Research Article

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Microstructural and mechanical characterization of Al6061-ZrB_2 nanocomposites fabricated by powder metallurgy

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Abstract: Al6061-ZrB_2-reinforced metal matrix nanocomposites were synthesized through the hot pressed process (PM) with varying the nano ZrB_2 content in wt% (0, 0.5, 1, 1.5, and 2) and studied their microstructural and mechanical properties. Mechanical properties such as hardness, compression strength, tensile strength, and yield strength were investigated for the fabricated composites. Field emission scanning electron microscope (FESEM) microstructural characterization of the PM samples revealed a uniform distribution of ZrB_2 nanoparticles in the matrix. X-ray diffraction (XRD) analysis confirmed the presence of aluminum and ZrB_2 particulates in the Al-metal-matrix composites. The incorporation of ZrB_2 particles into the Al matrix was shown to significantly enhance the microhardness of the fabricated nanocomposites by 11%. In addition, the incorporation of ZrB_2 reinforcements in the aluminum alloy significantly enhanced the compressive, yield, and tensile strengths of the AMCs. The density of composites was significantly influenced by the presence of ZrB_2 particulates. The best composite among the fabricated composites was identified as Al6061-2wt% ZrB_2.

Keywords: powder metallurgy, aluminum metal matrix nanocomposites, ZrB_2 particulates, mechanical properties, microstructural properties

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1 Introduction

Metal matrix composites provide a distinct set of characteristics that make them highly suitable for an extensive variety of engineering purposes, including aerospace, automotive, marine, construction, and sports industries. These composites continue to be an area of active research and development, aiming to further enhance their properties and explore new applications due to their high strength-to-weight ratio and superior stiffness [1]. AMMCs are developed by mainly two methods such as stir casting and powder metallurgy (PM). PM is one of the most widely used methods for improving the mechanical properties of composites, along with good interaction between the matrix and reinforcement. PM is the best method for synthesizing composites with homogeneous, fine structures, and unique properties [2,3]. The PM approach fabricates metal-matrix composite (MMC) made of aluminum alloys when compared to other conventional fabrication techniques [4]. Various industries, including automobiles and aviation, have used AMMCs for their high specific strength and low density [5]. When compared to Fe alloys, aluminum alloy has many advantages including lower density and greater conductivity [6].

Ravichandran *et al.* [7] fabricated aluminum as a matrix and graphene and TiO_2 as reinforcement in different wt% by using the powder metallurgy route and evaluated the stresses in fabricated composites. Dasari *et al.* [8] examined the properties of graphene oxide (GO)-reinforced aluminum composites made *via* powder metallurgy. Aluminum powders (35 µm) mixed with varying GO contents (0.05, 0.1, and 0.2 wt%) were cold compacted, sintered, and analyzed for uniform GO dispersion using scanning electron microscope (SEM)/energy dispersive X-ray (EDX). X-ray diffraction (XRD) and Raman spectroscopy identified the phases within the composite matrix postsintering. The results showed that when GO is evenly distributed, GO-reinforced aluminum composites can achieve hardness values comparable to those of rGO-reinforced aluminum composites. Zamani *et al.* [9] examined the mechanical and tribological properties of powder-metallurgically manufactured aluminum–graphite (Al-Gr) composites with different graphite contents (3, 5, and 7 wt %). A pin-on-disc tribometer was used to assess wear performance and mechanical attributes such as hardness, tensile, and flexural strength under dry sliding settings. According to the study, 3 wt% Gr provided the optimum combination of tribological and mechanical qualities, including a low wear rate and a smooth, self-lubricating graphite coating. Bodukuri *et al.* [10] studied the mechanical behavior of B_4C/SiC/Al composites produced *via* powder metallurgy. Their study showed a significant increase in microhardness with higher B_4C content, and the microstructure revealed a uniform distribution of particles within the metal matrix. Abdizadehet *et al.* [11] investigated the mechanical, microstructural, and corrosion properties of aluminum reinforced with zircon composites. The highest compression strength was achieved with the specimen containing 5% zircon, at 650°C sintering temperature. Sudhakar Srinivas and Balakrishna [12] developed four-layer functionally graded composites (FGMs) using aluminum, silicon carbide, and magnesium peroxide, fabricated through a sintering process with varying time, temperature, and pressure. FGMs were evaluated for their mechanical, tribological, and microstructural properties, demonstrating impressive compressive strength (315 MPa) and hardness (1.26 GPa micro and 1.87 GPa macro), outperforming standard composites. Taguchi optimization revealed that sintering temperature plays the most significant role in determining mechanical performance.

In this study, Al6061 reinforced with nano ZrB_2 with different wt% was fabricated by using the PM route. The microstructural behavior, density, and mechanical properties were investigated from the Al6061/n-ZrB_2 fabricated composites.

2 Experimental details

2.1 Materials

Al6061 is used as the matrix material, and the chemical composition of Al6061 is presented in Table 1. Al6061 powder was obtained from Venuka Engineering Pvt. Ltd., Hyderabad. ZrB_2 used as a reinforcement having 50–100 nm particle size was procured from NANOSHEL, Punjab. Al6061 ingots purchased from Vision castings, Hyderabad. Initially, Al6061 and ZrB_2 (0, 0.5, 1, 1.5, and 2 wt%) powders are mixed in a turbo mixer, and then the mixed compositions were hot pressed at 35 kg/cm^2 and 550°C for 10 min. The final fabricated composites are shown in Figure 1, and the detailed step-by-step fabrication procedure and setup for the hot pressing fabrication method are presented in Figure 2.

Table 1: Chemical composition of Al6061

Element	Mg	Fe	Cu	Mn	Si	Al
Composition (%)	0.69	0.23	0.31	0.33	0.52	Balance

2.2 Characterization

The phases present in Al6061 alloy and composite were identified using an X-ray diffractometry at an interference angle of 10°–80° at 15 kV. The average nanocrystallite size was estimated using the well-known Debye–Scherrer formula $D = k\lambda/\beta\cos\theta$ [13]. Hardness measurements of all composites were conducted under a 300 g load and 15-s dwell time. The compressive, tensile, and yield strengths of the nanocomposites at room temperature were assessed using an Instron 8801 MTL5499, a digitally controlled servo-hydraulic fatigue system. For metallographic studies, composites were polished with 200–1,000 grit emery papers and etched using Keller's reagent. Field emission scanning electron microscope (FESEM) micrographs, SEM-EDS analysis, and elemental mapping were performed using a TESCAN MIRA.

3 Result and discussion

3.1 XRD analysis

Figure 3(a) illustrates the XRD patterns of nanocomposites with 0, 0.5, 1, 1.5, and 2 wt% ZrB_2 fabricated through PM. Al and ZrB_2 peaks were identified in the composites, and

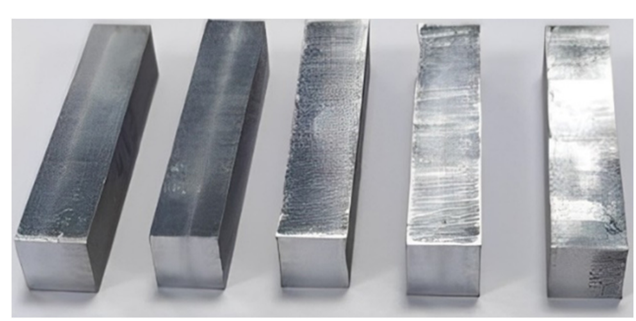


Figure 1: Final fabricated composites.

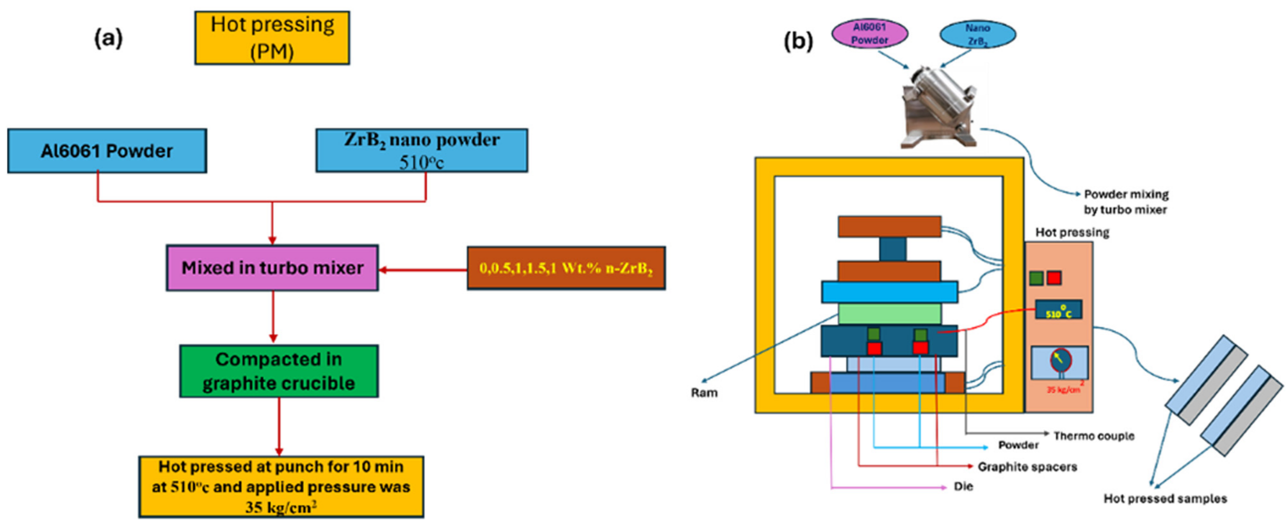


Figure 2: (a) Flowchart of hot pressed processes for Al 6061-ZrB_2 nanocomposites fabrication. (b) Hot-pressed unit setup.

there are no peaks from the other phases, which indicates the absence of adverse chemical responses between phases. Figure 3(b) shows the increasing weight percentage of ZrB_2 in the Al6061 matrix results in a noticeable reduction in the average crystallite size of the composite. This reduction is attributed to the ZrB_2 nanoparticles acting as nucleation sites, promoting grain refinement during solidification. As the ZrB_2 concentration rises, these particles effectively inhibit the grain growth by pinning the grain boundaries, leading to a finer microstructure. This refined grain structure enhances the mechanical properties, including strength and hardness, as smaller grains provide more grain boundaries that obstruct the dislocation movement, thus strengthening the material.

3.2 Porosity

Porosity: The pores in the composites are expressed in % of the total volume of the part. It affects the mechanical properties. The porosity was calculated by the following formula and presented in Table 2.

$$\text{Porosity}(\%) = [1 - \text{DR}] \times 100, \tag{1}$$

where DR is the relative density.

From the previous study, it was observed that the increasing ZrB_2 wt% from 0 to 2 in hot pressing increases the density [11]. Figure 4 shows that 1.5 wt% of ZrB_2-Al composites exhibits more porosity, which is due to clusters.

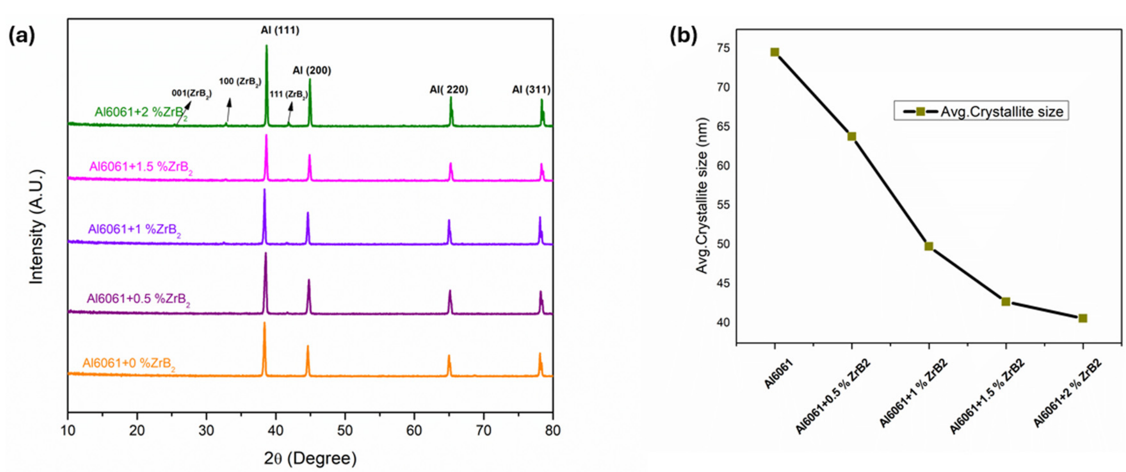


Figure 3: (a) XRD. (b) Average crystallite size of Al6061-ZrB_2 nanocomposites.

Table 2: Values indicating the porosity of composites

Composition	Porosity (%)
Al6061	3.73
Al6061 + 0.5% ZrB_2	3.57
Al6061 + 1 % ZrB_2	1.7
Al6061 + 1.5% ZrB_2	3.9
Al6061 + 2% ZrB_2	2.17

3.3 Microstructural studies

The distribution of the reinforced particles, the existence of pores, and agglomeration in the composites are determined by microstructural analysis, and these factors have a significant impact on the mechanical and physical characteristics of the composites. Figure 5 illustrates the FESEM micrographs of

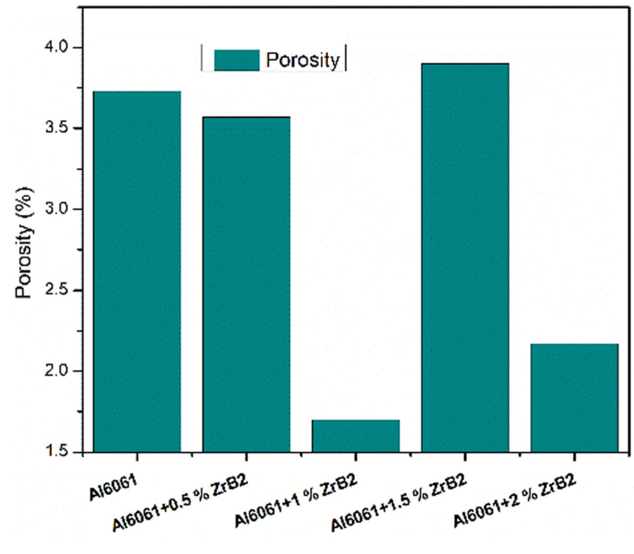


Figure 4: Porosity of Al 6061-ZrB_2 nanocomposites.

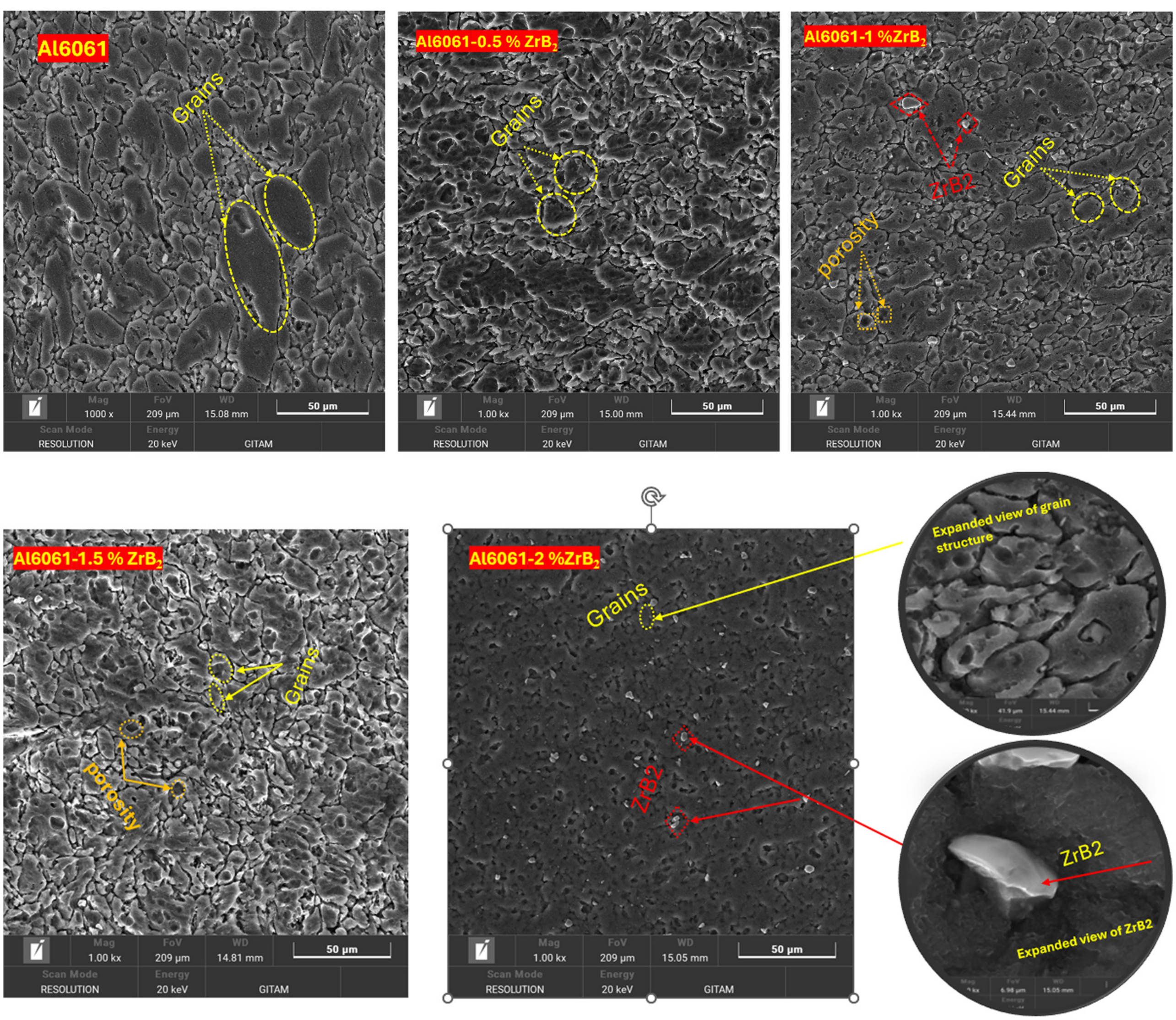


Figure 5: FESEM images of Al6061-ZrB_2 nanocomposites.

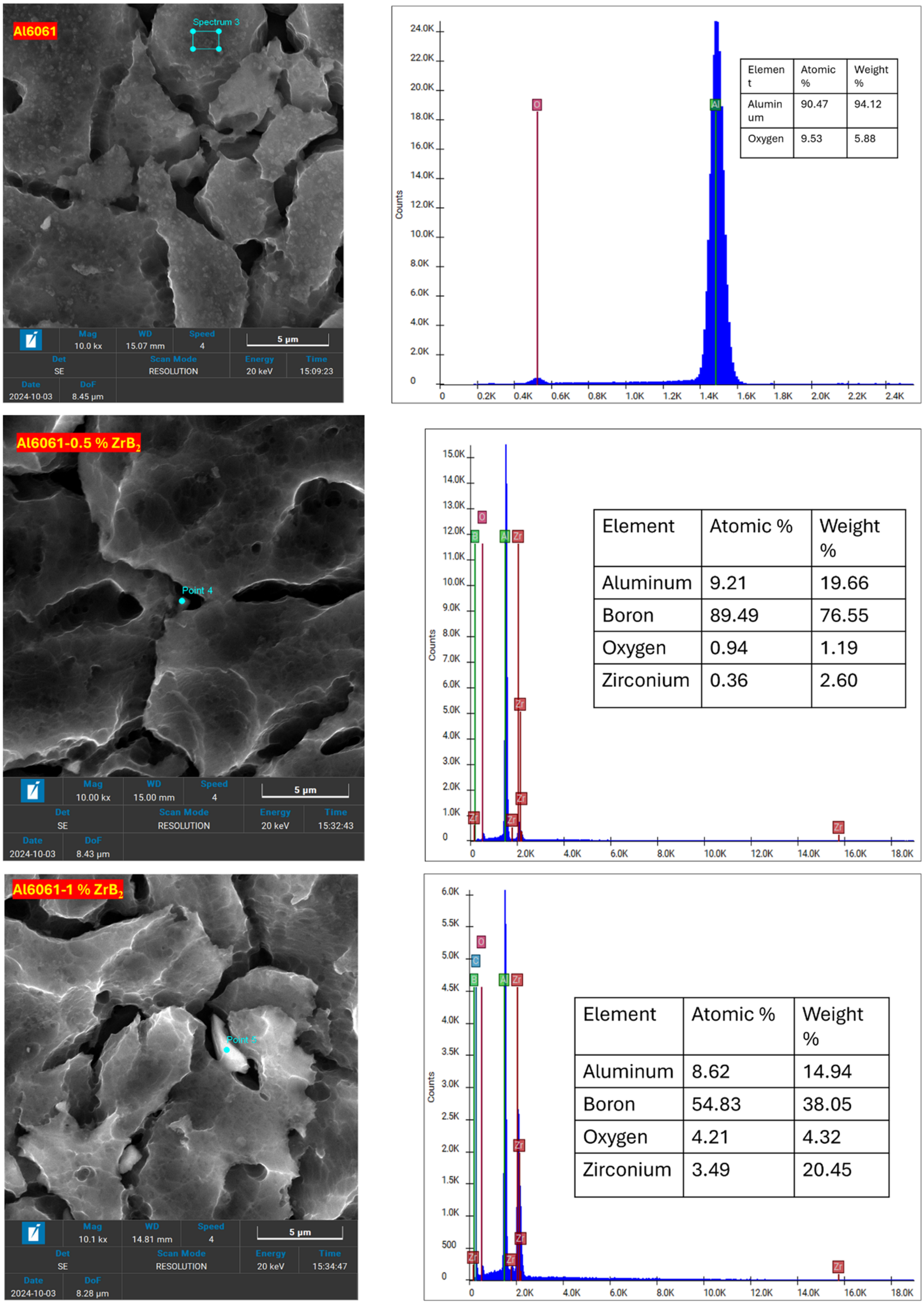
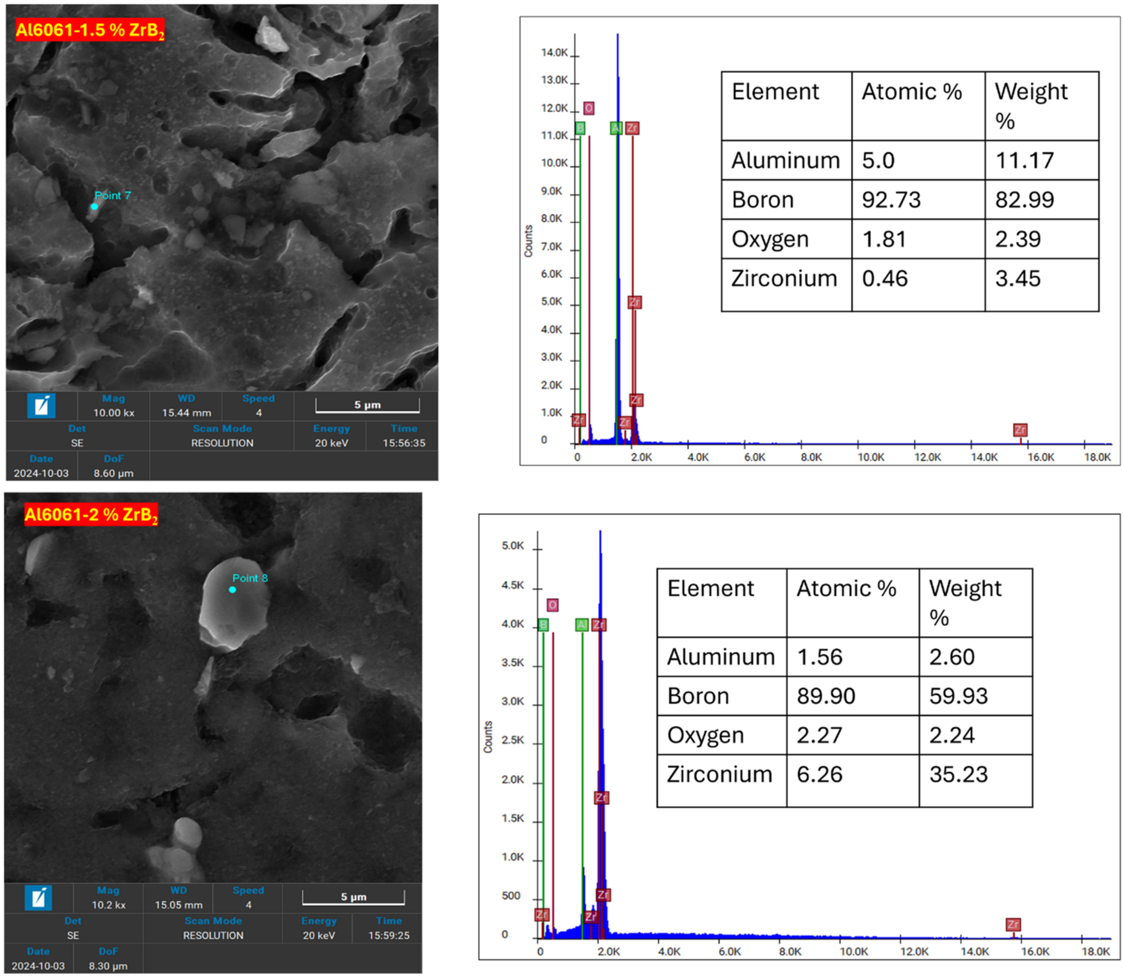


Figure 6: SEM EDS of Al6061-ZrB_2 nanocomposites.



Element	Atomic %	Weight %
Aluminum	5.0	11.17
Boron	92.73	82.99
Oxygen	1.81	2.39
Zirconium	0.46	3.45

Element	Atomic %	Weight %
Aluminum	1.56	2.60
Boron	89.90	59.93
Oxygen	2.27	2.24
Zirconium	6.26	35.23

Figure 6: (*Continued*)

Al6061 and Al/ZrB_2 nanocomposites. The SEM images clearly illustrate the even distribution of the matrix and ZrB_2 reinforcements within the aluminum matrix. The light gray areas represent the aluminum matrix, while the white spots and clusters indicate the presence of ZrB_2 particles. As nano ZrB_2 is added to Al6061 alloy in amounts ranging from 0.5 to 2 wt%, grain size decreases as shown in Figure 5, resulting in improved mechanical properties.

In Figure 6, energy-dispersive X-ray analysis revealed that the composite did not contain any reactive products. Furthermore, EDS analysis showed that ZrB_2 nanoparticles were well dispersed throughout the Al6061 alloy matrix. In contrast, sintered composites exhibited agglomeration of nano ZrB_2 and the presence of oxide layers, indicating surface and interface contamination. These oxide layers weaken the bond between the reinforcement and the matrix at contact areas, leading to poorer mechanical properties [14].

In Figure 7(a), elemental mapping with green and pink colors indicates the distribution of Al and zirconium in the composite, likely showing that the ZrB_2 reinforcement has been well dispersed within the Al6061 matrix. The fact that both boron (from ZrB_2) and zirconium are clearly visible suggests that the ZrB_2 has not decomposed or reacted undesirably during processing, supporting the suitability of the hot pressing.

3.4 Micro-hardness

Figure 8 illustrates the micro-Vickers hardness results of the Al6061-ZrB_2 composites. Compared to the base alloy, the Al6061-ZrB_2 composites have a substantially greater microhardness. During solidification, these ZrB_2 particles serve as nucleation regions for new grains and will support grain boundaries. With the inclusion of ZrB_2 particles from 0 to 2 wt%, there is an increase in microhardness by restricting the dislocation movement [15]. In this work,

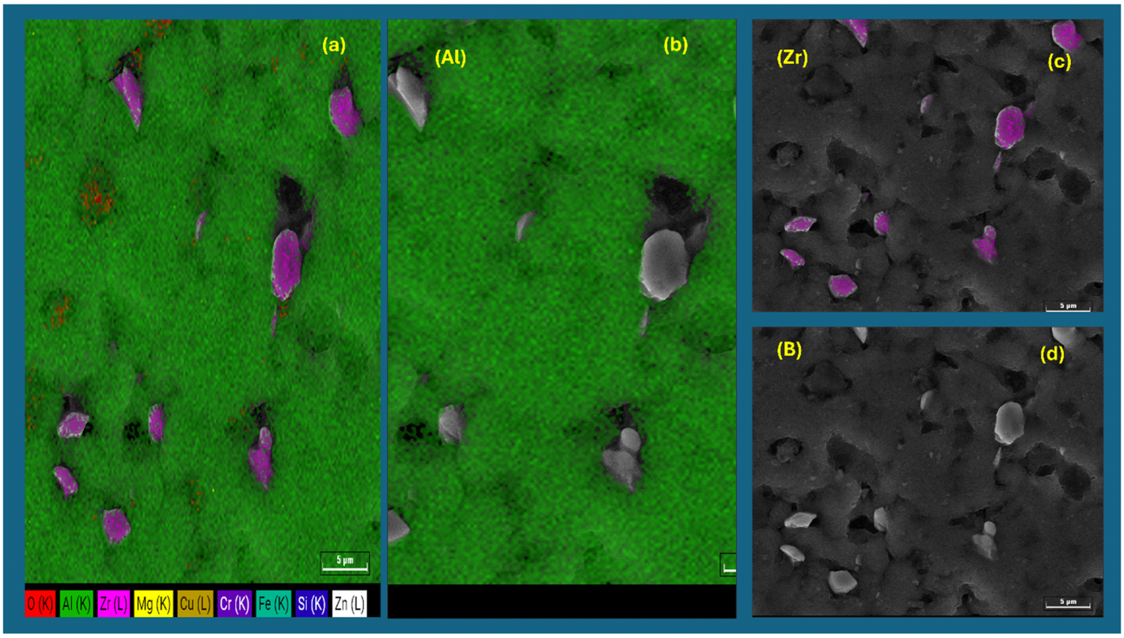


Figure 7: Elemental mapping of Al6061-2 wt% ZrB_2.

ZrB_2 serves as a load-bearing element, absorbing the greatest amount of load for plastic deformation by increasing its hardness [16]. When the ZrB_2 reinforcement content in the Al6061 alloy was increased from 0 to 2 wt%, there is a significant increase in microhardness (Table 3). The maximum hardness achieved was 34 ± 1 HV at 2 wt% of ZrB_2 [17]. The findings show that Al6061 alloy with 2 wt% ZrB_2 reinforcement had better surface hardness [18]. It might be because the Al alloy matrix's pores and spaces were filled with ZrB_2 particles [19].

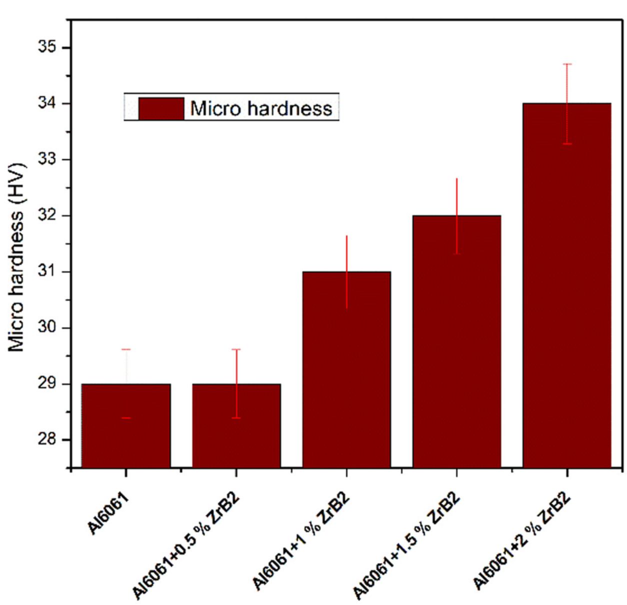


Figure 8: Microhardness of Al 6061-ZrB_2 nanocomposites.

3.5 Compression test

The compressive strength of Al-ZrB_2 nanocomposites is greater than the base alloy represented in Figure 9(b), demonstrating the importance of ZrB_2 nanoparticles in the prevention of grain growth, which improve the mechanical properties of composites when compared to unreinforced Al6061 alloy samples [20]. In addition, the compressive strength of the Al6061-ZrB_2 nanocomposites increased as more ZrB_2 content was added. The Al6061 nanocomposite with 2 wt% ZrB_2 nanoparticles demonstrated a significant improvement in compressive strength (65 ± 2 MPa). The elastic characteristics are significant in a material's deformation behavior. ZrB_2 has a higher elastic constant than Al, which prevents plastic deformation of the Al matrix and

Table 3: Values indicating the microhardness of fabricated composites

Composition	Vickers microhardness (HV)
Al6061	29 ± 2
Al6061 + 0.5% ZrB_2	29 ± 3
Al6061 + 1 % ZrB_2	31 ± 4
Al6061 + 1.5% ZrB_2	32 ± 3
Al6061 + 2% ZrB_2	34 ± 1

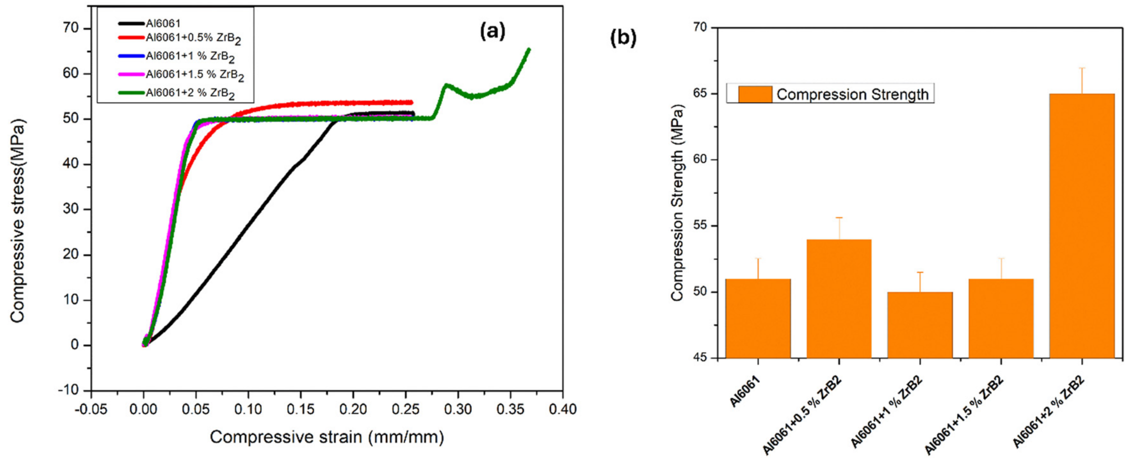


Figure 9: (a) Compressive strain and compressive stress. (b) Compressive Strength of Al 6061-ZrB_2 nanocomposites.

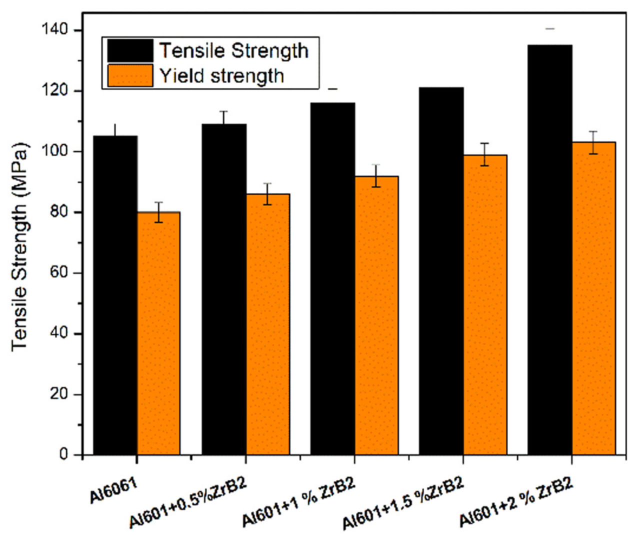


Figure 10: Tensile strength and yield strength of Al 6061-ZrB_2 nanocomposites.

thus boosts the compressive strength of the composite [3]. The enhancement in the strength and hardness of the composites can be attributed to several strengthening mechanisms such as the orowan strengthening mechanism, the dispersion hardening effect, and the load transfer mechanism [21].

Figure 9(a) displays the compressive stress–strain curves, which rise as the nanoparticle content increases the material's strength. The curves show that the compressive strength of the nanocomposites increases with higher nanoparticle content. Specifically, adding 2 wt% of nanoparticles boosts the nanocomposites' strength by 21.5% compared to the base alloy. The effective interfacial interaction between nanoparticles and the matrix, promoted by ultrasonication, leads to grain refinement. This grain refinement increases the grain boundary area through the pinning effect of nanoparticles, where these boundaries impede dislocation motion during deformation, thereby enhancing the strength of the nanocomposites [22]. Another reason for enhancement in composite's tensile strength is well-distributed nanoceramic particles and reduced porosity. The use of the powder metallurgy technique results in a homogeneous distribution of ZrB_2 particles, reduces air gaps between grains, and causes the low degree of porosity. In addition, thermal stress and high multidirectional grain refinement at the aluminum/ZrB_2 interface are significant elements that enhance the strength of the composites [23].

3.6 Tensile strength and Yield strength (YS)

Figure 10 shows how the YS and UTS of Al6061 alloy and Al6061-ZrB_2 composites vary. Increasing the ZrB_2 wt% in

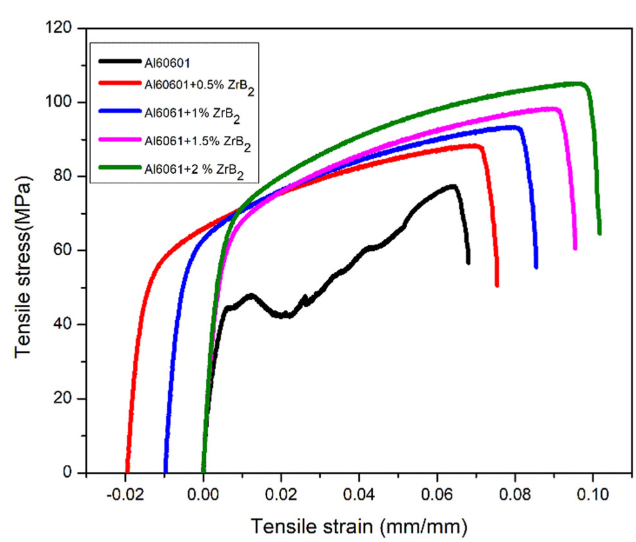


Figure 11: Stress and strain curve of Al 6061-ZrB_2 nanocomposites.

base alloy increased the UTS while decreasing the ductility percentage [16]. Increasing ZrB_2 wt% resulted in the ascending trend of yield strength and ultimate tensile strength from 0 to 2 wt% of ZrB_2. The higher ZrB_2 particle concentration will improve the particles' interaction with the matrix, which will boost UTS [24]. ZrB_2 and Al6061 have different thermal expansion coefficients, which causes dislocations to develop around the ZrB_2 particles during solidification. Results in higher stress are required to initiate cracks [25–27].

Figure 11 represents the stress and strain curve for Al 6061-ZrB_2 nanocomposite. The AA6061-2% ZrB_2 nanoparticles composite have ultimate strength and higher yield than pure AA6061. As the ZrB_2 content increases, there is a notable rise in tensile strength. The highest increase in strength is observed with 2 wt% ZrB_2, and this enhancement can be attributed to the effective load transfer, dispersion hardening, and grain refinement mechanisms facilitated by the nanoparticles. Although tensile strength increases with higher nanoparticle content, the ductility or strain-to-failure decreases slightly, indicating a trade-off between strength and ductility.

The consistent improvement in tensile stress with the increased ZrB_2 content suggests effective bonding and dispersion of nanoparticles within the Al6061 matrix, enhancing the composite's ability to withstand higher stresses before failure.

3.7 Ductility

Ductility decreases as reinforcement wt% increases. Figure 12 shows that when the ZrB_2 level in the matrix alloy increased, the ductility of the composite decreased. Increased reinforcing may reduce ductility due to the existence of hard ZrB_2 particles and grain refinement. The elongation declined due to the addition of ZrB_2 reinforcement increases and caused the crack initiation [28,29]. Ductility declined due to the existence of hard ZrB_2 particles present in the composites and grain refinement [23].

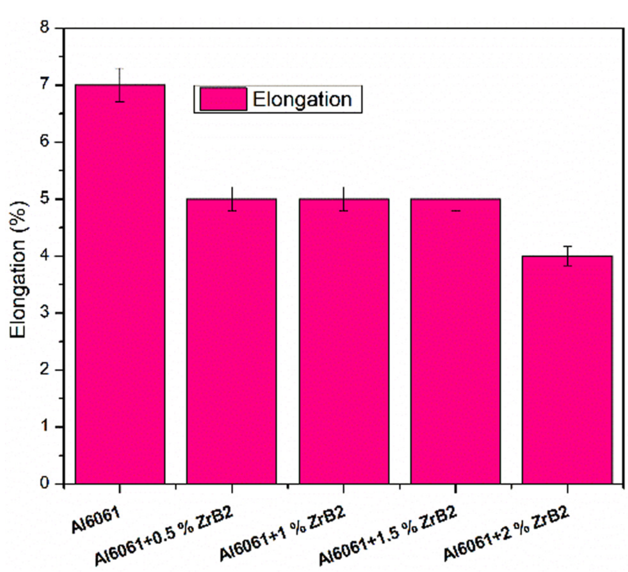


Figure 12: Ductility of Al 6061-ZrB_2 nanocomposites.

4 Conclusions

In the present study, the hot pressing route was used to fabricate the aluminum metal matrix composites. The powder metallurgy method has enhanced the Al 6061 alloy properties with the incorporation of the ZrB_2 reinforcement.

The following points were concluded from composites fabricated through the PM route:

- The powder metallurgy technique significantly refines the grain structure, ensuring uniform distribution of nanoparticles and effective interfacial bonding between the nanoparticles and the matrix powder.
- SEM analysis confirms that the aluminum matrix and ZrB_2 reinforcements are uniformly distributed throughout the composite and confirm that the grain size decreases with the addition of ZrB_2 ranging from 0 to 2 wt%.
- Increasing the ZrB_2 content in the Al6061 matrix reduces the average crystallite size, as ZrB_2 particles act as nucleation sites and inhibit the grain growth. This refined microstructure enhances the composite's strength and hardness by introducing more grain boundaries that limit the dislocation movement.
- The microhardness of the nanocomposites increased significantly from 29 ± 2 HV in the Al6061 alloy to 34 ± 1 HV in the Al6061-2 wt% ZrB_2 nanocomposite. This represents a 17% improvement in microhardness for the Al6061-2 wt % ZrB_2 composite compared to the unreinforced Al6061 alloy.
- The Al/ZrB_2 nanocomposite with 2 wt% ZrB_2 exhibited a tensile strength of 135 ± 3 MPa, a yield strength of 103 ± 4 MPa, and a compressive strength of 65 ± 2 MPa, marking respective improvements of 22.2, 22.3, and 21.5% over the unreinforced Al6061 alloy.
- Among all fabricated composites, the Al6061-2 wt% ZrB_2 composite demonstrates superior mechanical properties compared to the base alloy.

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Data availability statement: All data generated or analyzed during this study are included in this published article.

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