The influence of rare earth cerium addition on mechanical and corrosion properties cast Mg-6Al-1Zn magnesium alloy

S Manivannan1, J Vairamuthu2, Samuel Tilahun3, M D Vijayakumar4
C Ramesh Kannan5 and B Stalin6

1 Department of Mechanical Engineering, Karpagam Academy of Higher Education, Coimbatore-21, Tamil Nadu, India.
2 Department of Mechanical Engineering, Sethu Institute of Technology, Pulloor- 626 115, Kariapatti, Tamil Nadu, India.
3 Department of Mechanical Engineering, School of Mechanical and Automotive Engineering, College of Engineering and Technology, Dilla University, Dilla, Ethiopia.
4 Department of Mechanical Engineering, Chennai Institute of Technology, Kundrathur, Chennai-600 069, Tamil Nadu, India.
5 Department of Mechanical Engineering, PET Engineering College, Tirunelveli, Tamil Nadu, India.
6 Department of Mechanical Engineering, Anna University, Regional Campus Madurai, Madurai - 625 019, Tamil Nadu, India.

* Corresponding author: manivannan.meta@gmail.com

Abstract. The present study investigates the effect of Ce addition on the microstructure of Mg-6Al-1Zn alloy with the use of optical microscopy (OM), X-ray diffraction analysis (XRD) and scanning electron microscopy (SEM). The electrochemical corrosion behaviour of Mg-6Al-1Zn alloyed with different levels of cerium was investigated using static weight loss and polarization corrosion tests in 3.5% NaCl solution. The microstructural study reveals the grain refinement of the matrix and the stability of the phase β(Mg17Al12) were disintegrated into Al4Ce and distributed along the grain boundary. The literatures show that the intermetallic Al4Ce has a high melting point and expected to show better strength behaviour at relatively high temperatures. The electrochemical results show that cerium addition above 1.5wt.% increase the corrosion current density of the developed alloy which resulted in increasing corrosion rate. The best results of refined microstructure and corrosion resistance were obtained with 1.5wt.% cerium addition.

Keywords. Corrosion, Al4Ce, Microhardness, Immersion Test, Electrochemical analysis, Cerium.

1. Introduction
Being a lightest metallic structural materials with high specific strength, Magnesium alloys are likely to be applied in industries such as aerospace, automotive and railways [1-2]. However, the mechanical properties are generally lower compared with aluminium and ferrous alloys. The ductility is also less because of its HCP crystal structure [3-5]. These stated disadvantages are substantial obstacles against their utilities in many applications. To meet the property demands of such applications, magnesium alloys with better mechanical properties must be developed [6-10]. Magnesium alloys with some
amount of Al and Zn addition have found widespread in automotive applications [11-16]. Most of the studies show that the corrosion behaviour of Mg–Al alloys are highly dependent on their microstructure, which predominantly depends on the refined grain size, their amount and distribution of the intermetallic in the matrix [17]. The presence of Mg17Al12 phase in an AZ alloy could act as either a corrosion barrier or as a galvanic cathode accelerating corrosion [18-23]. Hence in the present study, the influence of cerium addition on the Mg-6Al-1Zn magnesium alloy and their effect on microstructure, mechanical properties and corrosion resistance has to be investigated.

2. Experimental procedure
Mg (99.98%), Al (99.99%) and Zn (99.98%) materials of high purity were purchased and melted in an electric resistance furnace with Inconel crucible under controlled atmosphere of 99.5%Ar+0.5%SF6 gas mixture. The gas mixture with a flow rate of 0.25 m³/hr had been sent continuously to the chamber so that liquid metal has been protected from the atmospheric air. The flow was maintained till the melt temperature reaches 750°C, then the rare earth element was added into the melt in amounts of 0.5, 1.0, 1.5 and 2.0 wt.% Ce and reaction time of 30 minutes was allowed for complete dissolution of Ce with melt. The alloy composition was confirmed by using Atomic Emission Spectroscopy (AES) technique and its compositions were listed in Table 1.

| Table 1. Level of control parameters. |
|--------------------------------------|
| Alloy | Al  | Zn  | Ce  | Mn  | Mg  |
|-------|-----|-----|-----|-----|-----|
| Mg-6Al-1Zn  | 5.546 | 1.008 | -   | 0.33 | Bal.   |
| Mg-6Al-1Zn+0.5Ce | 5.546 | 1.008 | 0.49 | 0.33 | Bal. |
| Mg-6Al-1Zn+1.0Ce | 5.504 | 1.009 | 8.910 | 0.33 | Bal. |
| Mg-6Al-1Zn+1.5Ce | 5.582 | 0.9163 | 1.504 | 0.31 | Bal. |
| Mg-6Al-1Zn+2.0Ce | 5.415 | 0.9604 | 2.010 | 0.32 | Bal. |

The alloys of different composition were cast using a preheated mild steel mould. The cast samples of 25 x 300 mm diameter cylindrical shape rods were produced. The samples were cut into appropriate sizes for chemical analysis, Microscopic analysis, corrosion test and XRD analysis. For microstructural analysis, the cut specimens were cold mounted, polished and etched by using 2% metal (2%HNO3+98% ethanol), held for 5-10 Sec and then observed by optical microscopy using DIC Leica microscope, Model No:DM750M. Phase analysis was confirmed with X-Ray Diffraction technique. Microhardness studies were performed in intermetallic phases and the matrix by using Matsuzawa Model NO. MMT-X7 microhardness tester. The density of the experimental alloys was studied using a Mettler density tester. Both the refined and un-refined alloys were observed using Hitachi Scanning Electron Microscopy (SEM) equipped with energy dispersive spectrometer (EDS). The specimen dimensions of 10mm x 10mm x 4mm in size was used for potentiodynamic polarization (PDP) test using ACM Gill equipment and the exposed area was 1 cm² in 3.5wt.% NaCl electrolyte with Mg-6Al-1Zn+XCe as working electrode, Platinum as auxiliary electrode and silver chloride as reference electrode (saturated calomel electrode). Finally immersion corrosion test also were conducted on the developed alloys.

3. Results and Discussions

3.1. Effect of cerium on microstructures of as cast alloys
Fig.1 shows the microstructure of base Mg-6Al-1Zn magnesium alloy. The alloy is composed of primary α (Mg) solid solution of aluminium in magnesium and the intermetallic compound β (Mg17Al12). The alloy starts to solidify from liquid state at about 620°C. On further cooling, complete solidification occurs at around 540°C. The material is in this state till around 300°C. Below 300°C β-phase precipitation and growth of mass fraction of secondary phase occur. At below 100°C only 7.5% material is in solid phase [17,18].
3.2. Microstructures of Mg-6Al-1Zn+XCe alloys
When cerium is added to Mg-6Al-1Zn, it is possible to form Al-Ce and Mg-Ce compound. This is dependent on its chemical activity and the capability of reaction with other elements like Al or Mg. Because it has positive electronic characteristics with both Al and Mg, Ce is likely to react with Al in priority. From Al-Ce binary phase diagram Al4Ce can be formed when Ce content is less than 60%. As a result, much Al is bounded by Al4Ce, and the amount of Al for the formation of β(Mg17Al12) phase is tend to be decreased. Therefore, the precipitation of β phase has suppressed by the formation of Al4Ce. It can greatly increases the Mg–Al–Zn magnesium alloy corrosion resistances with significant refinement of β phase.

3.3. Effect of rare earth addition on mechanical properties
The change in microhardness was observed on $\beta$ (Mg$\text{17Al}_{12}$) phase and $\alpha$-matrix in experimental alloys.

Figure 3. Microhardness of (a) $\alpha$-Phase (b)$\beta$ (Mg$\text{17Al}_{12}$) phase in Mg$\text{-6Al-1Zn +XCe}$ alloys

Fig.3 (a) and Fig.3 (b) shows the microhardness of $\alpha$-Phase and $\beta$(Mg$\text{17Al}_{12}$) phase in Mg$\text{-6Al-1Zn +XCe}$ magnesium alloys. The results indicate that the Ce addition increase the hardness of $\alpha$-phase also there is an enhancement in microhardness with increase in cerium content [as shown in Fig.3 (a)] which is due to the refinement of $\beta$(Mg$\text{17Al}_{12}$) phase and Al$\text{4Ce}$ strengthening phase. Cerium addition increases the stability and strength of the $\beta$(Mg$\text{17Al}_{12}$) phase, which has confirmed from the microhardness results in Fig.3(b). High hardness was obtained in 1.5wt.% Ce addition and further addition reduces the hardness. Fig.4 show the tensile results of Mg$\text{-6Al-1Zn +XCe}$ alloys. It was found that the high value of UTS and Yield Strength was obtained in 1.5wt.% Ce addition. And further increases of cerium content decreases the UTS and Yield Strength and with a better elongation obtained at 2.0wt.% Ce.

Figure 4. Mechanical Properties of Mg$\text{-6Al-1Zn +XCe}$ alloys (X=0.0, 0.5, 1.0 and 2.0wt. % Ce)
3.4. **SEM and EDS analysis**

![Figure 5](image)  
**Figure 5** (a) and (b) SEM and EDS Analysis of Mg-6Al-1Zn+1.5wt.%Ce alloy

Based on SEM analysis, the rod-like structure was confirmed to the $\beta$(Mg$_{17}$Al$_{12}$) intermetallic phase and small needle like phase was Al$_4$Ce. More refinement of alpha phase was obtained in 1.5wt.% cerium addition that was shown in Fig.5(a). The cerium addition refines the alpha phase thereby results in more rod like intermetallic $\beta$(Mg$_{17}$Al$_{12}$) phase. Fig.5 (b) show the EDS pattern indicated the compositions of alpha and intermetallic pages present in Mg-6Al-1Zn+1.5Ce. Further increases of cerium addition in the Mg-6Al-1Zn alloy, the volume fraction of Al$_4$Ce phase was increased and the size of the intermetallic phase was found it coarsened.

3.5. **XRD analysis**

Fig. 6 shows the XRD pattern of Mg-6Al-1Zn+XCe alloys (Where X=0.0,0.5, 1.5 and 2.0wt.% Ce). From XRD spectrum, it was confirmed that Ce-containing alloys have four different phases that are $\alpha$-phase, $\beta$-phase (Mg$_{17}$Al$_{12}$), MgAl and a new phase as Al$_4$Ce. It was found that from the XRD results, increasing cerium content in Mg-6Al-1Zn alloys decreased the amount of $\alpha$-phase, $\beta$-phase (Mg$_{17}$Al$_{12}$) and there were formation of new phases such as MgAl, Al$_4$Ce.
3.6. Density Test

Table 2 shows the density test results of Mg-Al-Zn+xCe alloys. Based on the results of density test (Table 2) it was found that, increasing the cerium content of this experimental alloy increases the density due to the presence of high density intermetallic phases like β-phase (Mg17Al12) and Al4Ce.

**Table 2. Density Results OF MG-6AL-1ZN+XCE Alloys (WHERE X=0.0, 0.5, 1.5 AND 2.0WT. %CE).**

| S.No | Alloy | Density (gm/cc) |
|------|-------|-----------------|
| 1    | Mg-6Al-1Zn | 1.7770 |
| 2    | Mg-6Al-1Zn+0.5wt.% Ce | 1.7710 |
| 3    | Mg-6Al-1Zn+1.0wt.% Ce | 1.7731 |
| 4    | Mg-6Al-1Zn+1.5wt.% Ce | 1.7749 |
| 5    | Mg-6Al-1Zn+2.0wt.% Ce | 1.7801 |

3.7. Immersion Test

Based on the immersion test results, the corrosion rate of the cerium alloyed sample was calculated through weight loss method as per the ASTM G31-72 standard [6]. The test specimens were prepared and evaluated as per the ASTM G1-03 standard [7].

The corrosion rate of Mg-6Al-1Zn+xCe alloys were calculated by using the equation 1.

\[
\text{Corrosion Rate (C.R)} = \frac{87.6 \times W}{(D \times A \times T)} \text{mm/year} \quad \text{equation 1}
\]

Where, \( W = \text{weight loss in milligrams (mg)} \)

\[ D = \text{Density of material (g/cm}^3\) \]
A = Area (cm²)

T = Time of Exposure (hour)

Fig. 7 shows the macro-structures of the exposed samples (in chloride solution). The influence of immersion time on the corrosion rate of Mg-6Al-1Zn+XCe magnesium alloy has been observed. The corrosion behaviour of these cerium alloyed samples was evaluated by performing immersion test with a total period of 144 hours at room temperature. The samples were taken periodically and observed at each 24 hours interval for any damage and also to find the weight loss of the material (Table 2). The corrosion data after the immersion test are presented in Table 3. The results show that increase in immersion time reduces the corrosion rate. The better corrosion resistance was observed in 1.5wt.% Ce addition.

Figure 7. Samples after immersion test (a.) 24, (b.) 48, (c.) 72,(d.) 96, (e.) 120, and (f.) 144 hours
Fig. 8 shows the plot between Corrosion rates (mmpy) versus Time (hour). Immersion test results indicates that the addition level of 1.5wt.% Ce to Mg-6Al-1Zn magnesium alloy improves resistance to corrosion and weight reduction in 3.5% NaCl environment when compared to other compositions of Mg-6Al-1Zn magnesium alloys. This is due to the refinement of β phase and Al4Ce strengthening phase. Normally the β phase is very corrosion resistant in AZ alloys and it is not corroded whenever exposed to a NaCl solution. The Ce addition resulted in more continuous β phase, which has been considered as a more effective barrier and hence it will acts as a corrosion barrier also the corrosion rate should be low after a steady state surface condition is reached.

### Table 3. Weight Reduction of samples after 24, 48, 72, 96, 120 and 144 hours

| Alloy (Mg-6Al-1Zn) | Weight after “X” hours (g) |
|---------------------|----------------------------|
|                     | 24 hours | 48 hours | 72 hours | 96 hours | 120 hours | 144 hours |
| 0.0wt.%Ce           | 1.648    | 1.591    | 1.585    | 1.578    | 1.593     | 1.417     |
| 0.5wt.%Ce           | 3.269    | 3.259    | 3.242    | 3.232    | 3.219     | 3.199     |
| 1.0wt.%Ce           | 3.460    | 3.450    | 3.432    | 3.423    | 3.411     | 3.399     |
| 1.5wt.%Ce           | 3.625    | 3.620    | 3.614    | 3.609    | 3.599     | 3.591     |
| 2.0wt.%Ce           | 4.293    | 4.280    | 4.273    | 4.259    | 4.243     | 4.230     |

### Table 4. Corrosion rate of samples after 24, 48, 72, 96, 120 and 144 hours

| Alloy(Mg-6Al-1Zn) | Corrosion rate after “X” hours (mmpy) |
|-------------------|--------------------------------------|
|                   | 24 hours | 48 hours | 72 hours | 96 hours | 120 hours | 144 hours |
| 0.0wt.%Ce         | 207      | 645      | 692      | 711      | 736       | 756       |
| 0.5wt.%Ce         | 60       | 104      | 154      | 165      | 180       | 190       |
| 1.0wt.%Ce         | 91       | 122      | 164      | 180      | 200       | 210       |
| 1.5wt.%Ce         | 30       | 54       | 65       | 68       | 85        | 91        |
| 2.0wt.%Ce         | 54       | 97       | 127      | 154      | 170       | 182       |
3.8. Microstructure of Mg-6Al-1Zn+XCe alloy after immersion test

![Microstructures of Mg-6Al-1Zn+XCe alloys](image)

**Figure 9.** Microstructures of Mg-6Al-1Zn+XCe (a.) 0.5, (b.) 1.0, (c.) 1.5 and (d.) 2.0 alloys after Immersion test.

Fig. 9 and 10 shows the corroded surface of both unrefined and refined alloy. In both cases the corroded surface exhibited the case of Mg-6Al-1Zn+XCe alloys two characteristics: one was the runoff of corrosion film from corrosion surface as in 1.5wt.% Ce the other was severe pitting corrosion. The addition of cerium in Mg-6Al-1Zn magnesium alloy reduce the pitting corrosion was avoided which shows in Fig.10 that its addition reduces the corrosion rate.
3.9. Potentiodynamic Polarization Test

![Graph A](image1.png)

**Figure 10.** Variations of current density with applied voltage for Mg-6Al-1Zn+XCe (X=0.0, 0.5, 1.0 and 2.0wt. % Ce) (a). Base Mg-6Al-1Zn magnesium alloy. (b). Mg-6Al-1Zn+XCe magnesium alloy.
Table 5. Ecorr and Icorr values of Mg-6Al-1Zn and Mg-6Al-1Zn+XCe samples

| Alloy                        | Ecorr (mV) | Icorr (mA/cm²) |
|------------------------------|------------|----------------|
| Mg-6Al-1Zn                   | -253.66    | 2.623×10⁻²     |
| Mg-6Al-1Zn +0.5w.%Ce         | -374.12    | 7.275×10⁻⁴     |
| Mg-6Al-1Zn +1.0wt.%Ce        | -315.07    | 1.118×10⁻⁴     |
| Mg-6Al-1Zn +1.5wt.%Ce        | -241.42    | 1.366×10⁻⁵     |
| Mg-6Al-1Zn +2.0wt.%Ce        | -237.26    | 3.342×10⁻⁴     |

The potentiodynamic polarization curves of cast Mg-6Al-1Zn+xCe (X=0.0, 0.5, 1.0 and 2.0wt. % Ce) magnesium alloy in 3.5wt.% NaCl solution are shown in Fig.11. Without the addition of cerium in Mg-6Al-1Zn base, magnesium alloy shows active-passive behaviour Fig.11 (a). With cerium addition in Mg-6Al-1Zn+xCe (X=0.0, 0.5, 1.0 and 2.0wt. % Ce) magnesium alloy shows different active-passive behaviour in polarization curves are shown in Fig.11 (b). The anodic current density increases with increasing applied potential [24-31]. The metal dissolution has been observed due to the high chemical activity of cerium. The dissolution takes place in increases of current density with increasing of anodic potential. The passive current density of Mg-6Al-1Zn+xCe(X=0.0, 0.5, 1.0 and 2.0wt. % Ce) magnesium alloy goes down with increasing cerium content. The values of corrosion potential (Ecorr) and corrosion current density (icorr) for base material Mg-6Al-1Zn and cerium added alloys were presented in Table 5. Based on the PDP curves and values (Table 5) were found that the Mg-6Al-1Zn+1.5wt.% Ce alloy having the high corrosion potential and less corrosion current density implies that 1.5wt.% Ce added Mg-6Al-1Zn alloy shows better corrosion resistance compared to other alloys. This is due to the refinement of β phase at the grain boundaries.

4. Conclusions

The addition of cerium refines the β phase and form more continuous β phase on grain boundaries. The microhardness results show that the addition of cerium to Mg-6Al-1Zn alloy increases the hardness due to the refinement of β phase and Al4Ce strengthening phase. Immersion test results clearly indicating that the addition of 1.5wt.% Ce to Mg-6Al-1Zn magnesium alloy showing good corrosion resistance and least weight reduction in 3.5% NaCl environment when compared to other compositions of Mg-6Al-1Zn magnesium alloys due to more refinement of β phase. Potentiodynamic Polarization test results indicating that the addition of 1.5wt.% Ce has high Corrosion potential (Ecorr) value and less Corrosion current density (icorr) value which shows the good corrosion resistance compare to other compositions of Mg-6Al-1Zn Magnesium alloys.

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