Mechanical and microstructural investigation of aluminum composites reinforced with boron carbide and zirconium

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Abstract. The current study investigates how the addition of zirconium (Zr) influences the mechanical and microstructural properties of composites consisting of aluminum and boron carbide (Al–B_4C) produced by powder metallurgy. To fabricate hybrid composites with improved mechanical properties, Al powder was reinforced with 7 wt% B_4C and additions of Zr in the range of 1–5 wt%. To achieve a uniform metallurgical bonding, the mixed powders were compacted and sintered for 2 h at 600 °C under an argon atmosphere. The influence exerted by Zr on impact energy, hardness, compressive strength, and densification has been investigated, and the results have been correlated to the microstructural features obtained using SEM. The results indicated that the addition of Zr significantly improved the densification and bonding characteristics between B_4C particles and the Al matrix. Indeed, porosity decreased from 2.5% (Al–7% B_4C) to 1.9% (Al–7% B_4C–4% Zr), suggesting improved diffusion and wettability at the reinforcement–matrix interface. Grain refinement and dispersion strengthening were promoted by the formation of fine Al3Zr dispersoids, which favored a gradual enhancement of the mechanical properties. For 4 wt% Zr, Vickers hardness and compressive strength reached maximum values of 102 VHN and 285 MPa, respectively, and the impact energy also improved with 6.8 J, showing a good compromise between toughness and strength. Particle agglomeration and development of pores were held responsible for the slight deterioration of properties beyond 4 wt% Zr.

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1 Introduction

In contemporary engineering applications where high specific strength, stiffness, and thermal stability are crucial, lightweight structural materials are crucial. Because of its low density, superior corrosion resistance, high electrical and thermal conductivity, and simplicity of production, aluminum (Al) and its alloys have drawn a lot of interest in the aerospace, automotive, marine, and military sectors [1]. However, monolithic aluminum's mechanical strength, hardness, and wear resistance are inadequate for harsh service environments, especially when high loads and high temperatures are present. In order to get over these intrinsic restrictions, scientists have concentrated on using ceramic particles to reinforce the aluminum matrix in order to create aluminum matrix composites (AMCs) that have a desirable blend of high mechanical qualities and lightweight design [2].

In the last several decades, AMCs have become a potential type of advanced material because their microstructures can be changed and their performance can be tuned. Researchers have looked into adding strong ceramic reinforcements including silicon carbide (SiC), boron carbide (B_4C), alumina (Al_2O_3), and titanium carbide (TiC). Boron carbide (B_4C) is one of the best reinforcements for aluminum because it is very hard (~30 GPa), has a low density (2.52 g/cm³), a high melting temperature (2763 °C), and is very stable chemically. Because of these qualities, B_4C-reinforced aluminum composites are good for use in armor panels, brake systems, aerospace parts, and parts that need to be resistant to wear. Adding B_4C particles makes aluminum composites much harder, more flexible, and more resistant to wear [3].

Even though B_4C makes a better mechanical contribution, using it in Al matrices might be hard to work with. The low wettability between B_4C and molten aluminum typically causes the particles to not be evenly distributed, the bonds between them to be weak, and holes to emerge throughout the manufacturing process. Furthermore, the reaction between Al and B_4C at elevated temperatures may form brittle interfacial compounds such as Al_4C_3 and AlB_2. These compounds reduce the strength of the composites and lower their corrosion resistance. Therefore, it has been investigated to incorporate alloying elements or other reinforcements into Al–B_4C composites in order to achieve better bonding and wetting and hence to attain an overall optimum combination of strength and ductility [4].

In this scenario, zirconium (Zr) has been found to be a very useful additive for aluminum-based composites. Zirconium has excellent solid solubility in aluminum and forms the thermally stable intermetallic complexes with the matrix, such as Al_3Zr. The formed Al_3Zr phase acts as a dispersoid and thus prevents the grain growth during both solidification and subsequent heat treatments. These fine dispersoids pin the grain boundaries, which in turn helps in refining the grains. This is an essential step in strengthening and toughening the material by the Hall–Petch process. Zr also facilitates heterogeneous nucleation during solidification and hence makes the microstructure more refined and uniform. This enables the homogeneous dispersion of ceramic reinforcements like B_4C within the aluminum matrix [5].

Another significant advantage of the addition of Zr is that it further strengthens the interfacial bonding between Al and B_4C. Zr induces diffusion-controlled reactions at the interface, which strengthens the bond without forming too much brittle reaction layer. The rate of interface reaction is altered by Zr, thereby hindering the formation of undesirable Al_4C_3. Due to this, load transmission from the matrix to the reinforcement becomes far more effective. Zr also acts to stabilize the oxide layer present on the aluminum surface, thereby reducing the number of oxide-related defects and improving the kinetics of densification during compaction and sintering [6].

From a mechanical viewpoint, the combined addition of B_4C and Zr improves the properties in a synergistic manner. The composite becomes harder because of the hardness of B_4C and finer Zr-rich particles precipitating to improve strength by dispersion. The compressive strength rises owing to successful stress transfer from the soft Al matrix into the hard reinforcement particles. Toughness remains comparable or slightly improved due to grain refinement and improved interfacial cohesion. The Zr also improves thermal stability of the composite, thereby sustaining mechanical integrity at elevated temperatures. Therefore, adding Zr to Al–B_4C systems forms a better way to achieve an optimal balance between strength, ductility, and toughness. This is a property combination which is rather difficult to achieve with the normal particle-reinforced composites [7].

Besides mechanical advantages, microstructural observations are essential in understanding the causes of strengthening and failure. SEM provides useful information on reinforcement distribution, porosity, surface interaction, and agglomeration. How these microstructural elements influence mechanical behavior has to be understood to arrive at the optimum processing conditions and reinforcement combination [8].

In the context of these benefits, this study focuses on the synthesis and characterization of aluminum composites reinforced with B_4C and Zr powders. It addresses issues on the influence of Zr addition on microstructure, distribution of reinforcement particles, and the mechanical properties-microstructural relationship [9]. Compression, hardness, and impact strength tests have all been widely conducted in establishing structure-property relationships. Correlative SEM analyses were performed to explain the synergistic role of ceramic-metallic reinforcements in enhancing performance in these hybrid composites of aluminum [10].

2 Materials and methods

2.1 Raw materials

The matrix material utilized in this investigation is commercially pure aluminum (Al) powder with an average particle size of around 45 µm and a purity of 99.7%. Boron carbide (B_4C) and zirconium (Zr) powders are the chosen materials for reinforcement. The B_4C powder came from a chemical source with a high level of purity. It had an average particle size of 20 µm and a purity level of 98%. The Zr powder had an average particle size of 15 µm and a purity level of 99.5%. Both reinforcements were utilized as they were, without any further surface treatment, to keep their original properties [7,10,11].

We chose B_4C as the main ceramic reinforcement because it has a low density (2.52 g/cm^3), is very hard, and is very resistant to wear. This makes it very good at making aluminum composites stronger and harder. Adding Zr as an additional metallic reinforcement was meant to make the particles and matrix more wettable, increase the bonding between them, and make the grains finer by forming fine Al_3Zr dispersoids during sintering [12].

Three different composite compositions were prepared by varying the weight percentages of Zr while keeping the B_4C content constant, as shown in Table 1. The unreinforced aluminum sample was also fabricated as a reference for comparison.

Table 1. Nomenclature of composites fabricated in the present work.

Sample Code	Reinforcement Composition (wt%)	Remarks
Al	0 B_4C + 0 Zr	Base Alloy
Al–B	7 B_4C + 0 Zr	Single Reinforced
Al–B–Zr1	7 B_4C + 1 Zr	Hybrid Composite – 1% Zr
Al–B–Zr2	7 B_4C + 2 Zr	Hybrid Composite – 2% Zr
Al–B–Zr3	7 B_4C + 3 Zr	Hybrid Composite – 3% Zr
Al–B–Zr4	7 B_4C + 4 Zr	Hybrid Composite – 4% Zr
Al–B–Zr5	7 B_4C + 5 Zr	Hybrid Composite – 5% Zr

The accurately weighed powders of Al, B_4C, and Zr were blended using a planetary ball mill to ensure uniform dispersion of reinforcements within the aluminum matrix. The mixing was carried out at a speed of 200 rpm for 2 hours under a toluene medium to prevent cold welding and oxidation of aluminum particles. Tungsten carbide balls were used as the grinding media with a ball-to-powder weight ratio (BPR) of 10:1. The milling process was intermittently paused to avoid excessive heating and to ensure homogeneous blending. After mixing, the powder mixture was dried in a vacuum oven at 80 °C for 2 hours to remove residual moisture and volatile substances. The homogeneity of the blended powder was verified through microscopic observation before compaction.

2.2 Compaction and sintering

The homogenized powder mixtures were compacted using a uniaxial hydraulic press to form cylindrical green compacts. The powders were pressed at a pressure of 450 MPa using a hardened steel die to achieve a uniform and dense preform. Each compact had dimensions of 10 mm diameter and 15 mm height.

The green compacts were then subjected to sintering in a controlled atmosphere furnace to achieve metallurgical bonding between the particles. Sintering was carried out at 600 °C for 2 hours in an argon atmosphere to prevent oxidation. The heating rate was maintained at 10 °C/min, and the samples were furnace-cooled to room temperature to minimize thermal stresses and crack formation. The sintering temperature was carefully chosen below the melting point of aluminum to retain structural integrity while enabling sufficient diffusion and reaction between the aluminum matrix and reinforcements [13].

2.3 Mechanical tests

2.3.1 Hardness test

The Brinell hardness test was performed on the polished composite specimens using a ball indenter of 5 mm diameter under a load of 250 kgf for 15 s dwell time. The reported hardness value for each composition is the average of five readings taken at different locations to account for microstructural inhomogeneities.

2.3.1 Compression test

Compression testing was conducted on a universal testing machine (UTM) as per ASTM E9 standards. Cylindrical specimens with an aspect ratio of 1.5:1 were loaded at a constant crosshead speed of 1 mm/min until failure. The ultimate compressive strength and yield strength were determined from the corresponding stress–strain curves.

2.3.2 Impact test

Impact energy was measured using a Charpy impact testing machine on notched specimens following ASTM E23 standards. The average of three test values was reported for each composite to ensure reliability of the results.

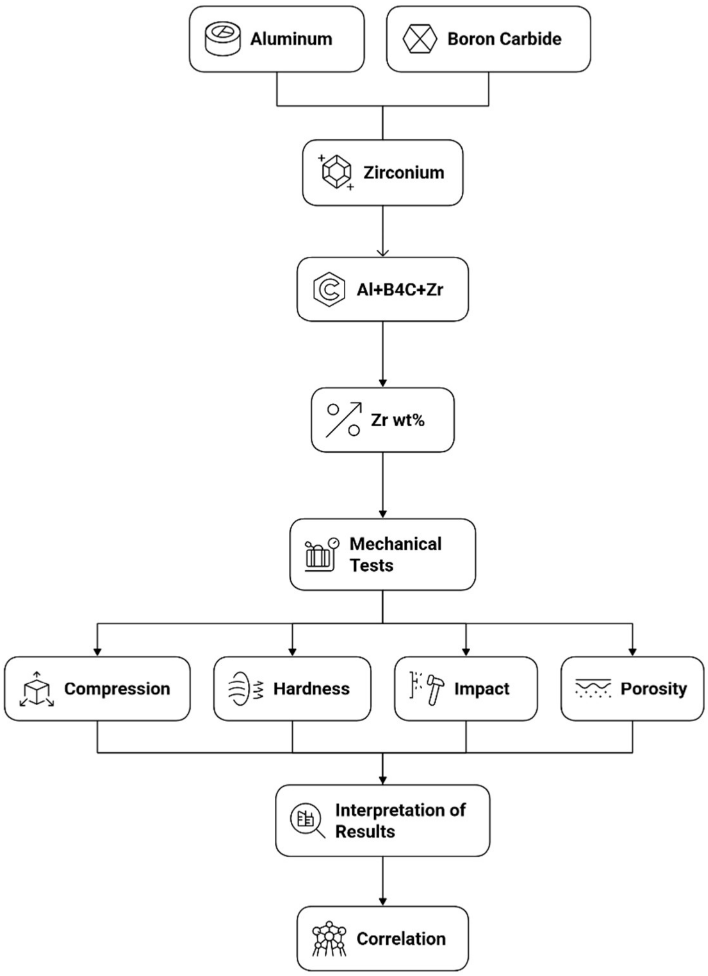

Fig. 1. Likely schematic or fabrication sequence of Al–B_4C–Zr composites.

3 Results and discussions

Figure 1 shows a diagram of the process used to make the Al–B_4C–Zr composites. The procedure included mixing powders, compacting them, and sintering them in a controlled environment to make sure that the reinforcement was evenly distributed and the metals were bonded together. The composites that were made had high structural integrity and very little surface oxidation, which showed that the powder metallurgy method works well for making Al-based hybrid composites. Figure 2 shows the microstructural study of the sintered specimens. It shows clear characteristics that match the distribution of the reinforcement and the shape of the pores. Figure 2(a) shows that the unreinforced aluminum area has an oxide layer and a few isolated pores that developed during sintering. These oxide layers make it harder for particles to bind with each other, which causes microvoids to develop. Figure 2(b), on the other hand, shows that dark B_4C and brilliant Zr particles are evenly spread throughout the aluminum matrix. The strong link between the reinforcements and the matrix shows that the interfaces are more wettable and diffusable, which is likely due to the presence of Zr. Figure 2(c) shows much more clearly that a few deep pores formed, which might have happened because gasses were trapped during sintering. But the fact that there aren't many of them and they're not very big suggests that densification is going well. Adding Zr to the mix made the matrix structure much better and cut down on oxide inclusions. This improved the bond between the aluminum and B_4C particles [14].

Figure 3(a) depicts the variation of porosity for the composites fabricated with Zr. The porosity of pure aluminum was about 1.8% and increased to 2.5% with the addition of 7 wt% B_4C due to poor wettability between ceramic reinforcement and metallic matrix. The porosity value then gradually dropped to 2.3%, 2.1%, 2.05%, and 1.9% with 1 wt%, 2 wt%, 3 wt%, and 4 wt% additions of Zr, respectively. This is attributed to the generation of fine-sized Al_3Zr intermetallic dispersoids which act as nucleation sites during sintering, enhancing diffusion bonding and reducing voids. Furthermore, Zr addition makes the contact between Al and B_4C more wettable, thereby enhancing packing and densification. With 5 wt% Zr, however, there was a slight increase in porosity to 2.4%, probably because Zr particles agglomerated and their diffusion was restricted during sintering. This indicates that around 4 wt% Zr is the optimum concentration for maximum densification and structure stability [15].

Figure 3(b) illustrates the effects of adding Zr on hardness and compressive strength. With the increase in Zr content to 4 wt%, the Vickers hardness and compressive strength increased steadily. Beyond this, both decreased slightly at 5 wt%. The Vicker's hardness of the unreinforced aluminum test sample is 47 VHN, and the compressive strength is 155 MPa. These are the basic parameters of ductile aluminum. Addition of 7 wt% B_4C enhanced these properties to 78 VHN and 228 MPa, respectively, as hard ceramic particles can support weight. Further addition of Zr resulted in an improvement in the mechanical properties of the composites. Hardness increased from 83 VHN for 2 wt% Zr to 102 VHN for 4 wt% Zr, and compressive strength increased from 242 MPa to 285 MPa accordingly. The major reason for this lies in the synergistic effect of the reinforcing materials, B_4C and Zr. While Zr refines the grains and strengthens the matrix through solid-solution and dispersion hardening mechanisms, B_4C assists in load transfer and dislocation blocking. As a result, the microstructure at 4 wt% Zr exhibited a uniform and finer grain morphology that imparted maximum hardness and compressive strength to the composite material. This slight deterioration of both the properties at 5 wt% Zr (97 VHN, 272 MPa) is most probably due to Zr particles that aggregated in certain regions, making the material more porous and hence incapable of efficient load transfer.

Figure 3(c) presents the variation of impact energy with the addition of Zr, showing thereby the effect of Zr on toughness of the composites. Pure aluminum exhibited the highest impact energy value of 7.2 J, which exhibited that the material is ductile. Addition of B_4C

reduced the impact energy to 5.9 J as the ceramic reinforcement is brittle. However, the hardness gradually improved upon the addition of Zr. Impact energy gradually increased from 6.0 J at 1 wt% Zr to 6.8 J at 4 wt% Zr, indicating the addition of Zr imparts more ductility to the materials and the material can absorb more energy. Improved microstructure and better interfacial bonding were responsible for such improvement. These allowed better stress distribution during impact loading. Beyond 4 wt% Zr, there was a slight decline in energy value (6.3 J) due to agglomeration and micro-voids acting as initiation sites for the cracks.

The combined effect of porosity, hardness, compressive strength, and impact energy indicates that the addition of Zr plays a very significant role in enhancing both the microstructure and mechanical properties of Al-B_4C composites. In this regard, the Al_3Zr dispersoids produced during sintering promote increased grain refinement and reduced porosity, hence improving interfacial bonding. So, composites with 4 wt% Zr have the highest overall performance, with high hardness (102 VHN), compressive strength (285 MPa), and moderate toughness (6.8 J), and very little porosity (1.9%). The little drop in characteristics at higher Zr levels (>4 wt%) is due to particle agglomeration and localized pore development, which make bonding less effective. So, a Zr content of around 4 wt% is found to be the best for getting a good mix of strength and ductility in Al–B_4C–Zr hybrid composites.

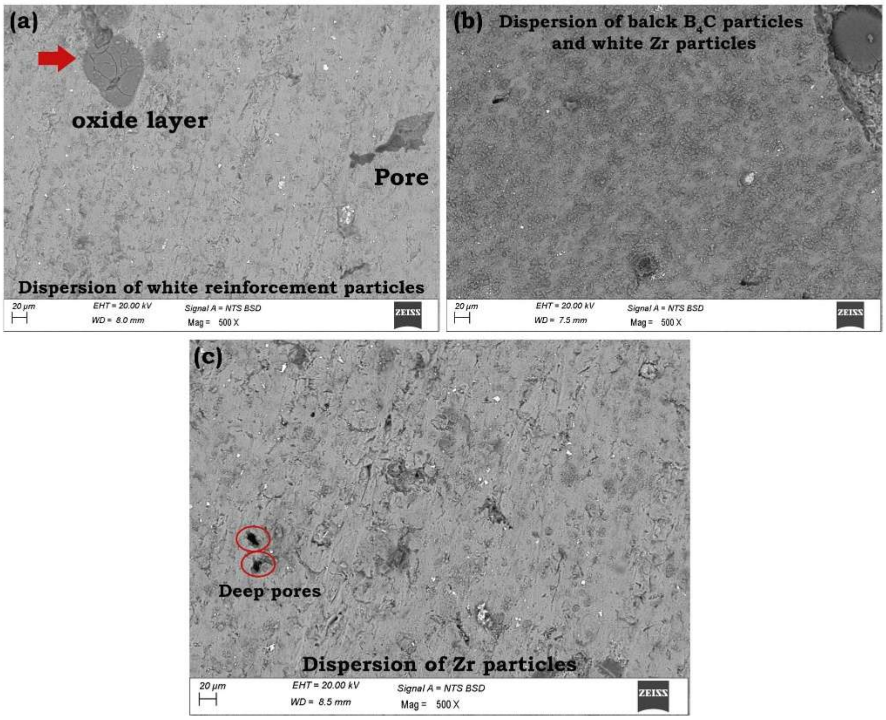

Fig. 2. SEM micrographs of Al–B_4C–Zr composites at 500× magnification: (a) Oxide layer and pore in unreinforced region; (b) Uniform dispersion of black B_4C and white Zr particles; (c) Deep pores observed in sintered specimen showing dispersion of Zr particles.

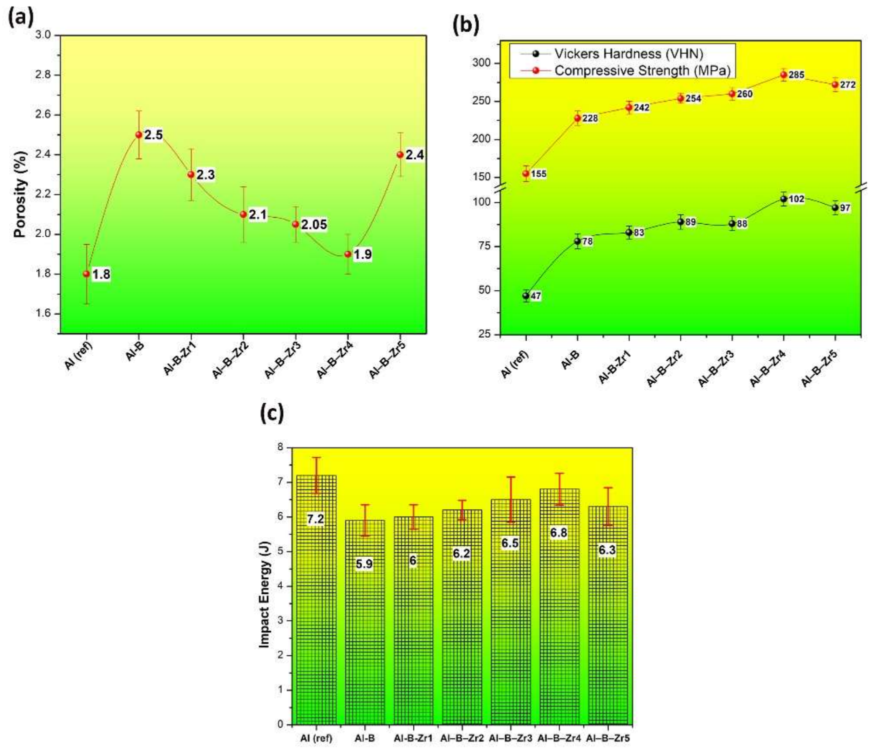

Fig. 3. (a) Variation of porosity (%) with Zr addition; (b) Variation of Vickers hardness (VHN) and compressive strength (MPa) with Zr content; (c) Variation of impact energy (J) for Al–B_4C–Zr composites.

4 Conclusions

In the present investigation, aluminum-based hybrid composites reinforced with boron carbide (B_4C) and zirconium (Zr) powders were successfully fabricated using the powder metallurgy technique. The influence of Zr addition (1–5 wt%) on the densification behavior, hardness, compressive strength, and impact properties of Al–7%B_4C composites was systematically examined and correlated with microstructural observations.

- The SEM analysis confirmed that the incorporation of B_4C and Zr particles in the aluminum matrix was uniformly dispersed, with minimum porosity at the intermediate contents of Zr. Zr promoted the formation of fine Al_3Zr intermetallic dispersoids, which took part in grain refinement and improved interfacial bonding between the matrix and reinforcement. Porosity progressively decreased from 2.5% in Al–7%B_4C to 1.9% in Al–7%B_4C–4%Zr, evidencing enhanced densification and wetting.
- Mechanical characterization showed that there was a great enhancement in hardness and compressive strength with the addition of Zr up to 4 wt%. Accordingly, the Vickers hardness increased from 78 VHN for Al-B_4C to 102 VHN for Al-B_4C-4%Zr, while the compressive strength increased from 228 MPa to 285 MPa. Impact energy also increased from 5.9 J to 6.8 J at the same Zr addition, reflecting enhanced toughness due to refined microstructure along with strong interfacial cohesion.
- Therefore, the Al–7%B_4C–4%Zr composite showed the best balance between strength, hardness, and impact resistance, validating the synergistic strengthening

effect of B_4C and Zr reinforcements. In addition, excess Zr (>4 wt%) induced slight deterioration, associated with agglomeration and pore formation. Accordingly, controlled addition of Zr (~4 wt%) is found to be optimal in achieving the maximum structural performance of Al–B_4C composites.

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