

Synthesis, Characterization and Mechanical Behaviour of Mg-Al Based Insitu Composites for Biomedical Applications

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Abstract: Magnesium based composites have emerged as potential material in biomedical applications owing to its biocompatibility and strength. Present work fabricates the Magnesium-Aluminium with insitu reinforced zirconium diboride composites (Mg-Al/ZrB₂). Synthesis was conducted in the presence of Argon gas through electric resistance furnace using stir casting route. XRD analysis was performed to see the presence of elements which confirmed the presence of Mg, Al and ZrB₂ peaks. Further Optical microscopy study was conducted to analysis the behaviour of microstructural features of alloy and composites. It was observed that inclusions of insitu formed ZrB₂ results in significant reduction in average grain size. Further, SEM was employed to see the presence of particle, particles distribution, their shape and size. SEM reveals uniform distribution of ZrB₂ particles with hexagonal morphology. However, some agglomeration is also evident which is due to nano particles size of reinforced particles. Also, some porosity is also visible in composite which is further confirmed through density and porosity investigation. Further the Vickers hardness test and ultimate tensile test was performed to analysis the mechanical behaviour of alloy and composites. Results clearly demonstrate increase in Vickers hardness value by ~ 67% and tensile strength ~ 30 % of composite when compared with Mg-Al alloy.

Keywords: Characterization; Fabrication; Insitu composite; mechanical properties; Mg-Al alloy

1. Introduction

Magnesium (Mg) and its alloys have garnered significant interest in the recent past owing to their excellent properties for a variety of applications, particularly in the biomedical field. Magnesium's low density (1.74 g/cm³), high specific strength, and natural biodegradability make it an attractive material for applications such as orthopedic implants¹. More importantly, Mg's biocompatibility and ability to degrade in biological environments eliminate the requirement for a second surgery to remove the implant, reducing patient risk and medical costs². However, pure Magnesium suffers from several drawbacks, including low mechanical strength and rapid corrosion rates, which restricts its use in load-bearing applications, magnesium alloys have been developed, with the incorporation of alloying elements such as aluminium (Al) to enhance mechanical strength, corrosion resistance, and overall performance³. Mg-Al alloys, such as AZ series alloys (e.g., AZ31, AZ91), are among the most widely researched magnesium alloys. Aluminium serves to increase the hardness and tensile strength of magnesium alloys while

reducing grain size through precipitation hardening. Despite these improvements, Mg-Al alloys still exhibit insufficient mechanical strength and corrosion resistance for long-term biomedical applications, particularly in harsh physiological environments⁴.

As a result, there has been a growing focus on developing Mg-Al-based composites that incorporate ceramic reinforcements to further enhance their mechanical properties and corrosion behavior⁵. Composites offer the advantage of combining the desirable properties of a metal matrix with the high hardness and strength of ceramic reinforcements^{6,7}. Among various ceramic reinforcements, zirconium diboride (ZrB₂) has emerged as a promising candidate due to its excellent hardness, high melting point, and chemical stability^{8,9}. ZrB₂'s high biocompatibility also aligns with the stringent requirements for biomedical applications, making it an ideal reinforcement for Mg-Al-based composites¹⁰.

The synthesis of Mg-Al based composites has become essential for improving the mechanical characteristics and corrosion resistance of magnesium alloys for biomedical applications¹¹. The use of ZrB₂ as an in-situ reinforcement

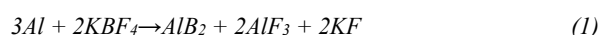
creates a strong interfacial bonding between the matrix and the reinforcement particles, which increases the load transfer and results in better performance under tensile loading¹⁰. Studies have demonstrated that in-situ formed reinforcements have superior bonding and mechanical properties compared to ex-situ reinforcements, where ceramic particles are mechanically mixed with the metal matrix^{12,13}.

Among the various fabrication methods, the stir casting route has been chosen for the synthesis of Mg-Al/ZrB₂ composites due to its simplicity, cost-effectiveness, and ability to produce uniform dispersions of reinforcement particles^{14,15}. Stir casting involves an introduction of a reactive element or compound into the molten magnesium alloy to form the reinforcement in-situ, avoiding the issues of poor interfacial bonding and agglomeration that often occur with ex-situ techniques¹⁶. Furthermore, the use of an argon gas atmosphere during the casting process helps to minimize oxidation, ensuring the quality and purity of the final composite material. Stir casting is a scalable technique, making it suitable for industrial production, which is critical for developing biomaterials for commercial biomedical applications^{17,18}. In this context, the current research aims to synthesize in-situ composites using stir casting and to characterize the microstructure and mechanical behavior of the composites. The study focuses on investigating the influence of in-situ formed ZrB₂ particles on the grain structure, hardness, and tensile strength of Mg-Al alloys, with the goal of developing a composite material that meets the mechanical and biocompatibility requirements for biomedical implants. By enhancing the mechanical properties of Mg-Al alloys, the study seeks to overcome the limitation of conventional magnesium alloys and contribute to the development of next-generation biodegradable implants for enhanced load-bearing applications.

2. Materials and Methodology

To fabricate the alloy and composites, commercially available pure Mg (purity $\geq 99.89\%$), Al (purity $\geq 99.9\%$), and inorganic salts of Potassium Fluorozirconate (K₂ZrF₆) and Potassium tetrafluoroborate (KBF₄) were procured from India mart. To fabricate the alloy, Mg and Al were taken in the ratio of 95 and 5 weight percent respectively. Initially, the furnace temperature was set at 680°C, and the required amount of Mg and Al in the form of small ingots were kept in graphite crucible having capacity of 5 liter. The heating rate was set 4°C/minute, and holding time was set for 45 minutes. The continuous supply of Argon gas was maintained to create inert atmosphere. After holding for 45 minutes at 680°C the molten alloy was poured into preheated (at 200°C for 2 hour) cast iron Mold having size of 50mm × 50 mm × 170 mm. For the fabrication of composites, initially the furnace temperature was set at 780°C, and required amount of Al

ingots was kept in graphite crucible was placed in furnace. Once the temperature reached at 780°C, the preheated (at 120°C for 1 hour to remove the moisture presents) inorganic salts of K₂ZrF₆ and KBF₄ wrapped in Al foil was charged into furnace. To form insitu ZrB₂, intermittent stirring (10 minutes stirring followed by 3 minutes holding) using mechanical stirrer having stainless steel blade was performed for 39 minutes at 200 rotations per minute (rpm). Once the reaction was completed the temperature was lowered at 680°C, and small ingots of preheated (at 100°C for 1 hour) Mg was incorporated into melts and was hold till the Mg was in molten condition. After the melting of Mg, once again the 10 minutes stirring was performed at 200 rpm for homogenization. Finally, the casted samples were poured in preheated mould and allowed to cool at atmospheric temperature. The three composites were fabricated having ZrB₂ weight percentage (wt. %) 1, 3, and 5 respectively. During the whole fabrication process of composites, the continuous supply of Ar gas was maintained. The following likely reaction may have taken place to form the insitu ZrB₂ particles in the melts. In the reactions, Aluminum reacts with the boron obtained from the decomposition of KBF₄ to form AlB₂ which is an intermediate boride. In the presence of Al, K₂ZrF₆ decomposes and forms Al₃Zr. Now both AlB₂ and Al₃Zr are intermediate forms that combines together to form ZrB₂ (an in situ formation) within the Al matrix and releasing excess Al back to the melt. The reaction takes place on the basis of negative Gibbs free energy concept. The decomposition of KBF₄ and K₂ZrF₆ is occurred due to the presence of Al, which is a strong reducing agent. At last, thermodynamically stable compounds are formed in which total enthalpy decreases and net entropy increases¹⁹.



3. Characterization and Testing

Desktop XRD of Malvern Pananalytical technique was employed to find the peaks present in the alloy and composites. XRD study was performed for 2θ values of 10°-90° with scan rate of 10° per minute. Because, at an angle of less than 10°, it has been observed from the literature that the peaks obtained are weak and has a probability of overlapping with noise or amorphous phase. Whereas, at an angle greater than 90°, the noise increases that leads to significant drop in the intensity. Optilux optical microscopy of Lietz was used to analyze the optical microstructure of alloy and composites. The square cross section of sample (20 mm × 20 mm × 10 mm) was prepared from as cast sample. Set of emery papers was used for grinding the surface followed by cloth polishing of



Fig.1: (a) stir casting setup (b) Mold of cast iron (c) as cast sample (d) prepared sample for optical microscopy and Vickers hardness study (e) Tensile sample

specimen. Once the sample was free from scratch, the acetic Picralwas used as etchant to find out the grain boundaries. Further, JEOL Bench top scanning electron microscopy (SEM) was utilized to see the particles size, shape and distribution of particles in the matrix. Experimental density was determined using Archimedes principal and porosity was calculated using ratio of difference of theoretical density to experimental density over theoretical density.

To study the impact of reinforced particles on mechanical properties, the hardness and tensile test was performed using Vickers hardness and ultimate tensile strength set up according to ASTM standard E384 and E8 respectively. Figure 1 shows the stir casting set up; Mold made of cast iron along with prepared samples for various study.

4. Results and Discussion

Figure 2 presents the X ray diffractometer (XRD) image of Mg-Al alloy and Mg-Al/5wt.% ZrB₂ composites. It can see from the image that in the case of alloy, the peaks of Mg and alloy of Mg i.e. Mg₁₇Al₁₂ is present while for composites along with peaks of Mg and MG₁₇Al₁₂, presence of ZrB₂ can be seen, which confirms the presence of reinforced particles in matrix.

Figure 3 shows the optical microstructure (OM) of Mg-Al alloy and Mg-Al/5 wt.% ZrB₂ composite. Figure 3 (a) shows the OM of Mg-Al alloy. The white region shows the α - Mg (magnesium-rich phase), while the darker region presents the Mg₁₇Al₁₂ phase. Figure 3 (b) shows the OM of Mg-Al/5 wt.% ZrB₂. A finer grain structure can be seen in the case of composites. This reduction in grain size is due to the insertion of reinforced particles, which act as a secondary phase leading to the region of generation of

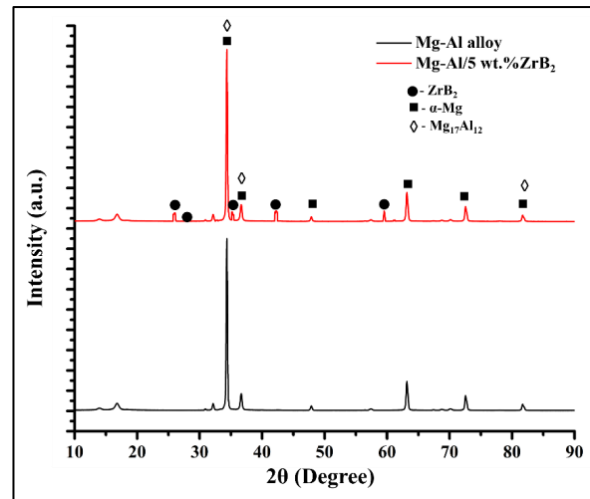


Fig.2: XRD of Mg-Al alloy and Mg-Al/5wt.% ZrB₂ composite

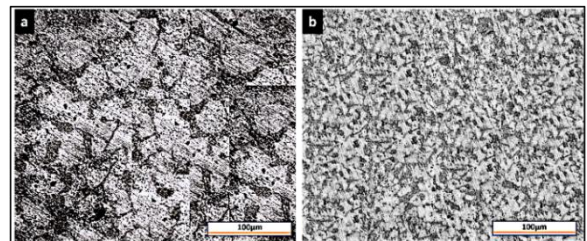


Fig.3: Optical microstructure of (a) Mg-Al alloy (b) Mg-Al/5 wt.% ZrB₂ composite

nucleation sites, which further hinders grain growth. Further, mismatch in the thermal coefficient of hard ZrB₂ particles with Mg-Al causes a restriction in grain growth, thus finer grain structure¹⁹⁻²¹. The average grain size was determined in accordance with ASTM E112 standard. At least five random fields were analyzed per sample to ensure statistical accuracy and given in Figure 5²²). In the linear intercept method, a set of straight, evenly spaced lines is superimposed over a micro structural image. These lines intersect grain boundaries, and each intersection point is counted. The basic idea is that the more the intersections the smaller the grain size. The average grain size is then calculated by dividing the total length of all test lines by the number of intercepts. This gives an estimate of the mean linear intercept length, which represents the average grain diameter. This can be observed that for Mg-Al alloy the average grain size is in the range of 27 μ m while decreases with addition of ZrB₂ and is observed to be lowest with reinforcement of 5 wt.% of ZrB₂ having order of 8 μ m.

Figure 4 presents the SEM image of Mg-Al/5wt.% ZrB₂ composites. This can be observed from the Figure 4(a) that particles are uniformly distributed with slight agglomerations which may be because of very fine size of the particles. Further Figure 4(b) shows the SEM image of particles at higher magnification which clearly demonstrate the shape and size of reinforced particles i.e. hexagonal shape²³⁻²⁵). Also, the average particles size was

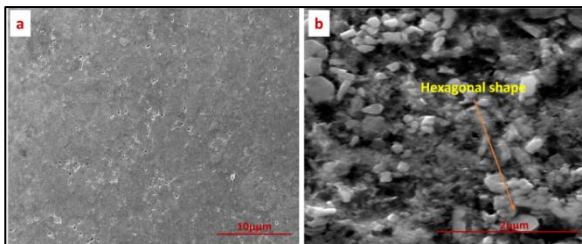


Fig.4: Scanning electron microscopy of (a) Mg-Al alloy (b) Mg-Al/5 wt.% ZrB₂ composite.

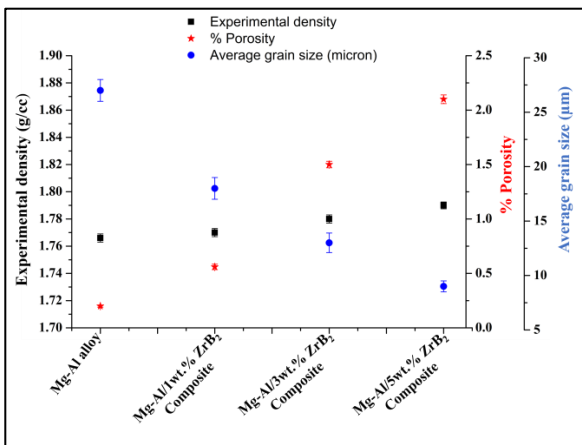


Fig.5: Density (g/cm³), % Porosity, and average grain size (μm) of alloy and composites

determined using Image J software from SEM micrograph and found to be 0.1 μm.

Figure 5 shows the physical properties (i.e. experimental densities and % porosity) and average grain size of the alloy and composites (Figure 4). It can be observed from Figure 4. That the mixing of ZrB₂ and Al is fairly homogenous however, at some sites the ZrB₂ agglomeration is noted which is probably attributed to the uneven cooling and shrinkage of boundaries due to solidification. The observed decrease in experimental density, despite the use of high-density reinforcement particles can be attributed to the presence of micro-porosity and possible particle agglomeration during solidification²⁶. These defects reduce the overall compactness of the matrix, leading to a lower measured density compared to the theoretical value²⁷. This observed porosity may be attributed to more stirring speed and trapping of atmospheric gases at the time of pouring²⁸). In addition to, phenomena such as grain boundary pinning (due to local grain coarsening), etching, and settlement of ZrB₂ particle due to not rapidly cooling are some of the probable reasons of density difference.

Figure 6 presents the mechanical (hardness and tensile strength) of Mg-Al alloy and its composites. This can be seen that with addition of ZrB₂, there is significant improvement in hardness with respect to Mg-Al alloy and maximum for Mg-Al/ 5 wt. % ZrB₂ composites. This is due to high hardness properties of reinforced particles which results in an increase in bulk hardness. Further, reinforced

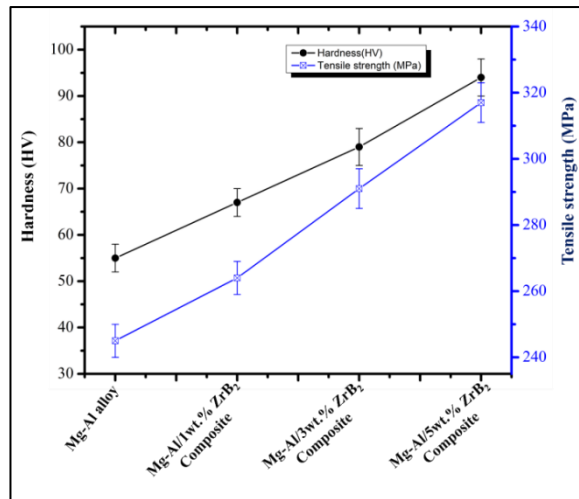


Fig.6: Hardness (HV), and tensile strength (MPa) of alloy and composites

ZrB₂ particles and Mg-Al alloy exhibits mismatch in thermal coefficient of expansion which results in increase in dislocation density thus causing restriction to plastic deformation further leading to rise in hardness^{10,29,30}.

Tensile behaviour of Mg-Al alloy and its composites was investigated at atmospheric temperature as given in Figure 6. This can be observed that with reinforcement of ZrB₂ particles, there is a significant rise in tensile strength of matrix of Mg-Al alloy and is maximum for Mg-Al/5 wt.% ZrB₂. The increase in tensile strength results from the refinement of matrix grains caused by the introduction of ZrB₂ particles, as explained in the previous section. This grain refinement leads to improved tensile strength, which can be explained by the Hall-Petch relationship in which the particles of ZrB₂ serve as heterogeneous nucleation sites that result in refined grain structure in the Al metal matrix. During tensile loading, dislocations are generated, and the second-phase particles obstruct their movement due to decrease in grain size and increase in total grain boundaries area, thereby contributing to the strengthening effect³¹⁻³⁴. Additionally, the mismatch in the thermal expansion coefficients between the matrix and particles further raises the dislocation density, enhancing the strength³⁵.

5. Conclusions

The Mg-Al alloy and Mg-Al/ZrB₂ in situ composites were successfully synthesized using the stir casting technique, with ZrB₂ reinforcement varying from 0 to 5 wt.%. Key observations include:

The in-situ formation of ZrB₂ particles significantly refined the grain structure, achieving up to ~70% grain refinement with 5 wt.% ZrB₂ addition.

Homogeneous dispersion of ZrB₂ particles in the matrix was achieved, contributing to enhanced mechanical properties.

Vickers hardness increased notably from 55 HV to 92 HV at 5 wt.% ZrB₂.

Tensile strength improved by ~30% for the Mg-Al/5 wt.% ZrB₂ composite compared to the base Mg-Al alloy.

With superior hardness and tensile strength, the newly developed Mg-Al/5 wt.% ZrB₂ composite holds promising potential for biomedical applications, particularly in load-bearing implants and devices where strength and biocompatibility are crucial.

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