Visual Abstract

# Investigation on microstructure and tensile fractography of RE Oxides ($CeO_2/Y_2O_3$) reinforced AZ91D Magnesium Matrix Composites

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**ABSTRACT.** The current work aims to investigate the mechanical properties of rare oxide reinforced Mg alloy based MMCs. Magnesium matrix considered in the study is AZ91D alloy, whereas rare earth oxides reinforced were $CeO_2$ and $Y_2O_3$. The $Y_2O_3$ particulate reinforcement percentage was varied from 1 to 3% in the steps of 1% to study its influence on mechanical properties of MMCs. Stir casting route was adopted to fabricate sample for study. Microstructure analysis illustrated the uniform distribution of particulate in matrix alloys. The obtained results revealed the enhanced mechanical properties such as tensile strength, yield strength, elongation and hardness of MMCs due to increased percentage of reinforcement. Fractography analysis of fracture surfaces demonstrated the microcracks and cleavage were dominant in pure alloy. While particle debonding, extensive plastics deformation were prominent in-addition to microcracks in MMCs.

**KEYWORDS.** Mechanical Properties, AZ91D, Rare earth oxides, Metal matrix composites.

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## INTRODUCTION

The low-density alloys and its composites are in high demand due to their versatile properties offered at low weight. Metal matrix composites (MMCs) have shown promising characteristics to meet demand of aerospace and spacecraft application which highly demands high specific modulus and high specific strength. Besides, particulate reinforcements comprising carbide, oxide and nitride based ceramics have enhanced the range of applications of metal matrix composites. Commonly employed reinforcements are SiC, $TiO_2$, TiC, $ZrO_2$, $SiO_2$, $B_4C$, graphite, $MoS_2$, etc. These developments have shown the path for further replacement of monolithic alloys in many engineering applications [1-5].

Magnesium (Mg) alloys were found to be more suitable and finding increased low weight applications due to its low density. In addition, Mg alloys as matrix material in MMCs demonstrated excellent characteristics and, also lighter than steel and aluminium [6]. Hence, Mg alloy based MMCs are widely used in structural, automotive, clinical applications. Haghshenas [7] presented an overview of various types of Mg alloys for biomedical applications and mechanical characteristics of biodegradable Mg based MMCs. Research reports published in the recent decade has paid lot of attention to explore the advantages of Mg alloys and its composites. Mehra et al. [8] illustrated that TiC addition to RZ5 Mg matrix has considerably enhanced tensile strength and hardness of MMC. Though, grain size has reduced and exhibited mixed mode fracture behavior due to TiC incorporation. Huang and Ali [9] shown that SiC particulate reinforced AZ61 Mg MMC yields better elastic modulus compared pure AZ61 alloy. Dey and Pandey [10] presented an comprehensive review of characterization of Mg based MMCs. The authors stated that, addition of $B_4C$ particles improves interfacial bonding and flexural strength of Mg based MMCs. While fiber reinforcement in Mg MMCs improves tensile strength at the cost of ductility. Ravichandran et al. [11] depicted that increase in the $B_4C$ concentration in $B_4C$/Mg MMC resulted in the increase of compressive strength and hardness. Fang et al. [12] illustrated that $TiB_2$ addition to Mg alloys aids for the grain refinement and also helps to enhance strength and ductility simultaneously. Meher et al. [13] investigated the influence of TiB2 % on mechanical properties of $TiB_2$/Mg MMC. The study revealed that 8% $TiB_2$ addition increase tensile strength of composite, besides further improvement was achieved through solution treatment of composites. Huang et al. [14] studied the influence of hybrid reinforcement on microstructure and mechanical properties of AZ61 Mg composites. The SiC and $Al_2O_3$ loaded hybrid nanocomposites shown improved hardness, tensile and compressive characteristics. Vijayakumar et al. [15] demonstrated that SiC and boron nitride reinforced hybrid Mg based MMCs Karuppusamy et al. [16] revealed tungsten carbide reinforcement into Mg MMCs has enhanced its load bearing capacity coupled with considerable weight reduction. Khrustalyov et al. [17] illustrated that aluminum nitride nano particles incorporation into Mg enhances the yield strength, tensile strength and plasticity of AZ91 alloy. Thus, hybrid and nanoparticle reinforced Mg based MMCs have resulted better mechanical properties compared to monotype reinforcements. The rare earth elements and particles are other advanced reinforcement types which are explored in the past few years. They have shown promising results with better reinforcement characteristics. Yuan et al. [18] studied the influence of reinforcement of rare earth oxides on microstructure and castable properties of alumina-magnesia alloys. Tun et al. [19] depicted that yttria addition has improved the yield and tensile strength of AZ41 Mg alloy without losing ductility. While yttria addition to AZ51 Mg alloy has shown enhanced compressive strength at same ductility. Further, in both cases fine refined grain structures were obtained due to addition of yttria ($Y_2O_3$) and helped to elimination of coarse and needle structured intermetallics formed during casting of AZ51 and AZ41 alloy. Ponappa et al. [20] have proved that $Y_2O_3$ particles incorporation to AZ91D alloy enhances hardness, Youngs modulus and yield strength of alloy and greatly influences on the microstructure as well as aids for grain refinement. Sharma and Kumar [21] shown that rare earth compound $CeO_2$ particles addition to Al6061/ SiC/$Al_2O_3$ hybrid MMC improves the hardness and causes grain refinement significantly resulting smooth and fine structure. Also, ultimate tensile strength and ductility improvement were also observed due to $CeO_2$ loading. The reported literatures paid attention on improving the mechanical properties and microstructural characteristics through various micro or macro inorganic and organic particles to pure magnesium alloys. In addition recent literatures have demonstrated that, rare earth elements or compound particles also enhances the mechanical properties without reduction of ductility and helps to obtain very fine and smooth microstructure of MMCs. However, there is a lot of scope to investigate the rare earth particulate reinforcements on mechanical and microstructural characterization of Mg alloys. Further, AZ91D alloy finds wide range of automotive applications [22, 23]. Besides, it has been less studied. In this regard, current work attempts to study effect of rare earth oxides viz. $Y_2O_3$ and $CeO_2$ incorporation to AZ91D alloy on its mechanical and microstructure properties.

## EXPERIMENTAL PROCEDURES

### *Materials and specimen fabrication*

Magnesium alloys are widely employed in light weight structural applications like automobile, aircraft, power tool industries. The properties of Mg alloys can be improved through alloying and reinforcement of particulates such as rare earth oxides. Current research employs $Y_2O_3$ and $CeO_2$ powders of 5μm size as reinforcements in the AZ91D matrix based MMC. The percentage of reinforcement was varied and different specimens were fabricated. The composition of matrix alloy is illustrated in Tab. 1.

In the present investigation, preparation of specimen was employed in two stages. In the first stage, AZ91D alloy was prepared by using the pure magnesium with 99.99% purity metal ingots, pure Aluminum with 99.99% purity metal ingots, pure Zinc with 99.99% purity metal ingots, and Al20Mn ingots were melted in a crucible. A microprocessor-monitored electrical resistance furnace under mixture of Argon 98% and 2% SF6 gases environment was used for heating and metals got melting and stirred well and get the AZ91D alloy. In the second phase, Magnesium alloy AZ91D based hybrid MMCs reinforced with different weight percentages of $Y_2O_3$ of Wt. 1%, 2% 3% and $CeO_2$ of Wt. 1% was fabricated through stir-casting method (Fig. 1). The pure $Y_2O_3$ and $CeO_2$ powders of 5µm size and 99.99% purity was procured from US Research Nanomaterials, Inc., USA. The required amount of reinforcement particles was pre-heated. Preheated reinforcements were added to the Magnesium alloy crucible. The customized steel stirrer was utilized to attain uniform dispersion of particulates. The molten mixture was rotated with the help of stirrer at 600 rpm speed for 4 min at 650°C. The melt was then heated to 700°C and poured into a preheated die steel mold. While desired specimens were yielded for mechanical and microstructural characterization as per ASTM standards.

| Element | Al | Zn | Mn | Fe | Cu | Ni | Si | Other | Mg |
|---|---|---|---|---|---|---|---|---|---|
| wt % | 9.1 | 0.85 | 0.15 | 0.005 | 0.003 | 0.002 | 0.05 | 0.03 | Bal. |

Table 1: The Chemical composition (wt. %) AZ91D alloy.

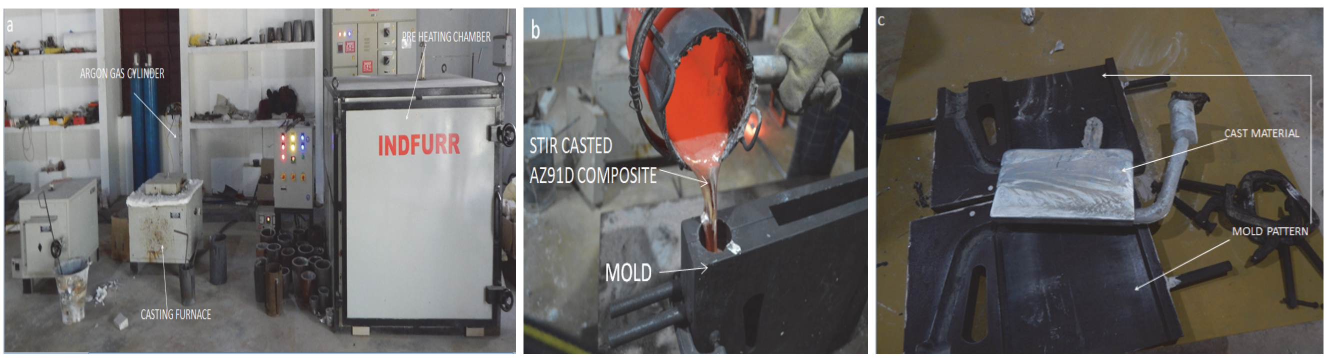


Figure 1: a) Stir casting setup b) Pouring into mold c) De-moulding.

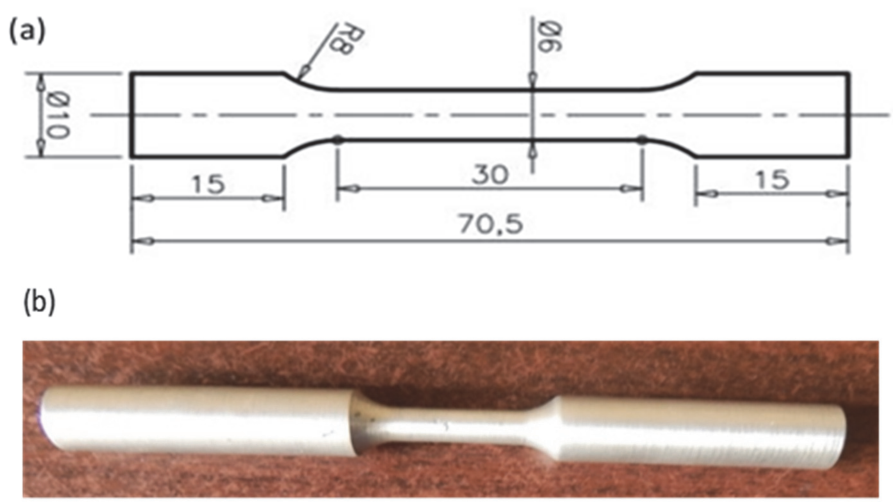


Figure 2: Specifications of tensile test sample (a) Dimensions of the model in mm as per ASTM E8M-16A and (b) Tensile test specimens as per ASTM E8M-16A.

### *Materials Characterization*

Tensile test specimens (Fig. 2) were yielded from the obtained casts as per ASTM E8M-16A standard. According to the ASTM E8M-16A standard, tensile test sample were prepared using CNC machine with gauge length of 30 mm and diameter of 6 mm. The specifications of tensile test sample model and test specimen as per ASTM E8M-16A are shown in Fig. 2. Subsequently, tensile testing was carried out to investigate tensile properties of prepared samples.

Rockwell ball hardness tests were performed to study hardness properties of prepared samples as per ASTM E18-20 standard and optical microscopy was used to study microstructural characteristics and to examine uniform distribution of particles in matrix alloy. Finally, scanning electron microscopy (SEM) images of failed specimens were analyzed to study their fracture behavior.

## RESULTS AND DISCUSSION

### *Microstructural characterization*

The rare earth compounds viz. $Y_2O_3$ and $CeO_2$ were incorporated into AZ91D Mg alloy in the current work. Microstructure of prepared samples of MMCs were studied through optical microscopy as illustrated in Fig. 3 (a) – (d). The microstructure of alloy and MMCs revealed the dispersion of β-phase in α-phase and nearly uniform distribution of particulates in the matrix. Further, pure matrix alloy illustrates large grain structure and intermetallic formation at the interface. Fig. 3(b) depicts that addition of 1% $Y_2O_3$ and 1% $CeO_2$ has refined the grain structure. While the refinement of grain structure enhanced with the increase in the percentage of $Y_2O_3$, which can be found in Fig. 3(c) and Fig. 3(d). Also, coarse structure present in the pure alloy has been broken and formed dentritic structure in the 1% $Y_2O_3$ +1% $CeO_2$ incorporated MMCs. It was further enhanced with 3% $Y_2O_3$ +1% $CeO_2$ reinforcement in the alloy. Fig 3(c) reveals the dentritic skeleton structure occurrence in the α-phase of MMC with fine and smooth microstructure. Fig. 3(d) shows the further refinement of α-phase, but simultaneously dispersion of β-phase has increased to greater extent. The β-phase increase has resulted in the better interconnected intermetallic compound. Also, Similar results were found in the earlier literature elsewhere for $Y_2O_3$/Mg MMC [24-29].

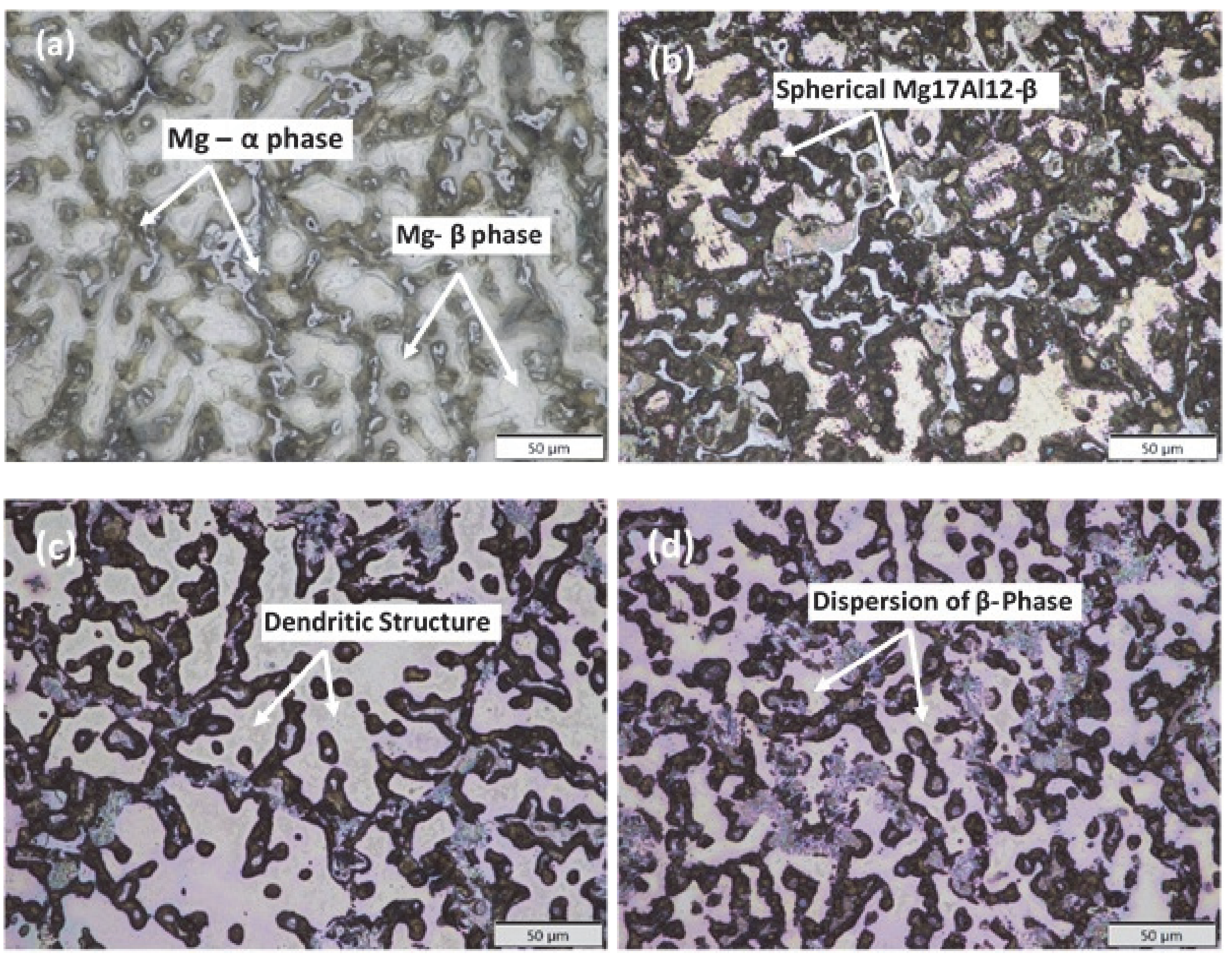


Figure 3: Microstructure of (a) AZ91D Magnesium Alloy; (b) AZ91D+1%$Y_2O_3$+1%$CeO_2$; (c) AZ91D+2%$Y_2O_3$+1%$CeO_2$; (d) AZ91D+3%$Y_2O_3$+1%$CeO_2$ hybrid composites.

The X-ray diffraction patterns of as-cast alloy, AZ91D+1%$Y_2O_3$+1%$CeO_2$, Z91D+2%$Y_2O_3$+1%$CeO_2$ and AZ91D+3%$Y_2O_3$+1%$CeO_2$ hybrid composites are shown in Fig. 4. The X-ray diffraction pattern of as-cast alloy sample indicated the Mg-α phase and Mg17Al12-β phases. Furthermore, X-ray diffraction patterns shows the some additional peaks in the AZ91D Mg alloy hybrid composites, which corresponded to Al11Y and Al11Ce intermetallic compounds. It is indicated that the X-ray diffraction patterns of all the AZ91D Mg alloy hybrid composites containing Mg-α phase and Mg17Al12-β phases along with Al11Y and Al11Ce components. It shows that the high affinity of yttrium (Y) and cerium (Ce) elements in RE reinforced AZ91D hybrid composites (Fig. 3(b) to (d)). It can be concluded that the β-phase helps in the better interconnected intermetallic compound. Also, The XRD analysis results revealed the β-phase growths in the AZ91D+3%$Y_2O_3$+1%$CeO_2$ hybrid composite (needle like structures in Fig. 3(d)) when compared to as-cast and other hybrid composites.

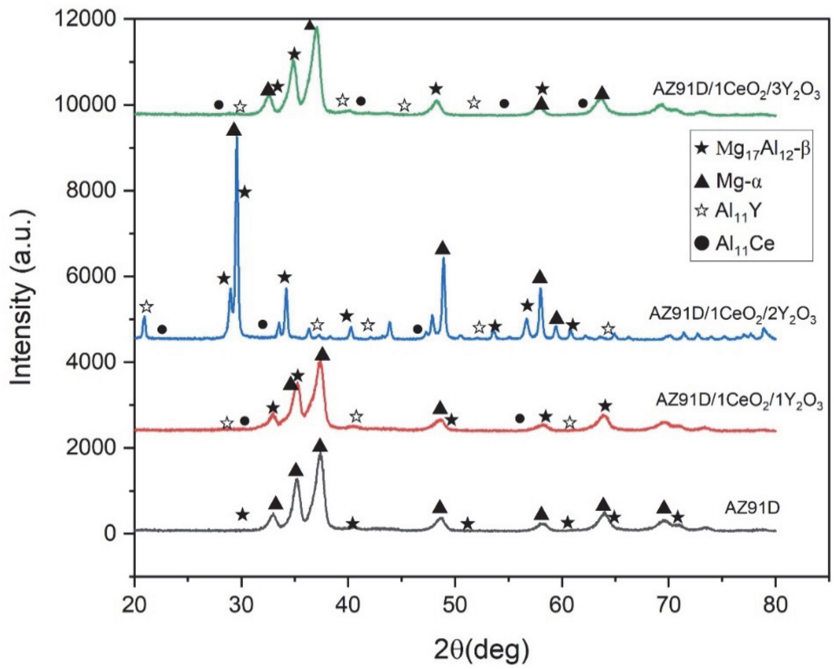


Figure 4: X-ray diffraction patterns of as-cast (AZ91D) alloy, AZ91D+1%$Y_2O_3$+1%$CeO_2$, Z91D+2%$Y_2O_3$+1%$CeO_2$ and AZ91D+3%$Y_2O_3$+1%$CeO_2$ hybrid composites.

### *Hardness*

In the current study, hardness of pure alloys and MMCs were characterized through Brinell hardness test using ball indenter of diameter 2.5mm at load 187.5kgf. Three readings were taken for each sample and average HBW values were considered for investigation. The hardness variation of different studied samples is illustrated as Fig. 5. Hardness was found to increase with the increase in the percentage of reinforcement particles in the MMCs till 2% of yttria. Also, compared to pure alloy, reinforcement of rare earth oxides has beneficial effect on improving hardness. It owes to the presence of uniformly dispersed $Y_2O_3$ and $CeO_2$, which yielded fine structure and increased resistance to plastic deformation. The hardness has decreased further with increase of yttria to 3%, suggesting lucrative effect of increasing yttria. It may be attributed to the increased β-phase with interconnected intermetallic phases. Thus, resistance to plastic deformation increased, which lead to greater hardness at higher value of reinforcement.

### *Tensile Properties*

Tensile characteristics such as ultimate tensile strength, yield strength and elongation of any material are of interest for material scientists to employ them for engineering applications. Thus, material characterization of studied samples is significant to demonstrate its applications. In the present work, tensile strength, elongation and yield strength of different MMCs were studied and represented respectively as Fig. 6, 7 and 8. Fig. 6-8 indicates the increase of studied mechanical characteristics with the increase in the percentage of yttria, also found that MMCs shown superior properties over pure Mg alloys. Besides increase in the percentage of elongation suggests that ductility has improved with the concentration of yttria in MMCs. Thus, it can be inferred as simultaneous enhancement of strength and ductility with the incorporation of yttria. The obtained results agree with reported literature for RZ5 alloy and RZ5 with 10% TiC MMC [8, 30, 31]. The increase in strength may be due to the increased particulate reinforcements, which aids to develop energy barrier for dislocation movement. The applied load during tensile testing is uniformly transferred and distributed among reinforcement particulates in the matrix alloys. Subsequently, stress concentration developed around particulate might have caused localized damaged such as inclusion micro-cracks, particulate de-bond, leading to improved strength at yield stress. In addition, the better bonding of reinforcement with Mg matrix and its uniform dispersion as evident from microstructure caused β-phase increase. Thus, it owes to resulted improved ductility of MMCs at greater reinforcement concentration. Therefore, it can be concluded that rare earth oxide incorporation causes simultaneous improvement of strength and ductility, which is highly lucrative for engineering applications.

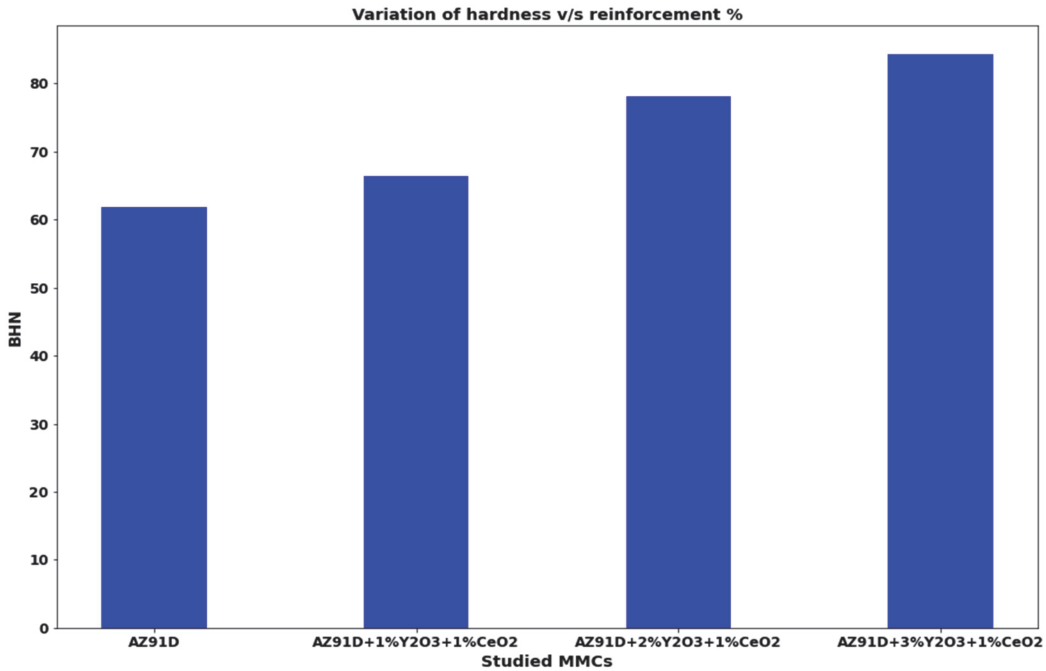


Figure 5: Hardness values of investigated materials.

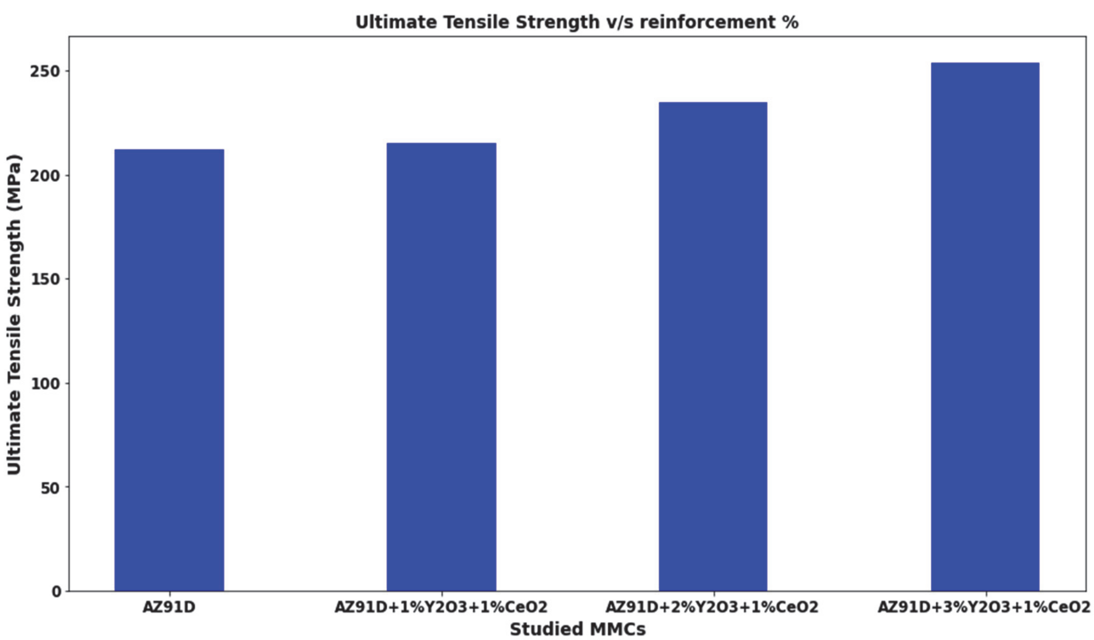


Figure 6: Ultimate Tensile Strength of investigated materials.

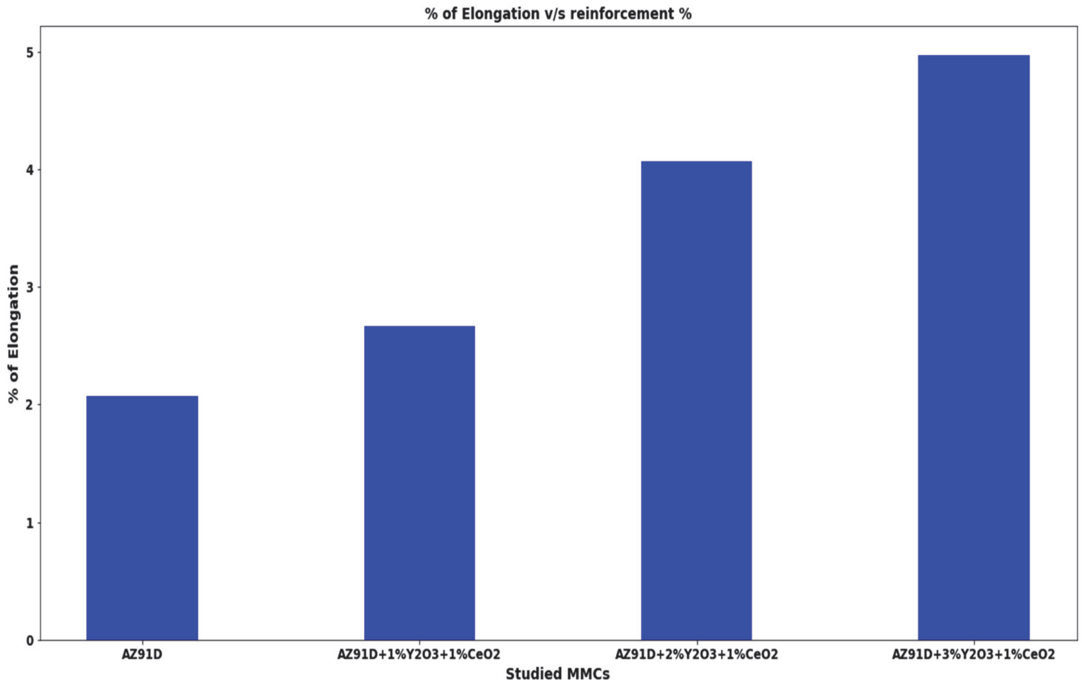


Figure 7: Percentage of Elongation of investigated materials.

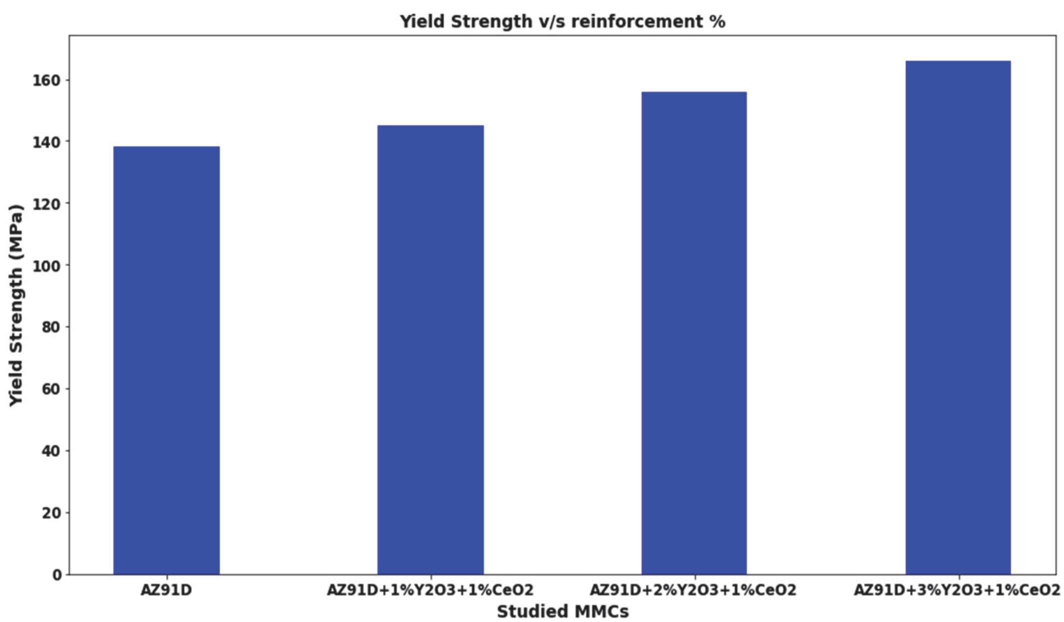


Figure 8: Yield Strength of investigated materials.

### *Fractography Analysis*

The fractured surfaces of studied samples were analyzed using SEM images as illustrated in Fig. 9(a)-(d). The fractographic analysis reveals existence microcracks, particulate debonding, cleavage, extensive plastic deformation of matrix and dimples on fractured surface. Fig.9(a) demonstrates that pure Mg alloy studied with widespread cleavage and nucleated microcracks. The initiated caused the movement of dislocation, resulted fracture of material. Fig. 9(b) illustrates fracture surface of 1% $Y_2O_3$ +1% $CeO_2$ incorporated alloy. It shows the presence of craters due to debonding of particulate reinforcements during tensile failure. During tensile, possibly inclusions get separated due to transferred tensile load. Fig. 9(c) suggests that extensive plastic deformation is one of the major failure mechanism for the tensile fracture of 2% $Y_2O_3$ +1% $CeO_2$ incorporated alloy. The increased plastic deformation may be due to the increased yttria incorporation, which increases the required energy resistance for dislocation movement. Thereby, increasing the strength of material studied. Whereas initiation of agglomeration was noticed in AZ91D+3% $Y_2O_3$ +1% $CeO_2$ MMCs (Fig. 9(d)), suggesting threshold limit for nano-particulate reinforcement percentage. The existence of threshold limit for the reinforcement percentage is also reported for $Si_3Ni_4$ reinforced Al MMCs[32-35]. Thus improved mechanical properties of MMCs at higher percentage was marginal compared to successive lower percentage of incorporation of particulate.

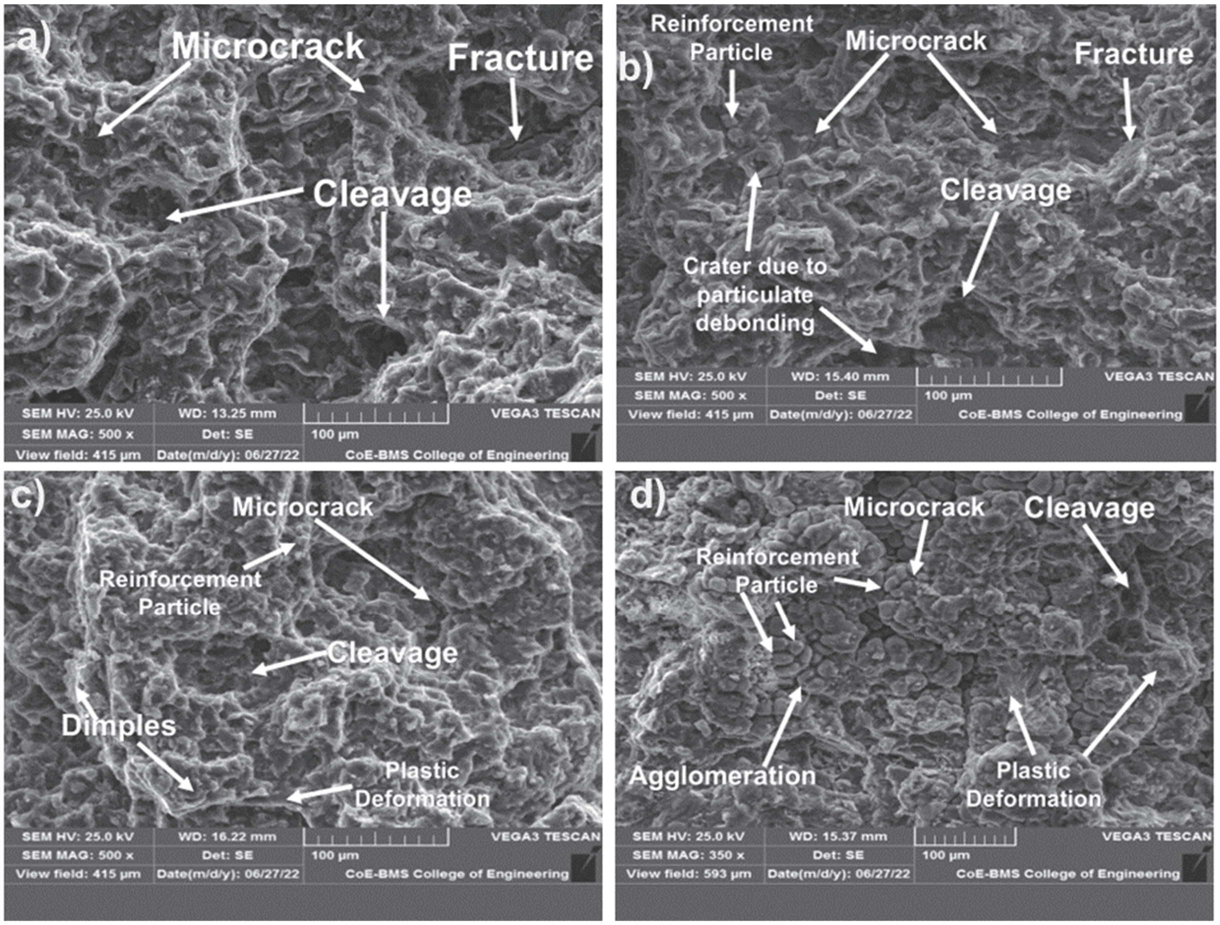


Figure 9: Fractured Surfaces of (a) AZ91D Alloy; (b) AZ91D Alloy+1% $Y_2O_3$ +1% $CeO_2$; (c) AZ91D Alloy+2% $Y_2O_3$ +1% $CeO_2$; (d) AZ91D Alloy+3% $Y_2O_3$ +1% $CeO_2$ hybrid composites.

## Conclusions

The present work investigates the mechanical properties of rare earth oxides viz. $Y_2O_3$ and $CeO_2$ incorporated AZ91D alloy based MMCs. The influence of $Y_2O_3$ % on mechanical properties and fracture behavior of Mg based MMCs. Summary of studied composites with their results and analysis is as follows:

1. AZ91D Mg alloy was reinforced with varying percentage of $Y_2O_3$ from 1 to 3 in the steps of 1% with addition of 1% $CeO_2$. Comparison of MMCs results with pure alloys was done to examine the improvement of mechanical properties. Samples were prepared through stir casting technique as per ASTM standards.
2. Microstructure of prepared samples were studied to understand the dispersion of particles in matrix. The microstructure of studied samples indicates that there was nearly uniform dispersion of particulates in matrix. The α-phase was dominant in pure alloy, whereas β-phase dispersion in α -phase was pronouncing in MMCs.
3. Mechanical properties such as tensile strength, yield strength, elongation and hardness were studied. The significant improvement of studied mechanical properties were achieved due to reinforcement of rare earth oxides considered in the study. It may be due to greater resistance offered for dislocation movement.
4. Fractography analysis was done to examine the possible fracture mechanism during failure. The pure Mg alloy indicated microcracks and cleavage as dominant failure mechanism. While, MMCs suggested, debonding and

displacement of reinforcement particles at higher tensile load, cleavages, microcracks and extensive plastic deformation were prominent mechanisms over fractured surfaces.

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