



Investigations on physical, mechanical and metallurgical characteristics of ZK60/ZrB₂ composites produced by stir casting route

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ABSTRACT

This research used Stir Casting [SC] technique through liquid metallurgy route to exert the ZK60-ZrB₂ composites. The dispersoid combination varies in increments of 4 weight percent ZrB₂ particles, from 0 to 12 weight percent for the study. We investigated the mechanical characteristics of the produced composites, including their compressive, flexural, impact, tensile strengths and macro hardness, as well as metallurgical properties like micro vickers hardness and micrographs like Optical Microscopy [OM] and Scanning Electron Microscopy [SEM] were done. We also measured a physical characteristic like density for further evaluation. Prepared and evaluated test specimens in accordance with ASTM standards. An increase in ZrB₂ reinforcement improves the mechanical, metallurgical, and physical characteristics overall, according to the data obtained. From the inferred results the increase in percentage of density, microhardness, macrohardness, tensile, impact, compression and flexural strength are 35%, 30.37%, 51.02%, 37.5%, 140%, 41.66% an 56.97% of values. The OM and SEM examinations confirmed that the ZrB₂ particles were spread out evenly in the ZK60 matrix.

Keywords: Density; Mechanical Properties; Metallurgical Characteristics; SEM; Stir Casting; ZK60; ZrB₂.

1. INTRODUCTION

More than two ingredient materials with significantly different mechanical, chemical, and physical properties combine to form engineered materials known as composites. The distinct qualities of each of its component pieces, as well as their specific volume fractions and configurations within the material system, give rise to the distinctive qualities of these composites [1]. Composites may be created by combining more than one constituent substance with distinct chemical as well as physical features in order to accomplish a job that none of the individual elements can do on their own. Composite materials play a crucial role due to the limited use of pure materials in modern times. In order to imbue the final product with desirable attributes like superior strength, hardness, and resistance to deterioration, it is common practice to combine several materials [2].

The engineering sector has relied on material scientists and researchers for quite some time to provide products that possess appropriate properties, which have allowed manufacturers to boost output while cutting costs. A certain pattern has been followed in order to meet this demand: recognized techniques such as alloy additions, heat treatment, grain modification, and the like are utilized to try and improve the materials that are currently being used. In the event that these methods reach their limits, whether it is due to budgetary restrictions, difficulties associated with mass production, or the refusal to make any more improvements, a different strategy for improving quality, reducing prices, and increasing efficiency will emerge [3]. Superior metal-matrix composites (MMCs) provide a new category of framework materials when traditional alloys and metals approach their maximum development capacity. MMC with greatly improved properties (such as lower density, higher specific modulus, and higher specific yield strength) can be produced by processing a metal matrix with different particle or fiber reinforcements. This makes MMC ideal for a broad spectrum of prospective automotive and aerospace applications [4]. Inorganic nonmetal reinforcement of various sizes and morphologies is artificially incorporated into the metal matrix to create MMCs. MMCs can display the matrix's and reinforcement's respective performance advantages through sensible design. Additionally, they can acquire unique qualities that the "alloy" material lacks, such as low expansion, high thermal conductivity, particular strength, definite stiffness, and elevated temperature resistance [5].

Magnesium-based metal matrix composites (MMMCs) and alloys have several uses in the various industries. In high temperature applications where intermetallics are unstable, MMMCs outperform alloys [6, 7]. Because of their unique properties, magnesium (Mg) alloys have been extensively used as lightweight technology materials. Because of their high strength, ZK60 (Mg-Zn-Zr) alloys, which are commercial Mg-Zn-based alloys, have piqued researchers' curiosity [8]. Because of their remarkable mechanical capabilities and lightweight design, magnesium matrix composites have garnered a lot of interest [9]. Zirconium diboride (ZrB₂), constrained by numerous reinforcements examined to this day, continues to be a cutting-edge and exceptional element with a very high melting point, exceptional hardness, strength, exceptional heat conductivity, and thermal shock resistance, making it an ideal fit for the severe conditions associated with the aerospace industry [10]. ZrB₂'s mechanical characteristics include excellent electrical and thermal conductivity, hardness, and Young's modulus. ZrB₂ ceramics possess two characteristics: high heat shock resistance and superior corrosion resistance against molten iron and its alloys [11].

There are three primary kinds of casting methods that are used in the preparation of MMCs. These include liquid-state casting, solid-state casting, and semi-solid-state casting. Traditional castings are more challenging, less flexible, and not ideal for bulk production that is characterised by defects. As a result, the investigators initiated the use of the innovative liquid-state casting technology [12]. In order to create a homogenous mixture, stir casting involves continuously swirling the molten matrix metal and the reinforcement particles using a mechanical stirrer [13]. A number of benefits are associated with the stir-cast dispensing technique in comparison to other approaches. These positive aspects include simplicity of utilisation, flexibility, scalability with a range of materials, low cost, and high productivity, especially when it comes to the creation of enormous-scale composites [14]. They [15] observed the mechanical characteristics of ZK60 firmed with low-content nanodiamond, and enhancement in mechanical properties was achieved while rising ZK60 reinforcement. This work [16] explored the mechanical behaviour of eggshell-reinforced AZ31 composites and reported that the inclusion of eggshell particles eventually improves the mechanical characteristics. They [17] assessed the mechanical properties. This work [18] analyzed the tensile behaviour of AZ91/CNT/SiC composites and observed that a rise in reinforcement percentages enhances the tensile property.

The key goal of this research was to modify the tensile and compressive characteristics of ZK60A by incorporating Al2O3 nanoparticles. The synthesis of the ZK60A/Al2O3 composites was accomplished by employing the technique of disintegrated melt deposition, which ensued from hot extrusion [19]. Improving the ductility and tensile strength of the ZK60A magnesium alloy using Si3N4 nanoparticles was a key objective of this work. Examining the compressive characteristics was an additional goal of the current investigation. Composites showed substantial increases in both tensile and compressive properties as contrasted with monolithic materials [20]. Through this investigation, the link with the microstructure as well as the corrosion behaviour of ZK60 alloys will be verified, which ought to be of great assistance. In addition to this, it provides a theoretical foundation for the enhancement of the corrosion resistance of ZK60 alloys through the modification of the microstructure in the future [21]. This work investigated deteriorated areas, counter-face substances, and wear debris to gain a better understanding of the predominant wear mechanism under specific wear situations. In order to make a prediction about the wear capacity of the specimens (ZK60/CeO2), five different machine learning algorithms were compiled [22].

The addition of compounds Y and Ce enhanced the mechanical characteristics of the ZK60, with Y demonstrating a more significant strengthening impact. The study showed a 12.6% enhancement in the composite alloy's tensile strength [23]. The inclusion of Ce in the ZK60 increased the material's ductility from 15% to 17%. The enhanced mechanical capabilities of composite alloys may be attributed to the strengthening effects of grain refinement and ageing [24]. The purpose of this study [25] is to deliver the first comprehensive examination of the microstructure and textural properties of an HPT-processed ZK60 using EBSD techniques. Furthermore, in order to ascertain the relevance of work hardening as measured by the alloy's hardenability exponent and to probe the possibility of attaining hardness subsequent to processing under high stresses, this was also investigated. The authors utilized disintegrating melt deposition (DMD) to synthesize TiC nanocomposite in ZK60A and ZK60A/1.5 vol%, and then proceeded with hot extrusion techniques. Upon comparing ZK60A/1.5 vol.% with DMDed ZK60A, the TiC nanocomposite simultaneously demonstrated extremely significant gains in both tensile and compressive properties. When compared with DNDed ZK60A, the nanocomposite demonstrated a significant reduction of 16% in microhardness [26]. The density of the samples experienced a minor reduction when the concentration of MWCNTs increased to a maximum of one weight percent. MWCNTs have a lower density than nanotubes, which could explain why. The sample's microstructure examination revealed that increasing the B₂C level hampered the identification of macro-clusters of particles. By increasing the percentage of B_cC to twenty percent, it improved in both density and porosity to an extent [27]. Primary grains surrounded

by secondary precipitates characterize the microscopic makeup of the ZX51 alloy composites. Precipitates mostly appear at the borders between grains, with occasional globular ones also visible. Higher aluminum particles enhance each hardness and yield strength with regard to tension and compression. Conversely, the presence of higher particle concentrations leads to a decrease in ductility. In the as-cast state, the ZX51/Al₂O₃p demonstrates a fragile characteristic [28].

After creating the ZK60/SiC_p composite using the stir casting procedure, the authors followed the KoBo extrusion and precipitation hardening processes. This study examined the behavior of the unreinforced ZK60 alloy and the ZK60/SiC_p composite under various stress levels and after heating and cooling at 150 °C. The analysis of the creep data and the examination of the microstructures introduce the dominant creep mechanism and the damage mechanism, respectively [29]. During the manufacturing process, the researchers used a novel strategy by using a technology known as semi-solid temperature stirring. The authors conducted comparative research to compare the results of compression and wear tests on composites with those of unreinforced ZK60 alloy. The results of this inquiry reveal a significant improvement: the addition of 5% SiC led to a 15% increase in the compressive strength of ZK60 alloy, whereas the addition of 5% B₄C led to a 26% increase. The investigation's findings demonstrated a significant improvement [30]. Numerous investigations have shown that alloying and improving techniques are both primary and relatively effective ways to enhance the mechanical characteristics of ZK-based materials [31, 32]. Thus, our study's overarching goal is to measure the various characteristics of the produced composite specimens reinforced with a varied percentage of zirconium diboride ceramics and evaluate their different properties for future research in multiple applications.

2. EXPERIMENTAL DETAILS

ZK60 alloy, nominally composed of magnesium and zinc, was utilised as the base material in this assessment, and its composition is shown in Table 1 which is purchased from Krish Met Tech Pvt Ltd. Figure 1 shows the SEM photographs of the ZK60 matrix and Figure 2 shows the Energy Dispersive Spectroscopy [EDS] photographs of the purchased base ZK60 matrix.

The reinforcing material was ZrB_2 , which has a 10–15 µm particle size range of purity 99% purchased from Matrix Nano Pvt Ltd, Hyderabad. Figure 3 and Figure 4 shows the SEM and EDS photographs of the purchased ZrB₂ ceramic.

Table 1: Chemical constituents of ZK60 alloy.

С	0	Mg	Al	Zn
10.74	15.37	68.39	0.06	5.45



Figure 1: SEM Photograph of ZK60 alloy.



Figure 2: EDS photograph of ZK60 alloy.



Figure 3: SEM photograph of ZrB, ceramic.



Figure 4: EDS photograph of ZrB, ceramic.



Figure 5: Fabricated ZK60/ZrB₂ composites.

The initial steps involved are maintaining the furnace temperature at 820 °C, melting around one kg of ZK60 alloy at 730 °C, and cooling it down to 585 °C, which resulted in a semisolid matrix alloy. The pretreated ZrB₂ particles, at around 300 °C, were added to the semisolid ZK60 alloy melt via crucible. There were differences in the percentages of reinforcement weight at 0%, 4%, 8%, and 12%. To prepare composites, the semisolid stirring speed and duration were 225 to 250 r/min and 18 minutes, respectively. After sufficiently stirring the melt, we quickly warmed it to 700 °C for five minutes. After that, the melt was raised to 730 °C, the pouring temperature, and it was poured into a permanent steel mold that had been heated to 350 °C beforehand. It is repeated for the varying percentages of reinforcements, and the fabricated samples are shown in Figure 5. Figure 6 shows the prepared samples for testing.

An inverted trinocular metallurgical microscope with De-winter material plus version 2 software was used for the micro-structural characterization examinations of the composites [33] in accordance with ASTM E3 for sample preparation and ASTM F410 for measurement. The surface characteristics of composites were examined with a scanning electron microscope (Zeiss sigma machine) as per ASTM E986-2004 standards. We applied the Archimedes principle to measure the density. In accordance with ASTM E384-99, a micro vickers



Figure 6: ZK60/ZrB, composite testing samples.



Figure 7: Universal testing machine for composite testing.

hardness tester and HDNS-Kelly instruments of least count 0.01 mm with a range of 10 g to 1 kg were used to perform the test.

Using rectangular tension test specimens with a size of 10 mm width, 6mm thickness and a gauge length of 25 mm, the tensile tests were performed on an Fuel instruments and engineers make tensile testing machine of load range 5 tons at a nominal strain rate of 1 mm with mattes material solution software. It was done in accordance with ASTM-E8 guidelines. With a crosshead speed of 1 mm/min, a universal testing machine called the "UNITEK-1600-ST" was used to evaluate the compressive responses of the composite specimens which is shown in Figure 7. The ASTM standard E9-09 was followed in conducting the test. Impact testing was done at room temperature with a conventional Izod impact testing apparatus (XJJU-5.5), a hammer with an initial energy of 14 kg/m, and a striking velocity of 3.5 m/s. Impact testing were conducted in compliance with ASTM D256. Using a universal testing machine, a three-point bending test was performed to determine the composites' flexural strength under varied situations in accordance with ASTM D 790 standard.

3. RESULTS AND DISCUSSIONS

3.1. Microstructure examination

The microstructural characteristics of the composites have a considerable impact on their mechanical properties. The microstructure's development is mostly influenced by the rate of cooling during the phase transition. In increments of 4 weight percent, the dispersion being added spans from 4 to 12 weight percent. ZrB₂ is uniformly distributed throughout the matrix of the composites, as shown by the optical micrographs in Figure 8, and no voids or discontinuities are seen. The particles and matrix material have a strong interfacial connection. These optical micrographs show that all of the ZK60-ZrB₂ composites have a consistent dispersion distribution and strong matrix bonding. This is caused by the gravity of the ZrB₂ particles in combination with careful parameter selection for the stirring process and good matrix melt soaking of the preheated reinforcement. Grain structures that are coarse are produced by ZrB₂ particles. Figure 9 shows SEM analysis of cast ZK60/ZrB₂ specimens,



Figure 8: Optical micrographs of (a) ZK60, (b) ZK60-4 wt.% ZrB_2 , (c) ZK60-8 wt.% ZrB_2 and (d) ZK60-12 wt.% ZrB_2 composites.

correspondingly. Proper dimensions were extracted from the produced specimens. These fragments are meticulously crafted by polishing the exterior with a graded series of abrasive compounds to provide a smooth, scratch-free finish for investigation. SEM was used to examine the dispersion of augmented ZrB_2 reinforced particles in a ZK60 matrix with weight percentages of 0, 4, 8, and 12. The photos below display ZrB_2 under 1000× magnification, revealing homogeneous particle dispersion in the ZK60 base alloy, and the changes are noticeable.

3.2. Density

Figure 10 shows the effect of reinforcement on the densities of ZK60, ZK60-4 wt.% ZrB_2 , ZK60-8 wt.% ZrB_2 , and ZK60-12 wt.% ZrB_2 composites. It is evident from Figure 11 that the addition of ZrB_2 reinforcement improves the density of the composites gradually. ZK60 has attained the least density, and ZK60-12 wt.% ZrB_2 has attained a higher density. From the results, it is strongly observed that the rise in ZrB_2 reinforcement closes the voids and pores; this is the major reason for the increase in density. A 35% increase in density was attained because of the increase in reinforcements.

3.3. Micro vickers hardness

Tests were conducted on ZK60 and its composite to determine the hardness of ZrB_2 particles within the ZK60 matrix. Figure 11 displays the results of the micro-hardness test. The micro-hardness of a pure ZK60 alloy specimen was measured and found to be 78 VHV. The strengthening of zinc in the melt by solid solution when zinc is present as an alloying element is responsible for a notable improvement in the hardness property of the magnesium alloy. Upon adding ZrB_2 particles, an effect that increased monotonically was noted. It is evident from the figure that the hardness of the composites increases with an increase in ZrB_2 weight percentage [34]. Particle hardness of ZrB_2 leads to better grain size and resistance against localized deformation. A 30.37% increase in micro hardness was attained because of increase in reinforcements.

3.4. Tensile test

Figure 12 displayed the change in ultimate tensile strength as ZrB_2 changed. Stress transference from the ZK60 matrix to the reinforced ZrB_2 particles was the primary cause of the increase in tensile strength observed with increasing ZrB_2 content. This is due to the Orowan mechanism, in which a dislocation bows out significantly to leave a dislocation loop around a particle, allowing it to avoid impenetrable obstructions. Tensile strength is



(a)



(b)



Figure 9: SEM image of (a) ZK60-4 wt.% ZrB₂, (b) ZK60-8 wt.% ZrB₂ and (c) ZK60-12 wt.% ZrB₂ composites.



Figure 10: Density vs reinforcement weight percentage.

enhanced as a result of the dislocations' interaction with ZrB_2 . A 37.5% increase in Tensile strength was attained because of increase in reinforcements.

3.5. Impact test

Figure 13 shows the impact strength of ZK60 alloy and the ZK60 composites with various wt% of ZrB₂. The ZK60 alloy shows a lower impact strength of 3.2 Joules on comparison with ZK60-4 wt.% ZrB_2 , ZK60-8 wt.% ZrB_2 and ZK60-12 wt.% ZrB_2 . The impact strength of ZK60-12 wt.% ZrB_2 has attained 7.7 Joules was greater than that of the ZK60 alloy. ZK60-ZrB₂ composites can be made more impact resistant by implementing a number of techniques that improve the material's capacity to absorb energy and withstand fracture under



Figure 11: Micro vickers hardness vs reinforcement weight percentage.



Figure 12: Tensile strength vs reinforcement weight percentage.



Figure 13: Impact strength vs reinforcement weight percentage.

rapid loads. The impact strength of $ZK60-ZrB_2$ can be greatly increased by adding ZrB_2 reinforcing elements. Reinforcements made of ZrB_2 can increase the material's resilience to crack propagation and toughness. To maximize impact strength, it is essential to regulate the size and distribution of reinforcing particles inside the magnesium matrix. Uniformly dispersed ZrB_2 particles can effectively impede the propagation of cracks and enhance absorption of energy during impact events. A 140% increase in impact strength was attained because of increase in reinforcements.

3.6. Compression test

Figure 14 illustrates the compression strength of as casted ZK60 and the ZK60 composites with distinctive wt% of ZrB₂. The ZK60 alloy shows a lower compressive strength of 240 MPa on comparison with ZK60-4 wt.%



Figure 14: Compression strength vs reinforcement weight percentage.



Figure 15: Flexural strength vs reinforcement weight percentage.

 ZrB_2 , ZK60-8 wt.% ZrB_2 and ZK60-12 wt.% ZrB_2 . The compressive strength of ZK60-12 wt.% ZrB_2 has attained 340 MPa was greater than that of the ZK60 alloy, which happens due to the excellent inter atomic bonding between the ZK60 alloy matrix and the reinforcement.

The tested ZK60 alloy and the composites show compressive strength higher than ZrB_2 . In general, the compressive strength of composite can be improved by considering factors such as grain size and dislocations. A 41.66% increase in compression strength was attained because of increase in reinforcements.

3.7. Flexural test

Tests were conducted on three-point bend trials to determine the flexible characteristics of the $ZK60-ZrB_2$ composites as the ZrB_2 content increased. Figure 15 demonstrates that the inclusion of ZrB_2 resulted in an increase in flexural strength. Simultaneously, including ZrB_2 in the ZK60 matrix improves the flexural characteristics of the composites.

Across all degrees of reinforcement, the 12% ceramic particle-reinforced composite exhibited better properties in comparison to other levels of reinforcement. When the particle size is small, the interparticle distance decreases. The presence of ultrafine particles hinders the ability of plastic deformation. This finally results in an escalation of rupture. A 56.97% increase in flexural strength was attained because of increase in reinforcements.

4. CONCLUSIONS

- Stir casting was a successful method of producing ZK60-ZrB, composite.
- The study using an optical microscope and SEM verifies that ZrB₂ reinforcement with ZK60 matrix is present and uniformly distributed.
- ZrB₂ addition has an effect on composite density, micro-hardness, and this effect increases as ZrB₂ particle concentration does.



• The tensile, impact, compression and flexural strengths of the composite are increased by the addition of ZrB, particles, and they are higher than those of the basic alloy.

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