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Fracture Analysis and Morphological Characterization of the Mg - 3 Zn -1Ti /TCP Layered Composite using Centrifugal Casting Process

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Abstract

Objectives: To analyze the mechanical properties and microstructural attributes of functionally graded metal matrix composites (FGM- matrix) made from magnesium alloy as the base material (Mg). Magnesium, Titanium, Zinc compositions are widely used in the creation of bio-materials for implants because of their beneficial corrosion properties. **Methods:** Functionally graded materials (FGM) have been effectively utilized in the development and production of engineering components that possess location-specific properties. In this research, the processing and characterization of Magnesium metal as the base material, Zinc and Titanium as alloying elements, and Tricalcium phosphate (TCP) as a reinforcement with composites of Mg - 3 Zn -1Ti are discussed. The composite melt preparation was carried out using the stir casting method, by the formation of FGM-metal composites through the vertical centrifugal casting method. FGM-metal composites exhibit a centrifugal graded distribution of reinforcements, resulting in distinct zones including a rich-matrix inner zone, transition zone, and rich-particles outer zone. **Findings:** The mechanical properties are significantly improved in the rich-particles outer zone, showcasing enhanced hardness, and ultimate tensile strength (UTS) of 351 MPa by following corresponding to the particle concentration, with a peak value of 106 Hv achieved in the particle-rich region. This highlights the importance of microstructural design in achieving superior mechanical characteristics in FGM-metal composites. **Novelty:** The study highlighted a lack of research on these alloys compared to biomaterials like mesh or lattice structured Stainless steel, which have shorter lifespans in the human body due to rapid degradation and inherent defects such as porosity.

Keywords: Functionally graded material metal matrix composites (FGM-matrix composites); Centrifugal casting; Tensile Characteristics; Micro hardness; Microstructural behavior; Bio-implants

1 Introduction

Continuous expansion of new industries in material and scientific-technological advancements has led to an increasing demand for extra sophisticated and smart materials with precise properties. The emergence of Functionally Graded Materials (FGM) can be attributed to recent advancements in material processing methods, representing a complex type of multilayered materials with a gradual transition of characteristics between two phases within the sample[(1)].

Kumar MA et al. highlighted the role of biomaterials and enabling exploration on repairing or replacement of damaged bones and joints. Major considerations for positive biomaterials encompass elemental stability, tissue-compatibility, and mechanical properties. Remarkably, FGM has garnered attention from the automobile and bio/green medical industries for part sample manufacturing[(2)].

Magnesium is increasingly acknowledged for its potential in bio-medical implants owing to its bio-green and tissue-compatibility, but its rapid degradation can cause corrosion and implant failure, necessitating the development of new FGM-composites with greater mechanical properties and corrosive challenge. This research delves into different material combinations, including magnesium-based alloys with zinc, titanium, and Tricalcium phosphate (TCP) to enhance the overall performance of biomedical implants. The focus of the study is on Mg - 3 Zn -1Ti composites with Tricalcium phosphate as a reinforcement to tackle the issues related to magnesium degradation in physiological conditions. The amalgamation of these metallic elements has the potential to improve biomedical devices and promote contact between metal surfaces, providing advantages such as improved stress shielding in nearby bone and greater load-bearing capacity with smaller designs. On the other hand, manufactured materials might exhibit some imperfections and disadvantages that could be rectified through various trials, as well as unfavorable characteristics such as low resistance to deformation over time, limited ability to be shaped at low temperatures, and insufficient protection against corrosion.[(3)]

The investigation of different layers of FGM particles, including inner, middle, and outer layers, is essential to know the mechanical and morphological properties of FGM-based composites. By examining microstructural features and Vickers micro hardness, researchers can obtain valuable insights into the performance of these alloys. The study results show that FGM-based alloys demonstrate promising characteristics such as corrosion resistance, mechanical strength, and biocompatibility, indicating their potential for use in applications requiring these properties for optimal performance. Ongoing research aims to understand the mechanical behavior of magnesium-based alloys at a deeper level.

Researchers are striving to enhance the performance of alloys in various environments and applications by delving into the mechanisms and developing effective surface treatments. The study highlighted on poor research on these alloys associated to biomaterials like mesh or lattice structured Stainless steel, which have shorter lifespans in the human body due to rapid degradation and inherent defects such as porosity. The FGM samples underwent mechanical and microstructural characterization using various experimental methods.[(4)]

2 Methodology

2.1 Material

The magnesium powder was selected as the matrix material in this research due to its abundance and low density, while Zinc, Titanium, and Tricalcium phosphate (TCP) were added as alloying elements and reinforcement, respectively. The metal matrix AZ31, with a density of 1740 kg/m3 (108.6 lb/ft3), a melting point of 650°C, and a hardness of 55.0-05 Hv, was utilized in the formation of composites[(5)]-[(6)].

The alloying element and reinforcement material are added to the melting Mg. Then, the solidified liquid material is transferred to the centrifugally manufactured molding system to attain the desired graded composite. This technique is aided by a software tool for stir casting, and the resulting component are shown in Figure 1(a-c) shows the outer layer. Subsequently, the chamber is triggered, and the temperature is incessantly supervised with the help of a thermocouple junction.[(7)]. When the magnesium substance reaches a heat of 740°, it is further transferred to furnace chamber, and then the container's cap is closed. And to ensure a uniform composition of the zinc and titanium alloying powder with the reinforcement material TCP, preheating them to 450 °C was essential. The excess moisture content was effectively eliminated through the preheating process. Following that, the equipped melt was subjected to stir, which operated at a 450 rpm range of speed. The reinforced material was progressively fused into the melt whirlpool while the stirring.

To ensure an even distribution of reinforcements in the molten magnesium, the liquid in its molten state is discharged into a heated spiral mold at a heat of 450 °C. To prevent abrupt solidification, then the spiral mold is set to a speed of 1300 RPM. Once all preparations are complete, the chamber valve is unlocked, allowing the fused liquid to be drained into the revolving die. The centrifugal force propels the molten melt towards the die wall in a radial direction, initiating the solidification process.

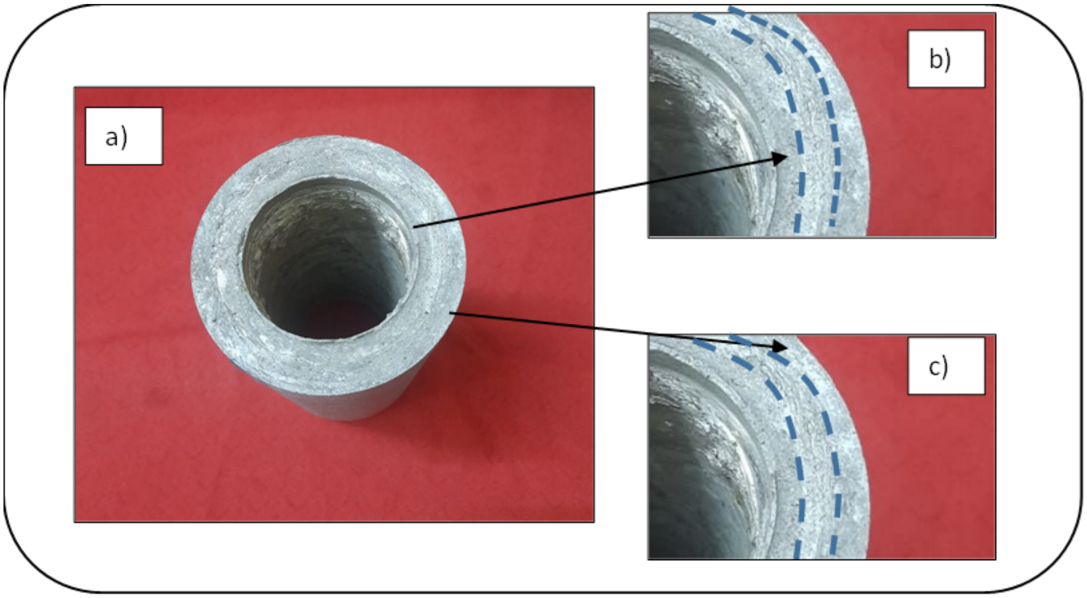

Fig 1. a) FGM fabricated component, b) Inner layer, c) outer layer

Table 1. FGM fabrication parameters: (Anil Kumar et al) [2]

Gravity casting Parameters	Output values
Stirrer RPM	450 and 750 RPM with vertical movement for 30mm
Furnace Temperature	700^0 C
Melt Temperature	740^0 C
Die Temperature	450^0 C
Centrifugal RPM	1300 RPM

Necessary time is given for the centrifugal part to cool before it is detached from the die, cleaned, and prepared for additional testing by cutting samples. The experimental parameters used in this research are detailed in Table 1, and the same procedure was followed for every sample. [8]

2.2 Microstructure

Observations of the microstructures of the fabricated FGMs were conducted through the use of a scanning electron microscope (SEM). Microstructural examinations were carried out on the samples following mechanical and chemical polishing procedures. Thin strips measuring $15 \times 10 \times 10$ mm were obtained from the hollow cylinder by cutting them longitudinally by the axis, with 10 mm width. The strip was then further cut into a 15 mm length, leaving a distance of 10 mm from the top surface in the transverse direction. The strip was positioned in such a manner that facilitated the examination of how particles were distributed radially. The outer region, which contained a high concentration of particles, was in contact with the mold wall, while the inner region was closer to the principal axis of the casted cylinder. [9]

Following the completion of the polishing process on the mount using emery paper and velvet cloth, the microstructures were analyzed. The mount was then rinsed with running water before capturing microstructural images at the outer, middle, and inner zones with specific distances from the periphery. The ImageJ software was utilized to determine grain size and phase volume in all areas for each sample.

2.3 Mechanical behavior

Micro hardness Tester was utilized to determine the hardness of a casted sample by applying a load of 1kg for 15 seconds. The sample was then longitudinally cut and transformed into slim strips, further cut to 5mm length in the crosswise direction, and 2mm away from the top region. After undergoing the processes of polish enhancement to achieve a smooth surface, the hardness of the material was assessed in various regions. To ensure accuracy, six readings were found at nine dissimilar longitudinal locations, the resulting average readings were documented [(10)]. A Sensotech technologies test system with a 25 kN force capacity was employed for the Tensile test at room temperature, with fixture movement facilitated by a hydraulic wedge grip. The compression test utilized an Mtest-647 hydraulic material test system capable of exerting a maximum force of 100 kN. Each sample three average tensile samples were cast according to ASTM standards and examined using UTS equipment. Fractured surface macrographs and microstructure images were captured using a ZEISS stereo microscope to examine surface morphology, whereas JEOL-SEM images were utilized for the analysis of mechanical and microstructural properties.

3 Results and Discussions

3.1 Microstructural behavior

The schematic diagram presented in Figure 2(a) illustrates a centrifugally cast functionally graded material (FGM) matrix composites disk, highlighting various areas and positions where test specimens are extracted for a range of analyses including microstructure, hardness, tensile strength, compression properties, and wear resistance assessments. In contrast, Figures 2(b) and (c) showcase the optical micrographs of the inner zones of the composite material, revealing areas with poor particle distribution and others with a more moderated distribution. Specifically, the micrographs display regions with smaller grain size in Mg-3Zn-1Ti + TCP gravity castings, with the composite material exhibiting a uniform distribution of 3 Zn-1Ti + TCP particles in the Mg alloy matrix. Furthermore, Figure 2(d) a provide optical micrographs of the centrifugally cast Mg alloy, highlighting the presence of shady needle-like eutectic phases, gray-colored phases, and white 3 Zn-1Ti + TCP matrix phases. Additionally, the micrographs reveal consistent spaced secondary dendritic arms within the microstructure, contributing to a comprehensive understanding of the material's composition and structure.

There are four distinct microstructure regions that can be observed in both cases. These regions include the matrix-inner zone, which is the inner region that does not contain any reinforcement particles. The middle (moderate particle zone) is another region that can be identified, and it is characterized by a moderate concentration of reinforcement particles. Finally, the rich-particle matrix is the outermost periphery of the disc and is characterized by a high concentration of reinforcement particles. The solidification process of a cast disk measuring 300 mm in diameter and 28 mm in thickness usually takes approximately 2 to 3 minutes to complete. In centrifugal casting, the solidification front progresses from the mold's outermost boundary near the inner diameter of the casting in a centrifugal innermost path. On the other hand, the deeper reinforcement particles and secondary phases move from the inner to the outer diameters in a radial outward direction due to the centrifugal force applied during the casting process.[(11)]

The position, dimensions, and highest particle density of the particle-laden area are impacted by the solidification duration and the centrifugal force (gravity effect). Although there is an outer region with a radial extent of 2 to 3 mm, where the solidification obverse and particle diffusion fronts travel in opposite directions, the intersection of these fronts beyond this radial boundary is vital in establishing the exact position and strength of the peak particle concentration. Jayakumar et.al observed that the presence and distribution of alpha-Mg phases differed between the top/outer surface (reinforcement zone) and the inner region surface. They found that the alpha-Mg phases were more prevalent and tightly packed in the top/outer surface, indicating a higher concentration of these phases in this region. This concentration of alpha-Mg phases in the reinforcement zone contributed to the enhanced mechanical properties observed in this area. On the other hand, the inner region surface exhibited a lower density of alpha-Mg phases, suggesting a relatively lower concentration of these phases. Consequently, the mechanical properties of the inner region surface were not as improved as those of the top/outer surface.[(12)]

Ansari et al. (2021)[(13)] examined the FGM of Ti-6Al-4 V and pure Molybdenum. Also, Ansari performed Mechanical testing's and bio-compatibility tests using 3 Zn -1Ti composites. Figure 3 shows EBSD maps of four interfaces, and the primary transition of material reinforcement, β grains were restructured from α grain alignment. EBSD transition maps shows decent steadiness of β grains at the interface. On other phase, 100% weight percentage of 3 Zn -1Ti + TCP, there is a solid surface of coarse columnar β grains mounting similar to the centrifugal build track. Without adding reinforcement, columnar β grains were lesser by specific orientation. The increasing reinforcement (3 Zn -1Ti + TCP) material, the morphological structure has transformed from columnar to equiaxed β grains. Furthermore, the existence of non-fused melt reinforcement material is obvious. The slope from Mg-3 Zn -1Ti + TCP is another example of FGM composite. The FGM composite material has dissimilar crystal structures, micro-hardness, physical properties, and melting temperature.

Figure 4 displays the XRD patterns of FGM sample manufactured under centrifugal Gravity. Noted the dependence position on the XRD patterns that is 3 Zn -1Ti + TCP FGM composite. It is identified and evident that the peaks at outer area, and the weak peaks presented at the middle and inner areas.

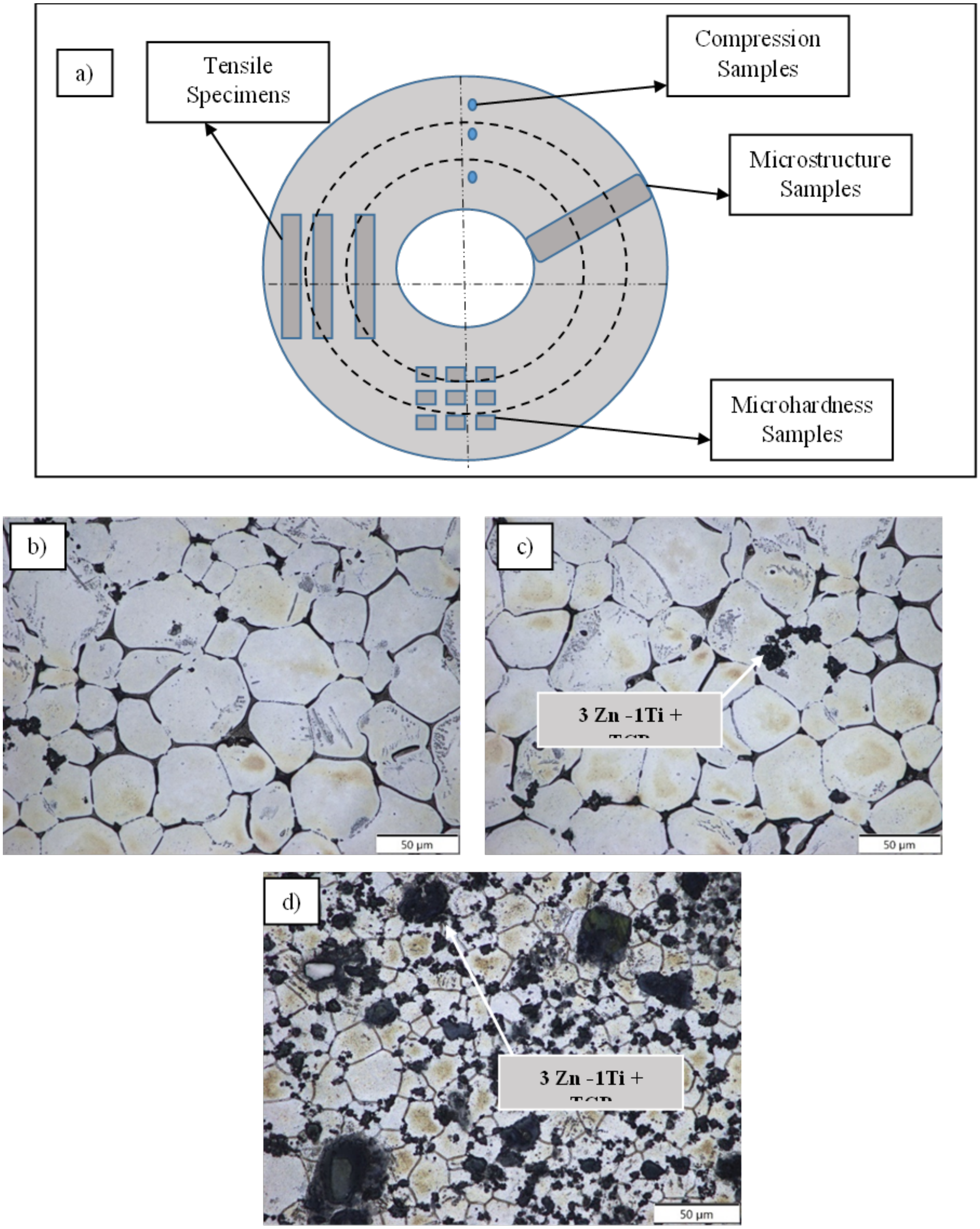

Fig 2. Microstructures of Mg - 3 Zn -1Ti (a) the schematic illustration depicts the casting layout, highlighting specific zones and indicating the positions of samples designated for further analysis and characterization. (b) Mg base inner zone and (c) Mg based middle zone (d) Mg based outer zone

3.2 Micro-Hardness

As a result of this force, denser particles are projected radially outward, leading to their segregation towards the periphery. Consequently, the outer region of the samples displays a greater concentration of 3Zn-1Ti+10TCP elemental segregation, while the inner region exhibits a lower concentration. Additionally, it is worth noting that the elemental segregation gives rise to the formation of laves phase pattern, which is non-beneficial to the ductility of the specimens and contributes to an increase in their hardness.

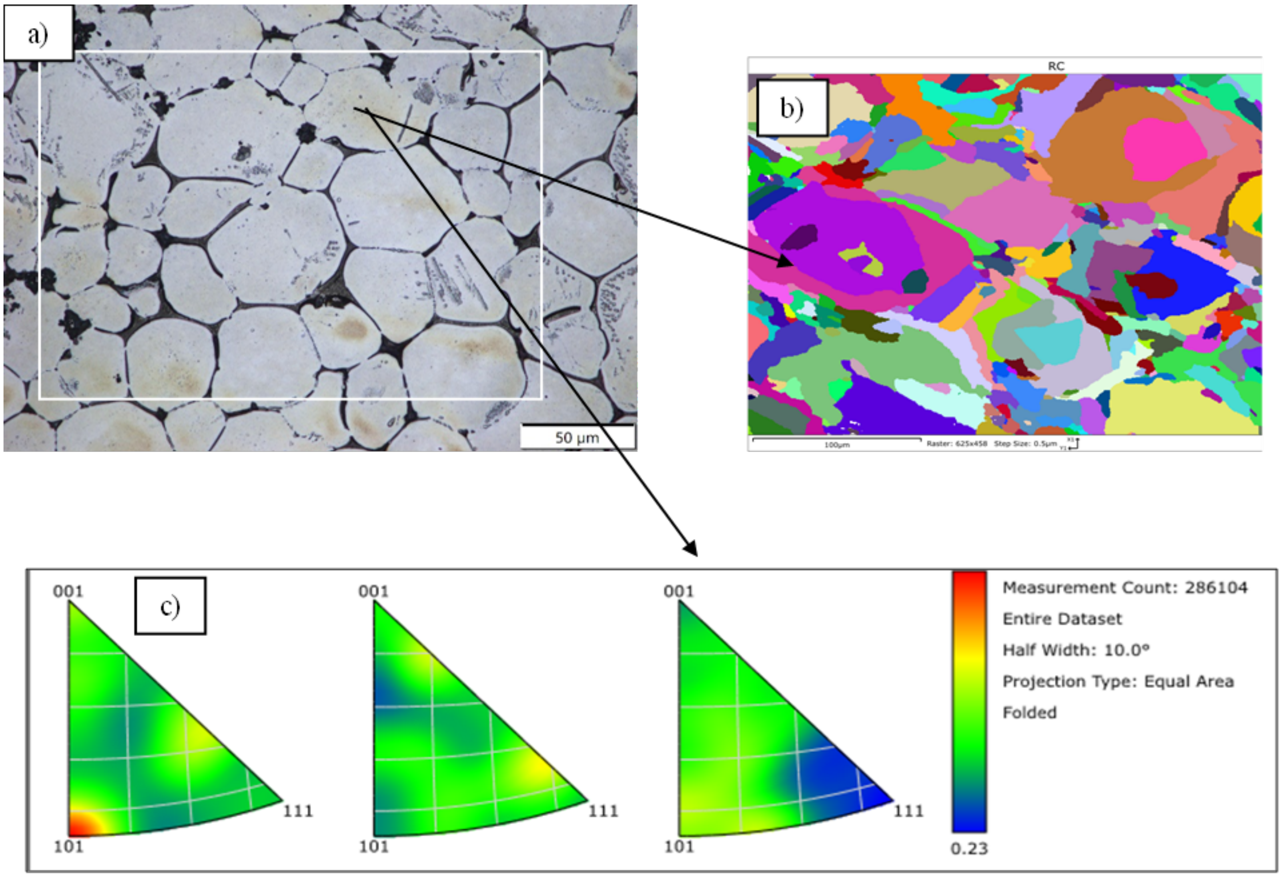

Fig 3. Coloring maps derived by EBSD of Mg - 3 Zn -1Ti (a) top region sample selected area. (b) Colors of grains (c) reverse pole area of three layers

The hardness measurements further support these findings, as the rich-particle outer zone sample demonstrates a hardness of 106 ± 10 Hv, which is 12% higher than that of the middle particle zone sample and 19% higher than that of the inner samples. These results provide evidence that denser particles exhibit higher micro hardness in the top surface region.

3.3 Tensile strength

The particle inner zone has an ultimate tensile strength (UTS) of 270.4 MPa, while the gravity casting of the rich particle outer and middle zone composites (with 3Zn-1Ti+10TCP) has UTS values of 351 and 301 MPa, respectively. The inclusion of 3Zn-1Ti+10TCP in the magnesium matrix leads to a decrease in elongation, resulting in reduced ductility. As a result, the brittle failure mode occurs during testing. Figure 5 provides a graphical representation of the UTS values for the Mg-3Zn-1Ti+10TCP composite alloy. The UTS values exhibit a consistent trend, showing high properties in particle-rich zones and a strong sensitivity to magnesium FGM matrix composite concentrations.

The rich particle outer zone demonstrates superior Ultimate Tensile Strength (UTS) and Yield Strength (YS) compared to the middle and inner zones. The UTS of the rich particle outer zone is 351 MPa, showing a 19.21% increase over the inner zone 266 MPa and an 8.36% increase over the middle zone 301MPa. This difference can be attributed to the unique microstructure

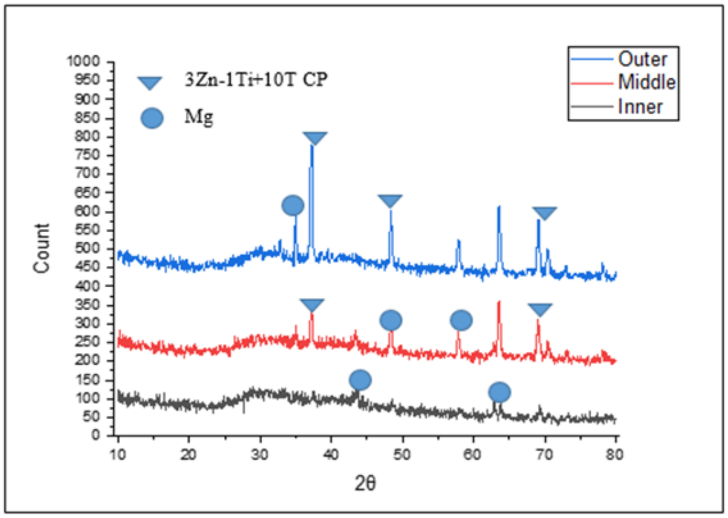

Fig 4. XRD Pattern of FGM samples

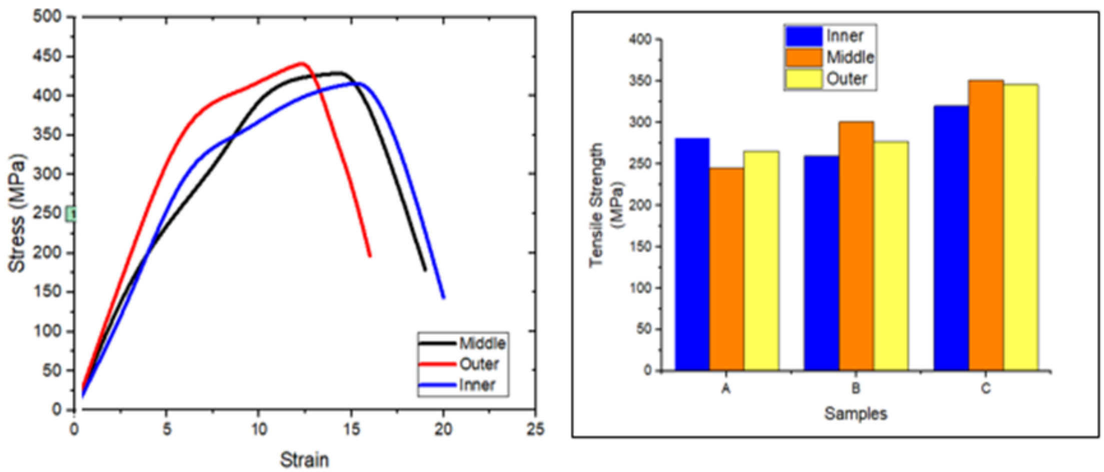

Fig 5. Tensile Strength (MPa) of Mg-3Zn-1Ti + TCP gravity castings

resulting from the rapid cooling rates during centrifugal casting. However, the elongation value at rupture of the outer zone sample indicates lower ductility when compared to the middle and inner zone samples. This reduced ductility is linked to a slight embrittlement caused by the development of inter-dendritic areas with nominal Laves phase. These observations align with the research of **Sumanth et.al**, which highlights how the interdendritic particle propulsion in columnar grains, growing against gravity, contributes to an increase in the brittle nature of the samples. [14]

3.3.1. The tensile fracture surfaces of FGM-matrix composites:

Rich-particle outer zone sample displayed transgranular failure with fine intermetallic-platelet particles and equiaxed dimples, while the FGM-matrix composites showed secondary particles like metal component carbides, resulting in brittle behavior with low plastic deformation. The cracks were notably found in the Ti-rich MC carbides of magnesium.

Composites function as sources of cracks that then spread through the interdendritic areas, leading to the eventual fracturing of the material. The fractured surface of the middle and inner zone samples, as depicted in Figure 6(a) & 6(b), exhibits identical

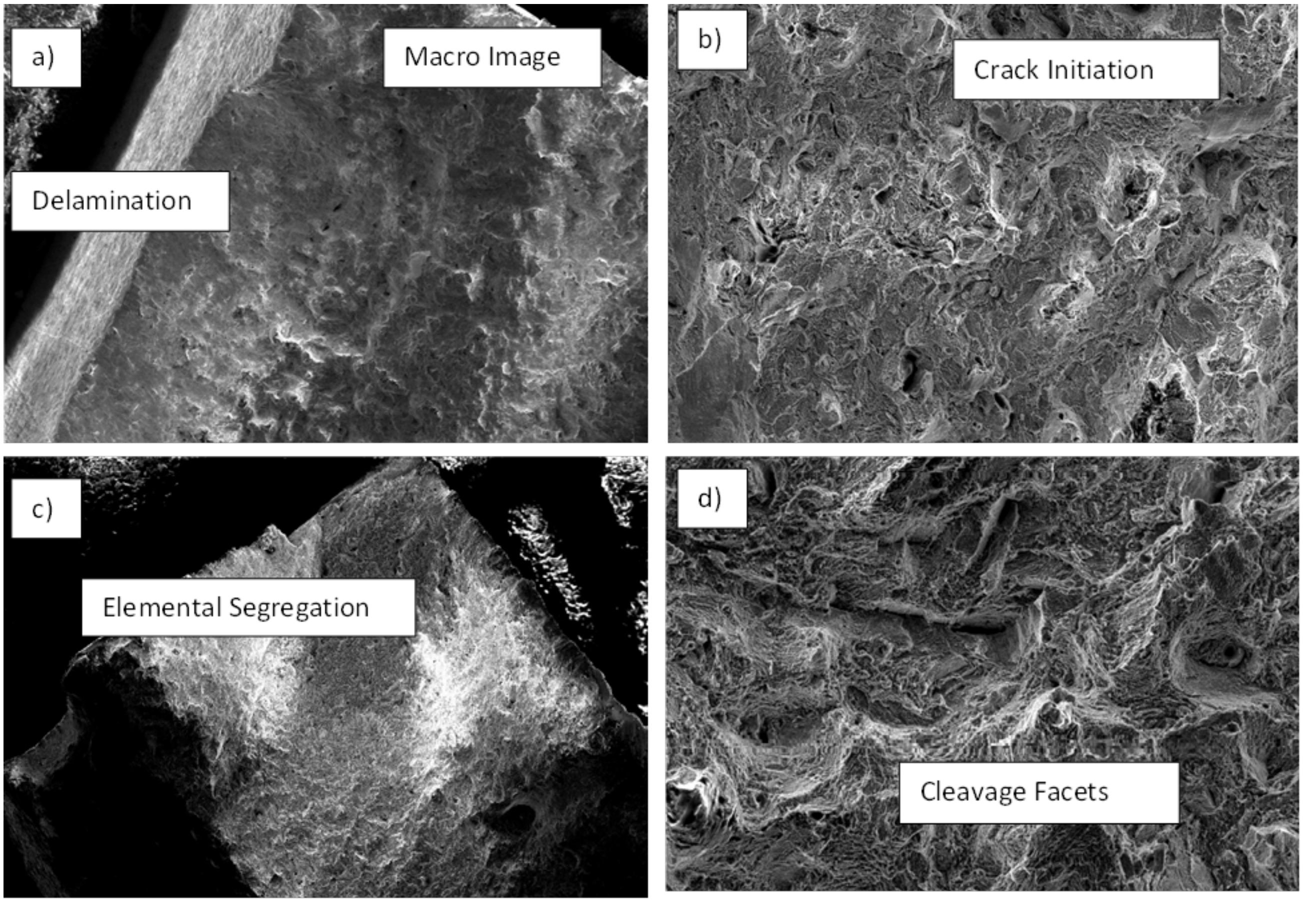

Fig 6. Fractography of (Mg-3Zn-1Ti + TCP) gravity castings

dimple surfaces that are ductile in nature and formed through gravity casting. On these fractured surfaces, intergranular cracking through grain boundaries is predominantly observed. When subjected to tension, the grains in the gravity processed casting samples undergo delamination. The magnesium FGM-matrix composites in the middle and inner zone samples demonstrate dendritic patterns on the fracture surface, suggesting enhanced cracking resistance along the interdendritic regions. The fractures show consistent rupture and equiaxed failures, along with deep dimples.

The fractured surfaces of the outer zone sample, as shown in Figures 6(c) and 6(d), exhibit a unique appearance with Mg-FGM rich particles. Large cleavage facets with irregular dimple locations suggest tensile fracture due to cutting through aligned platelets, while plastic instability between these facets likely resulted in dimpled rupture. Additionally, the ductile fracture pattern of the outer zone sample resembles a cup-and-cone shape, with fine dimples dispersed throughout the fracture surface.

4 Conclusion

This study investigated the FGM- matrix composites (inner, middle, and outer) layers, to analyzing the mechanical and microstructure properties of FGM alloys.

- The microstructure of the Mg FGM matrix composites show the concentration of alpha-Mg phases in the reinforcement zone contributed to the enhanced mechanical properties observed in this area.
- The mechanical properties are improved in the rich-particles outer zone, displayed enhanced hardness, and ultimate tensile strength of 351 MPa. The hardness of the graded nature composites corresponding to the particle concentration, with a peak value of 106 Hv achieved in the particle-rich region.
- This research article pointed the less research on existing Mg or different alloys as compared with sandwich layered FGM composites, which degrade with human body and inherent defects such as porosity, unfused layers.

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