

## Revolutionizing the material performance of AZ64/ZrB<sub>2</sub> composites for engineering applications

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### ABSTRACT

The Zirconium Di- Boride (ZrB<sub>2</sub>) reinforced AZ64 magnesium metal matrix composite's (MMCs) tribological performance was studied for potential use in engineering applications. The composite was developed using the stir-casting method with the help of ultrasonic vibrations for mixing molten AZ64 and preheated ZrB<sub>2</sub> particles as it achieves uniform dispersion and better wettability. The physical characteristics was studied through density measurement and the result showed that 3% ZrB<sub>2</sub> reinforced composites had an increase in 1.275% of density when related to 0% reinforced MMCs. The absorbed energy values from charpys impact test of reinforced composites showed an increase of around 85% from the as-cast alloy. The micro hardness of the ZrB<sub>2</sub> particles reinforced composite was significantly improved after ultrasonic dispersion. From XRD and EDX it is evident that inclusion of the ZrB<sub>2</sub> increased beta-phase precipitation in the Mg alloy, which in turn enhances the strength of the composites. Sliding wear tests were conducted in dry conditions utilizing pin-on-disc (POD) tribometer at standard loads (20–60N) and speeds (1.2–2.4 m/s). Improved wear resistance was seen in the 3% ZrB<sub>2</sub> reinforced composites as a result of its finer grain and relatively uniform distribution of ZrB<sub>2</sub> particles. Increasing the load resulted in a higher wear rate of the composite at all sliding speeds. Increased capacity of the reinforcement and other characteristics of the produced composite proved to be superior to the AZ64 as cast alloy in all wear test situations.

**Keywords:** AZ64; Metallurgical; Microhardness; Pin on Disc; Wear.

### 1. INTRODUCTION

The engineering sectors place a premium on materials that combine extreme durability with low weight. Researchers in the field of materials science and professionals in the engineering industries have focused on magnesium alloys because of their potential to reduce energy consumption in a wide variety of applications [1]. Composites have the potential to displace traditional metals like aluminium, zinc, and steel. Because of their favorable strength-to-weight ratio, magnesium alloys like AZ31 [2] and AZ64 [3] have seen explosive increase in demand over the past decade in the commercial sector. Despite their other desirable properties, these alloys have poor characteristics, restricting their usefulness in a variety of critical components [4]. Machine parts typically wear out faster, function less well, and degrade in general due to the friction created by their constant

meshing and sliding contacts [5]. In order to increase magnesium alloys' use in engineering, wear resistance qualities should be improved [6]. Composites made of ceramic particles embedded in magnesium alloy have shown amazing energy absorption capacity, improved mechanical qualities, and wear resistance in recent years, thanks to advances in production [7]. Ultrasonic agitation in a stir casting process has been shown to be a useful tool for producing MMCs [8].

The stir-casting method was used by the authors [9] to create magnesium AZ31 MMCs reinforced with graphite particles at 1 wt. %, 5 wt. %, and 9 wt. %. Results demonstrated that the reinforcing particles improved damping by raising dislocation density. This work [10] created ZX51 magnesium composites reinforced with  $\text{Al}_2\text{O}_3$  particles in altering the volume percentage via stir casting. In addition, the homogeneous microstructure greatly improved the mechanical characteristics. However, the produced composite showed a modest increase in porosity. Another study used stir casting to create AZ64 magnesium MMCs that contained 15% Sic and had mean particle sizes of 15  $\mu\text{m}$  and 150  $\mu\text{m}$ . Composites with a particle size of 15  $\mu\text{m}$  exhibited improved particle dispersion, elastic modulus, and hardness in comparison to composites with a particle size of 150  $\mu\text{m}$ . However, it was found to be inferior to a monolithic alloy in terms of ultimate tensile strength and ductility [11]. The mechanical behaviour of Mg-based metal matrix composites were enhanced by reinforcing with  $\text{ZrB}_2$  powder particles. As a result, the grain structure became more refined. The matrix around the powder particles also generated numerous dislocations [12].

Researchers [13] have shown that incorporating ceramics particles into a magnesium matrix improves mechanical qualities such as hardness, tensile strength, and wear resistance. In addition to these, the incorporation of  $\text{ZrB}_2$  particles has had a major impact on the recrystallization, fine-tuning, and overall grain-size reduction of the magnesium matrix [14]. Nonetheless, some impact on the development of textures was noted. Using POD tribometer, the wear pattern under dry sliding conditions was analyzed. The results showed that the micro hardness and wear resistance of the manufactured composite specimen improved as the particle concentration rose. Furthermore, the particles were distributed consistently across all of castings [15]. AZ64 combinations were extruded to comprehensively examine the influence of Sn incorporation on microstructure development and mechanical characteristics. The Sn concentration used here is no over three wt.%, which has been observed to result in the creation of coarser undissolved particulates [16]. Published studies indicate that very few researchers or no research have investigated  $\text{ZrB}_2$ -reinforced with AZ64 magnesium MMCs by stir casting. Since magnesium-based MMCs have many applications in engineering, they have been the subject of most prior research. Wear behaviour of  $\text{ZrB}_2$ -reinforced Mg based metal matrix composites under various speeds and loads has not been studied. Hence in this research, ultrasonic stir-casting was used to create AZ64 with 3%  $\text{ZrB}_2$  MMCs. Then, the materials' physical, mechanical, metallurgical properties were measured by different experimentations. The effects of varying loads and sliding speeds on the wear characteristics of produced MMCs were also investigated.

## 2. EXPERIMENTATION

### 2.1. Fabrication of composites

AZ64 were selected as the matrix material and reinforcement particle of  $\text{ZrB}_2$  with a size of 15–24  $\mu\text{m}$  were used for this study and purchased from Krish Met Tech Pvt Ltd, Chennai and the SEM image is shown in Figure 1. First, the AZ64 Mg tiny ingots were placed in the crucible of the stir casting furnace and heated to 700°C to ensure effective melting. A motorized steel stirrer rotated at 300 rpm as it slowly mixed with 3 wt% of preheated  $\text{ZrB}_2$  particles at temperature of 400 degree Celsius into the molten AZ64 for 5 minutes. Argon and SF6 gas were mixed in the right proportions (70:30), creating a controlled inert gas atmosphere, in which composites were synthesized. Composite castings are made with the help of the ultrasonic attachment to the stir casting system. 20 kHz frequency, 2 kW power titanium alloy ultrasonic probe was submerged in the molten mixture for 3 minutes after the stirrer was removed as ultrasonic vibrations are applied to ensure that the particles in the  $\text{ZrB}_2$ -reinforced composite are dispersed uniformly and do not agglomerate. The composite material combination was placed into a cast-iron mould cavity preheated to 250°C. All of the casting pouring was done with a 1 minute stir period. The entire process of the research work carried out is shown in Figure 2.

### 2.2. Physical test

The density of 0% of  $\text{ZrB}_2$  reinforced AZ64 and AZ64 with 3% of  $\text{ZrB}_2$  composite samples was calculated with the help of Equation (1). Using mixture rule method, to determine theoretical densities of matrix and reinforcing materials, which came out to 1.810 g  $\text{cm}^3$  and 4.50 g  $\text{cm}^3$ , respectively. The 0% of  $\text{ZrB}_2$  reinforced AZ64 and AZ64 with 3% of  $\text{ZrB}_2$  MMCs densities were measured using immersion fluid and Archimedes principle.

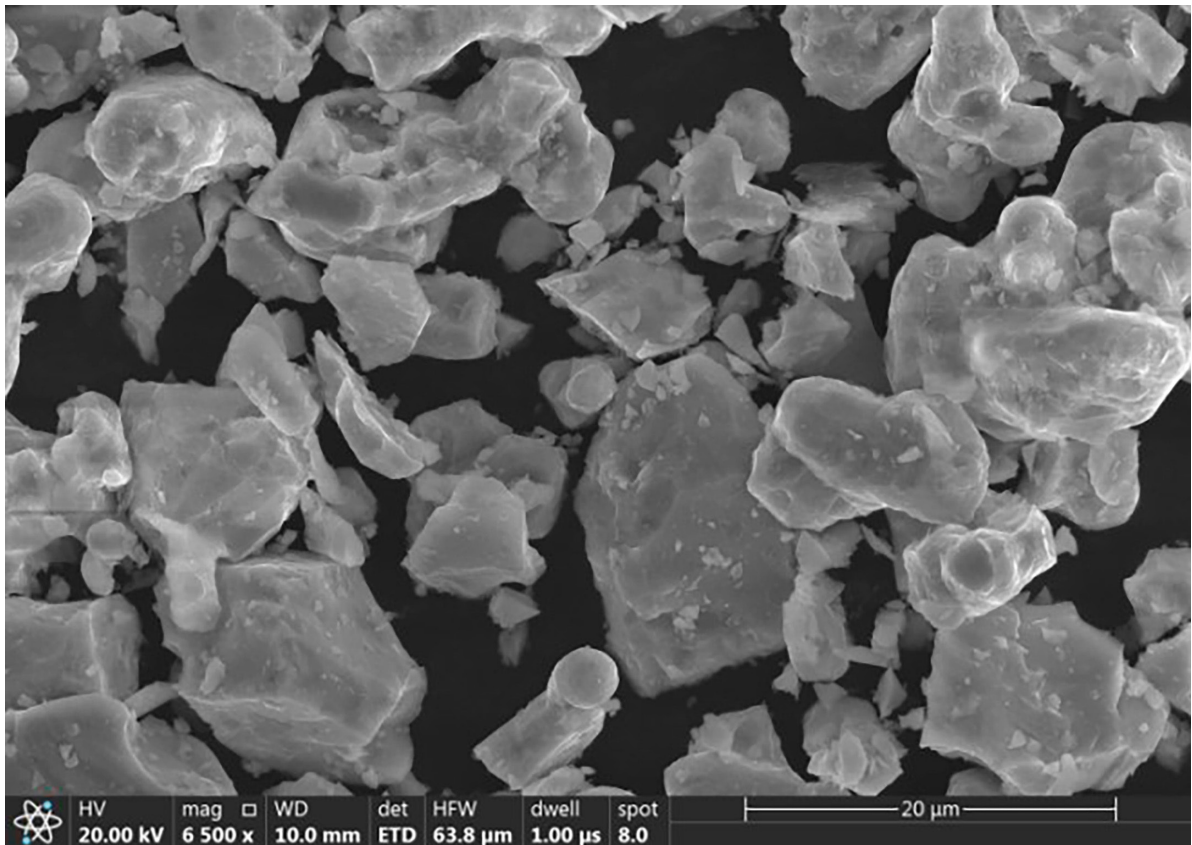


Figure 1: SEM image of  $\text{ZrB}_2$  particles.

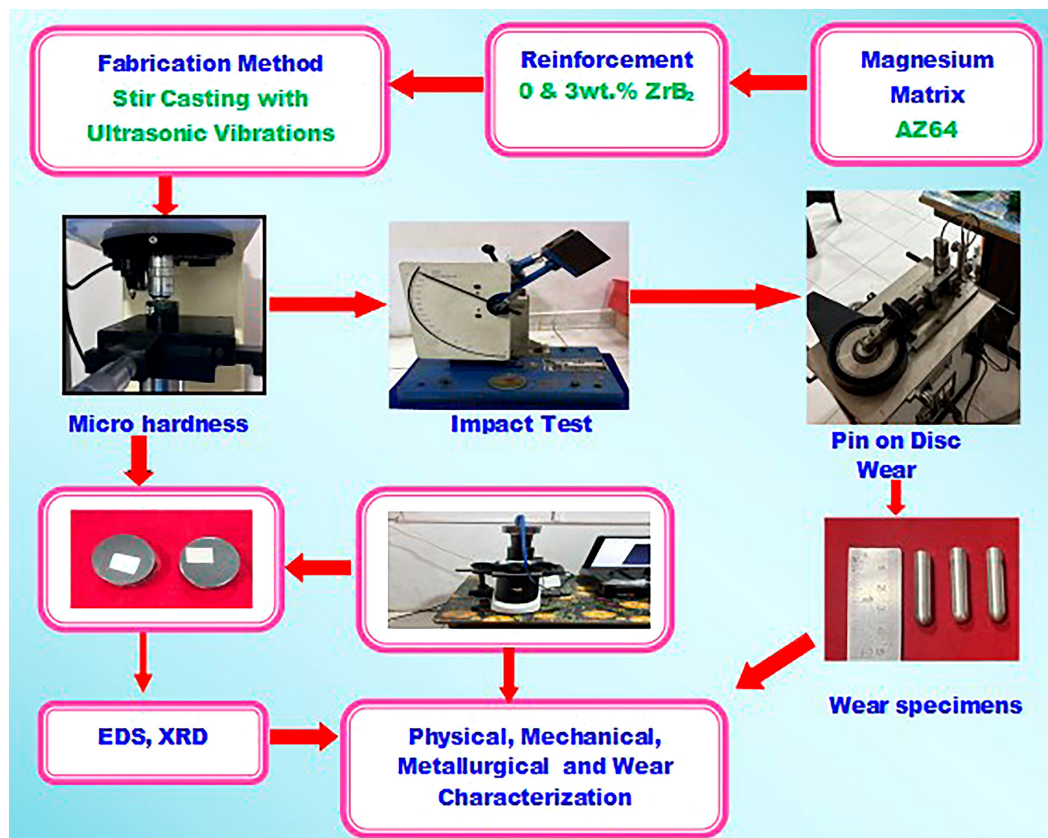


Figure 2: Entire work of the research.

$$\rho_x = \frac{Wa}{(Wa - W_w)} \times \rho_w \quad (1)$$

Where

$\rho_x$  Density of the composite

$\rho_w$  Distilled water's density

### 2.3. Mechanical testing

The mechanical characteristics of AZ64 with 0% of ZrB<sub>2</sub> and AZ64 with 3% ZrB<sub>2</sub> composite are evaluated using micro hardness and charpy impact test. A Vickers micro hardness tester was utilized to evaluate the composite's micro hardness with a diamond indenter having an included angle of 136° applied with a load of 100 kgf and a dwell duration of 15 seconds were taken for the research. The average micro hardness was calculated from 5 indentations on composites with ASTM E384-99 standard. Charpy Impact toughness was analyzed using an impact testing machine, adhering to the ASTM E23-18 standard, with a V-notch specimen and size of 55 × 10 × 10 mm and a mean value was derived from three tested samples for accuracy.

### 2.4. Wear testing

Dry sliding wear tests were performed on both the AZ64 with 0% of ZrB<sub>2</sub> composite and AZ64 with 3% of ZrB<sub>2</sub> composite specimens to determine the wear resistance. The specimens are formed as cylindrical pins were polished using SiC emery paper (with grit sizes ranging from 180 to 2000) to achieve higher surface finish. For the wear tests [17], the device's steel disc counter face was slid over round pin samples (ϕ8 × 30 mm). The extent of sliding wear on the specimens was quantified using a weighing scale. For this study to get credible results, this research averaged the weight loss of three specimens produced of the AZ64 with 0% of ZrB<sub>2</sub> and AZ64 with 3% of ZrB<sub>2</sub> composite. The wear rate under each load condition was calculated by separating total weight loss by total sliding distance. Table 1 lists the characteristics and factors that were examined in these wear testing. ASTM G99 was used for the standard testing for tribology.

### 2.5. Metallurgical testing

In preparation for metallographic analysis, specimens underwent a series of polishing procedures. The components were first polished using different grades of SiC emery paper with a grit of 180 to 2000. Then they were immersed in an etchant picric acid with an etching time of 15 seconds. The standard used for optical microscopy for sample preparation was ASTM E-3 and for measurement and analysis ASTM F410 was used for the study. The elemental composition of the AZ64 with 0% of ZrB<sub>2</sub> and AZ64 with 3% of ZrB<sub>2</sub> composite were determined using Energy Dispersive X-ray Spectroscopy (EDX) analysis. Also, the presence of different phases in the AZ64 with 0% of ZrB<sub>2</sub> and AZ64 with 3% of ZrB<sub>2</sub> composite was ascertained using X-ray Diffraction (XRD) analysis with the ASTM standard of C1365-98. A scanning range of 10° to 90° and a scanning speed of 1°/min were part of the XRD examination parameters.

**Table 1:** POD wear settings.

PARAMETERS	SPECIFICATIONS
Disc rotation speed	200 RPM
Counter surface	EN-32 steel disc
Applied load	20 N, 40N, 60N
Cylindrical pin dimensions	ϕ8 × 30 mm
Test duration	45 minutes
Sliding distance	2000 m
Track diameter (d)	50 mm
Sliding speed	1.2, 1.8, 2.4 m/s

### 3. RESULTS AND DISCUSSIONS

#### 3.1. Density

The density of AZ64 with 0% of ZrB<sub>2</sub> and AZ64 with 3% of ZrB<sub>2</sub> composite samples was calculated and the results for the density are illustrated in Table 2. Before weighing the specimens in distilled water (W<sub>w</sub>), they were weighed in air (W<sub>a</sub>) using a precision electronic scale. Afterwards, they were weighed again. three readings were taken from each sample, and an average was then calculated. The MMCs specimen's density dropped below the theoretical value after casting.

#### 3.2. Impact test

The impact tests were conducted that was kept at room temperature. The AZ64 with 0% of ZrB<sub>2</sub> specimen had a low impact value of 2.87 J and an impact toughness value of 3.22 J of energy. Composites reinforced with 3% of ZrB<sub>2</sub> showed absorbed energy of 5.21 J and an impact toughness value of 3.59 J and the values are shown in Table 3. This proves that composite samples have greater toughness than unreinforced AZ64 magnesium alloy. Energy absorption during impact testing provided a quantifiable measure of AZ64 with 0% of ZrB<sub>2</sub> and AZ64 with 3% of ZrB<sub>2</sub> composite's impact toughness. Impact strength increases as the distance between particles decreases also the Hall-Petch effect has showed the composite improved fracture toughness. According to these phenomena, smaller powder particles have the potential to increase strength. The homogeneous distribution and disorientation of the ZrB<sub>2</sub> particles contained in the composite hinder further fracture development and propagation. Constraining the motion of dislocations led to a decrease in grain size and a strengthening of the particles, both of which greatly improved the fracture toughness. Furthermore, the Mg matrix was replaced by the ZrB<sub>2</sub> reinforcements as the primary load transmission mechanism. The work-hardening capacity was improved because to the ZrB<sub>2</sub> reinforcing phases [18]. Meanwhile, in the AZ64 matrix alloy, the Mg<sub>17</sub>Al<sub>12</sub> phase tends to diffuse along the grain boundaries. This diffusion process adversely affects the impact strength of the material. In the composite specimen, micro-cracks were observed to initiate and propagate during the processing phase. This occurrence is attributed to the presence of the magnesium oxide heterogeneous phase, which renders the casting more brittle [19].

#### 3.3. Microhardness test

In this study, the microhardness of AZ64 with 0% of ZrB<sub>2</sub> and AZ64 with 3% of ZrB<sub>2</sub> reinforced composite was measured. A microhardness value of 62.33 HV was measured for the AZ64 with 0% of ZrB<sub>2</sub>, while a value of mean of 96 HV was measured for the composite reinforced with 3% of ZrB<sub>2</sub> particles. The composite and matrix alloy had a microhardness gap of over 54%. The ZrB<sub>2</sub> powder particles boost the composite's load-bearing capability significantly by limiting dislocation movement inside the matrix. MMCs inherent strengthening mechanism is also important in making them stronger. The results demonstrate that the composite has a significantly greater micro hardness than the unreinforced AZ64 which is displayed in Table 4. ZrB<sub>2</sub>

**Table 2:** Density outcomes of AZ64 with 0% and 3% of ZrB<sub>2</sub> composite.

SPECIMEN	THEORETICAL DENSITY (g/cm <sup>3</sup> )	COMPUTED EXPERIMENTAL DENSITY (g/cm <sup>3</sup> )
AZ64 with 0% of ZrB <sub>2</sub>	1.810	1.804
AZ64 with 3% of ZrB <sub>2</sub>	1.839	1.827

**Table 3:** Findings of impact for AZ64 with 0% and 3% of ZrB<sub>2</sub> composite.

SPECIMEN	ENERGY ABSORBED (J)	IMPACT TOUGHNESS (J)
AZ64 with 0% of ZrB <sub>2</sub>	2.87	3.22
AZ64 with 3% of ZrB <sub>2</sub>	5.21	3.59

**Table 4:** Findings of micro hardness for AZ64 with 0% and 3% of ZrB<sub>2</sub> composite.

SPECIMEN	VICKERS MICRO HARDNESS (HV)			MEAN (HV)
AZ64 with 0% of ZrB <sub>2</sub>	62	62	63	62.33
AZ64 with 3% of ZrB <sub>2</sub>	96	95	97	96

powder particles are evenly dispersed throughout the composite, which prevents dislocations from moving during specimen deformations [20].

### 3.4. Tribology test (weight loss)

Figure 3 (a–c) shows the AZ64 with 0% of  $\text{ZrB}_2$  and AZ64 with 3% of  $\text{ZrB}_2$  composite losing weight under typical loads of 20 N, 40 N, and 60 N at 1.2 m/s, 1.8 m/s, and 2.4 m/s, respectively. When sliding speed rose from 1.2 to 2.4 m/s under current loading conditions, both the AZ64 with 0% and 3% of  $\text{ZrB}_2$  lose a much greater amount of mass. Both the AZ64 with 0% and 3% of  $\text{ZrB}_2$  reinforced composite lost weight at higher sliding speeds. These results are consistent with those found in other investigations [21–22].

The AZ64 with 3% of  $\text{ZrB}_2$  composite showed lesser weight loss than the AZ64 with 0% of  $\text{ZrB}_2$  under all wear conditions studied. In particular, in the most extreme loading condition, with a greater sliding speed (2.4 m/s) and a load of 60 N, the composite's wear resistance was greatly improved. This suggests that with greater loads the generated composite's wear rate deviates further from that AZ64 alloy. The composite's enhanced hardness may account for the reduced weight loss observed, especially at higher sliding speeds. The  $\text{ZrB}_2$  particles in the composite contribute to its increased hardness and higher wear resistance. Less friction and wear occurred because the AZ64 with 3% of  $\text{ZrB}_2$  composite's and counter face material shared a smaller surface area. Similar findings were observed in [23]. These findings provide credence to the Archard rule, which

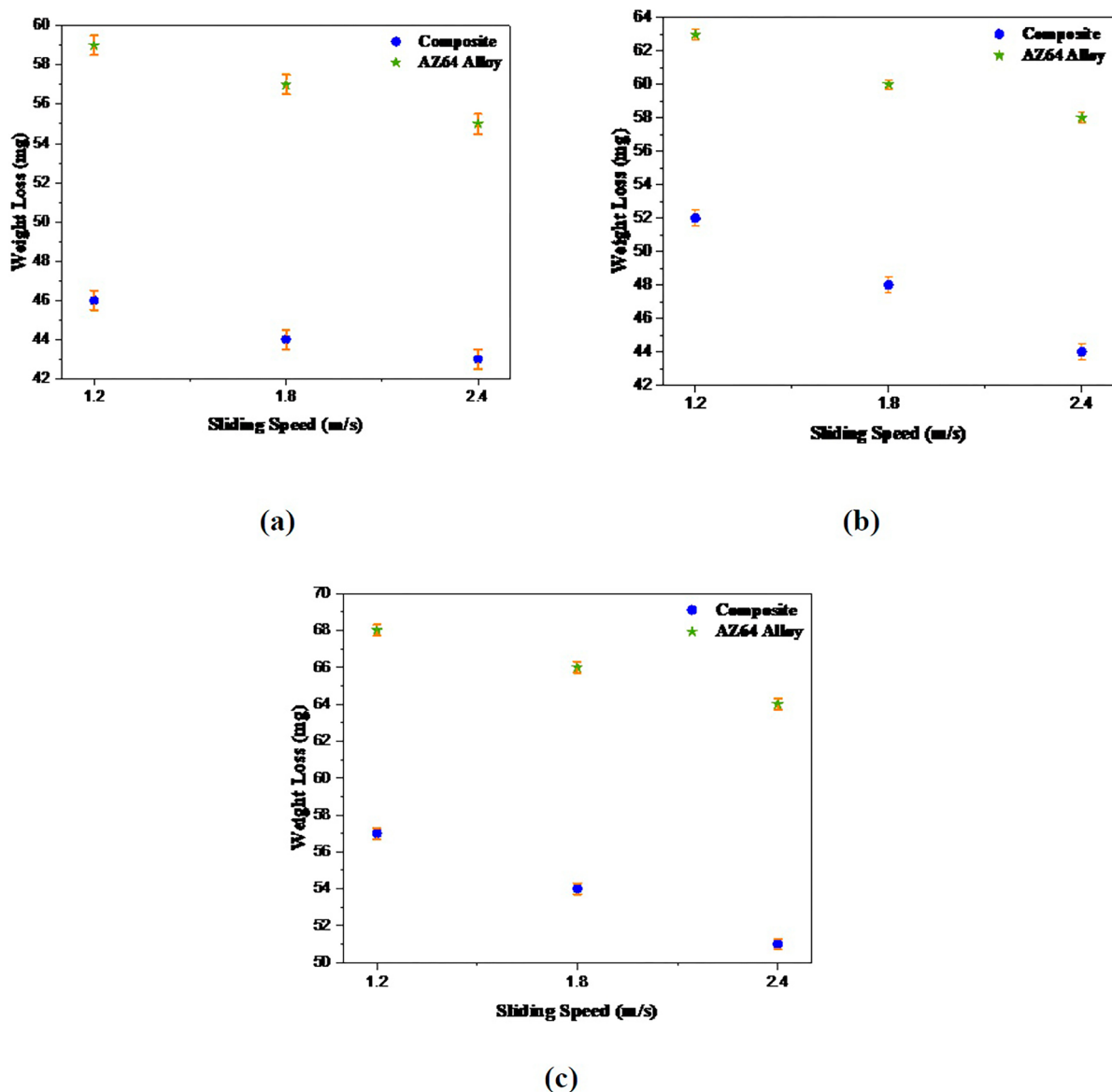


Figure 3: (a–c): Comparison of AZ64 and composite wear rates at sliding speed at loads of (a) 20 N, (b) 40 N and (c) 60 N.



asserts that wear is reduced in materials with higher hardness. The 3% of  $\text{ZrB}_2$ -reinforced AZ64 composite's higher hardness and high strength allowed for increased wear resistance at a reduced stress level (20 N). When subjected to a greater stress, however, the AZ64 matrix's work-hardening capability increased more substantially (60 N).

### 3.5. Tribology test (coefficient of friction)

The coefficient of friction (COF) [24] for the AZ64 with 3% of  $\text{ZrB}_2$  composite is shown in Figure 4 for a variety of sliding situations, including normal loads of 20, 40, and 60 N with sliding speeds of 1.2, 1.8, and 2.4 m/s.

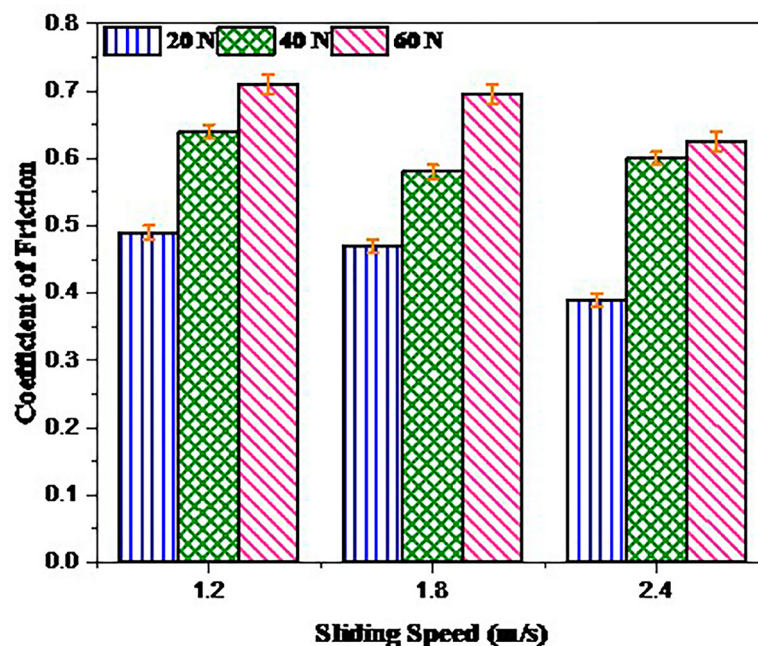
### 3.6. Metallurgical characterization through XRD and EDX

Significant peaks associated with  $\alpha$ -Mg,  $\text{Mg}_{17}\text{Al}_{12}$ , and  $\text{ZrB}_2$  were seen in XRD in AZ64 with 0% reinforcement and AZ64 with 3% of  $\text{ZrB}_2$  composite are shown in Figure 5 (a–b). Origin software was utilized to calculate grain size of AZ64 with 0% of  $\text{ZrB}_2$  and AZ64 with 3% of  $\text{ZrB}_2$  composite from the XRD peaks from scherrer formula [25–26]. In order to find the dislocation density, the amount of dislocation lines per unit volume - the Williamson-Small man relationship was employed. This relationship provides a method to estimate the dislocation density based on the XRD data, offering insights into the structural integrity and mechanical properties of the material studied. This was done for each peak to determine an average grain size. Table 5 displays the dislocation densities and crystal grain sizes. These numerical values as in Table 5 demonstrate that the  $\text{ZrB}_2$ -reinforced composite has seen a significant reduction in grain size as because the ultrasonic agitation impact on the molten substance led to large differences in crystalline size, which in turn refined the composites' grain structure. However, the ultrasonic vibrations were able to eliminate the dislocations and speed up the recrystallization process. Because the AZ64 matrix and the reinforcing particles have different rates of thermal expansion, formation, and strengthening effects.

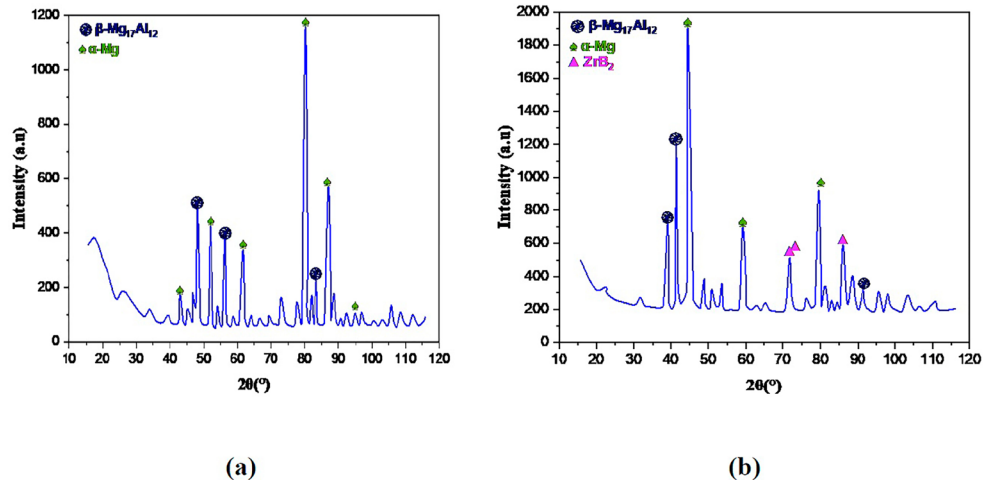
Figure 6(a–b) of the EDX displays the elemental makeup of the AZ64 with 0% of  $\text{ZrB}_2$  and the AZ64 with 3% of  $\text{ZrB}_2$  composite. The EDX report contains a breakdown of the primary components present in both AZ64 and AZ64 with 3% of  $\text{ZrB}_2$  composite. The dislocation density is formed due to the discrepancy among AZ64 and  $\text{ZrB}_2$  particles in terms of their elastic moduli. This happens because the reinforcing particles take on the load instead of the matrix alloy.

### 3.7. Metallurgical characterization through microscopy

For 3% of  $\text{ZrB}_2$  composites, the reinforcement distributions in the AZ64 matrix are rather constant. The microphotograph in Figure 7(a–b) highlights the 3% contents in the composites at different locations. The  $\text{ZrB}_2$  particles were seen experiential within the dendrites zones of the obtained microscopy.



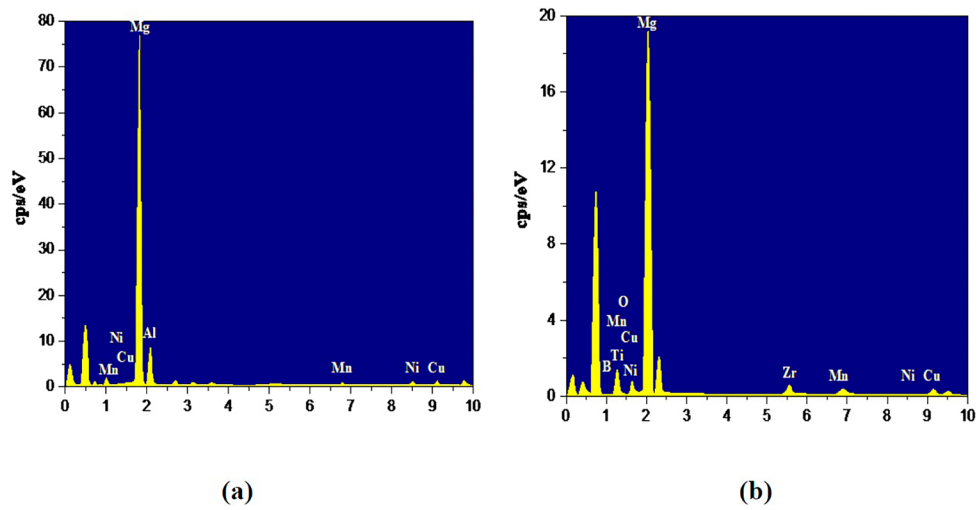
**Figure 4:** COF for AZ64 with 3% of  $\text{ZrB}_2$  composite for varying sliding speeds of 20 N, 40 N and 60 N.



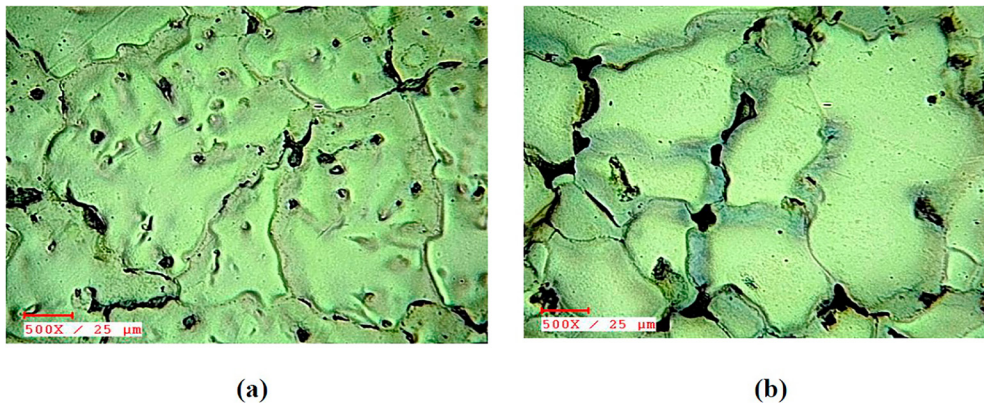
**Figure 5:** XRD spectrum of (a) AZ64 with 0% of ZrB<sub>2</sub> (b) AZ64 with 3% of ZrB<sub>2</sub> composite.

**Table 5:** AZ64 with 0% and 3% of ZrB<sub>2</sub> composite dislocation density and grain size.

SPECIMEN	GRAIN SIZE (μm)	DISLOCATION DENSITY ( $\delta \times 10^{14}/\text{m}^{-2}$ )
AZ64 with 0% of ZrB <sub>2</sub>	55	16.373
AZ64 with 3% of ZrB <sub>2</sub>	23	25.741



**Figure 6:** EDX spectrum of (a) AZ64 with 0% of ZrB<sub>2</sub> (b) AZ64 with 3% of ZrB<sub>2</sub>.



**Figure 7:** (a–b): Optical micrograph of AZ64 with 3% of ZrB<sub>2</sub> composite at different locations.



#### 4. CONCLUSIONS

- From this research the synthesization of AZ64 with  $ZrB_2$  is successfully made with ultrasonic method through liquid metallurgy stir casting route.
- Ultrasonic vibrations are effective in the preliminary stages of treatment because they disperse the agglomerates of  $ZrB_2$  particles that form at contact in grain boundary area.
- Density, Micro hardness, impact, are all greatly improved by using hard 3% of  $ZrB_2$  powder particles in the composite. The increase in these characteristics is 1.27%, 54%, 11.49% respectively.
- With EN-32 steel disc serving as counter face, this study set out to investigate the effect of  $ZrB_2$  particles on wear performance behaviour of AZ64 composite across a range of sliding speeds and loads.
- Due to the improved mechanical properties leading to significantly less weight loss in all wear conditions by reducing wear rate and COF.
- Because of the  $ZrB_2$  powder particles, the AZ64 with 3% of  $ZrB_2$  composite is both harder and more resistant to damage during the work-hardening process.
- Wear rates were reduced across the board as sliding speeds were increased.
- From metallurgical characterization with XRD and EDS, it is evitable for the presence of  $ZrB_2$  incorporation reduced the grain size and uniform dispersion over the matrix.
- The proposed composite can be used in wear-resistant applications hitherto reserved for magnesium and related composites and it is appropriate for application in continuous sliding machine components due to its excellent qualities.

#### 5. ACKNOWLEDGMENTS

This project was supported by Researchers Supporting Project number (RSP2025R315), King Saud University, Riyadh, Saudi Arabia.

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