were measured. Based on the present research study, the following conclusions are drawn:

- Microstructural observations revealed that there was no interface reaction product between the ZrB<sub>2</sub> particles and the matrix during the production of the composites.
- A homogeneous dissemination of ZrB<sub>2</sub> particles along the AA6082 aluminium matrix can be noticed in the SEM micrographs.
- The manufactured composite of 9 w/% of ZrB<sub>2</sub> offers a maximum hardness of 81 HV.
- The tensile strength of the composites was increased from 138 MPa to 219 MPa with respect to incorporation of the weight fraction of ZrB<sub>2</sub> particles. Correspondingly, the hardness of the composites increases with increasing ZrB<sub>2</sub> weight fraction.
- The tensile fracture surface analysis reveals different mode of failures, namely, ductile, brittle and ductile. The tensile fracture morphology of the AA6082/ZrB<sub>2</sub> AMCs shows the unified mode of fracture.
- The AA6082/9 w/% of ZrB<sub>2</sub> composites revealed superior compression strength when compared to the parent matrix alloy.

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