



## EFFECTS OF GADOLINIUM ON MICROSTRUCTURE AND HARDNESS OF Mg-Zn-Ce-Zr CAST ALLOY

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### ABSTRACT

The effects of 3 wt.% heavy rare earth Gadolinium (Gd) addition on the microstructure and mechanical properties (Hardness) of as-cast Mg-Zn-Ce-Zr alloy (base alloy) were investigated using optical microscopy (OM), scanning electron microscopy (SEM), energy-dispersive spectrum (EDS), X-ray diffraction (XRD) and Vickers hardness. The purpose of this research is to investigate the variations occurred in grain size and the formation of intermetallics with Gd addition, as well as their effect on hardness. The results of microstructure indicated that Gd addition led to significant grain refinement, which decreased by 28%. Both grain refinement and hard secondary phases  $(\text{Mg,Zn})_{12}\text{RE}$  and  $(\text{Mg,Zn})_3\text{RE}$  led to the improvement in hardness of Mg-Zn-Ce-Zr by 34%.

**Keywords:** magnesium alloy, gadolinium, microstructure, hardness.

### INTRODUCTION

The most outstanding characteristic of magnesium is its density 1.74 g/cm<sup>3</sup>, which is the lowest of all the structural metals, therefore, its alloy are used where light weight is an important consideration. Vehicle light weighting for example is among the available strategies to improve the fuel economy and performance of vehicle and thus has attracted major automotive companies to increase the usage of magnesium alloys in their structure and powertrain components, particularly the engine block. In recent years, there have been renewed interest from aerospace, military industries and medical application in development of new magnesium alloys (Luo 2013). Moreover, magnesium alloys have better castability over other cast metals, such as aluminium and copper, and molten magnesium does not attack iron, thus magnesium alloys can be melted and held in crucibles and molds fabricated from ferrous materials (Pekguleryuz, Kainer *et al.*, 2013).

Although there is increasing interest in using magnesium alloys, the actual applications are still limited in comparison to their major competitor, aluminium alloys and steel and this is due to their lower mechanical properties especially at elevated temperature and poor corrosion resistance (Celikin, Kaya *et al.*, 2012). Rare earth elements, such as Ce, Gd and Nd have been found to generate favorable effects on microstructure and mechanical properties of magnesium alloys, especially on improving creep and corrosion resistance (Mordike 2002). The basic function of REs is formation of reinforcing intermetallic phase that remain stable at elevated temperature and moreover, REs with high solubility will form solid solution in  $\alpha$ -Mg matrix, which is suggested as the main strengthening mechanism in developing high performance magnesium alloys (Zhu, Gibson *et al.*, 2010). Among these systems, Mg-Zn-RE alloys have recently drawn widespread attention due to REs solution strengthening and their unique characteristic microstructure, because of significant effects of Zn content

with RE (Wu, Xu *et al.*, 2013, Yang, Guo *et al.*, 2013, Srinivasan, Huang *et al.*, 2014, Langelier and Esmaeili 2015). (Langelier and Esmaeili 2015) found that Ce additions reduced hardening of Mg-Zn alloy, due to large secondary phase, with Ce and Zn were present in the alloy, which reduced the amount of Zn solutes for precipitation, thereby reduced hardening. Among these effects of Zn/RE ratio, Mg-Gd-Zn is one of the very important systems. The solid solubility of Gd found to be reduced when Zn content increased.

In the Mg-Gd-Zn system, Gd and Zn are prone to form compound of Mg-Zn-Gd and this is due to large value of electronegativity difference between Zn-Gd. In addition, Gd addition to Mg-Zn, Mg-Zn-Zr and Mg-Zn-REs (double RE-alloying) alloys possibly results in the formation of many different phases, such as I phase  $(\text{Mg}_3\text{GdZn}_6)$ , W phase  $(\text{Mg}_3\text{Zn}_2\text{Gd}_3)$ , long period stacking ordered (lamellar 14H-LPSO) phase and  $(\text{Mg,Zn})_3\text{Gd}$  phase, thus resulting in different microstructure morphologies and mechanical properties (Friedrich and Mordike 2006, Huang, Qi *et al.*, 2013, Srinivasan, Huang *et al.* 2014). (Srinivasan, Huang *et al.* 2014), reported that Mg-2Gd-xZn alloys mainly consisted of I phase or  $(\text{Mg,Zn})_3\text{Gd}$ , depending on Zn content, which only  $(\text{Mg,Zn})_3\text{Gd}$  phase was seen in Mg-2Gd-2Zn, whereas both phases present in Mg-2Gd-6Zn.

Recently, research has been focused on Mg-Zn-REs (double/multiple RE alloying additions) such as Mg-Gd-Y-Zn-Zr and Mg-Y-Nd-Gd-Zn-Zr alloys (Wu *et al.* 2013, Kang *et al.*, 2014). Double REs system have attracted much attention based on precipitation strengthening found in Mg-Y-Nd system, well known as WE series alloy, which the most successful Mg alloys developed to date (Kang *et al.* 2014). Fei *et al.*, (2015) reported that increasing the amount of Gd in Mg-Zn-Y alloys enhanced the nucleation site, promoted the nucleation and growth of LPSO phase, then reduced the volume fraction of W phase, contributed for refinement of  $\alpha$ -Mg dendrite. It was reported that  $(\text{Mg,Zn})_3(\text{Gd,Y})$  and



(Mg,Zn)<sub>12</sub>(Gd,Y) phases were found in Mg-Gd-Y-Zr alloys, when amount of Zn  $\geq$  0.5wt.% (Wu, Xu *et al.* 2013). In addition, Gd elements found to increase inside the Mg matrix when amount of Gd addition increased and then discovered that Gd solid solution strengthening was the main factor contributed to the increasing value of hardness and strength. However, the alloys investigated were without Zn (Yang *et al.*, 2013).

The grain boundaries of ZRE1 alloy is decorated with a discontinuous network of (Mg, Zn)<sub>12</sub>Ce due to low solubility of Ce in magnesium and small precipitates zirconium & zinc inside the grain which is suggested that the main strengthening mechanism is contributed to the grain boundary reinforcement with intermetallic phase (Rzychoń *et al.*, 2012). Compared to Ce, Gd has relatively high solubility in Mg (23.5%) and low solid diffusivity (larger atomic size than Mg) has promising potential to strengthening the  $\alpha$ -Mg matrix (Friedrich and Mordike 2006).

The combination of low and high RE in Mg has not been investigated in detail. Therefore the aim of this research is to investigate the effects of high solid solubility rare earth (3 wt.% Gd) on microstructure and hardness of Electron ZRE1 (Mg-Ce-Zn-Zr) alloy.

## EXPERIMENTAL DETAILS

Electron ZRE1 (Mg-Ce-Zn-Zr) magnesium cast alloy was used as a base alloy. Table-1 shows the chemical composition of the as-cast ZRE1 magnesium alloy. The alloys were prepared in an electrical resistance furnace, under a protective gas mixture of Ar + SF<sub>6</sub>. Base alloy was melted in H13 steel crucible and 3 wt% Gd was added to melt at 730 °C. After the addition, the melt was stirred for a few minutes for complete dissolution of Gd and to achieve uniform composition in melt, before pouring into a H13 steel permanent mould.

**Table-1.** Chemical composition of experimental alloys in wt.%.

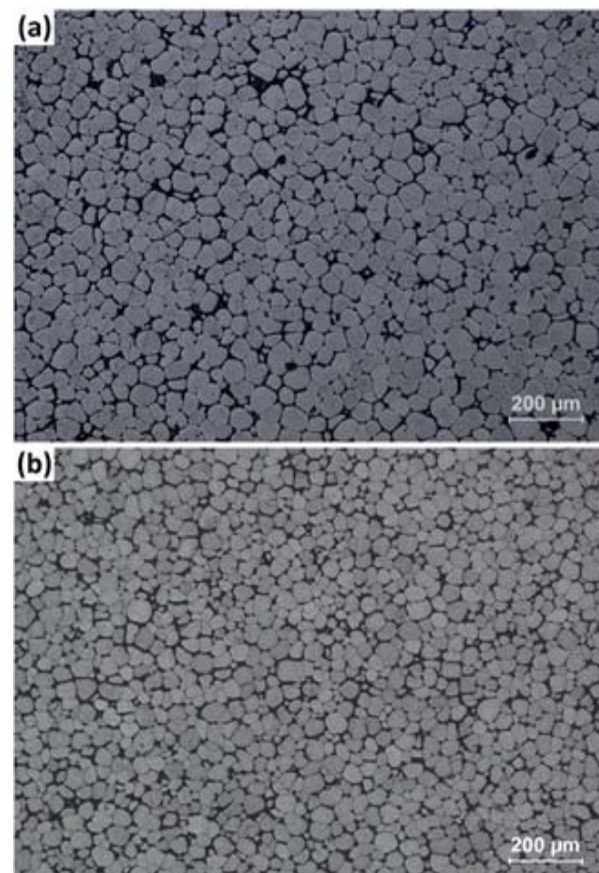
Alloy	Zn	Ce	Zr	Gd	Mg
0 wt.% Gd	2.5	3.1	0.6	-	Bal.
3 wt.% Gd	2.5	3.1	0.6	3.0	Bal.

The samples taken from the central section of ingots were ground and polished using standard methods. After polishing, the specimen for microstructural studies were etched with a reagent containing of 3 ml HNO<sub>3</sub> and 97 ml H<sub>2</sub>O and then rinse with water and alcohol. The microstructure was characterized by optical microscopy and scanning electron microscopy SEM equipped with an EDS detector. IMT iSolution DT V12.0 image analyser was used to examine the microstructure; grain sizes were measured using the linear intercept method based on ASTM standard E112. The hardness test was carried out by (Matsuzawa DVK-2) Vickers hardness tester with a 5kg load and holding time of 10s.

## RESULTS AND DISCUSSION

### Microstructure

Optical micrograph of as-cast Mg-Zn-Ce-Zr (ZRE1) base alloy and the addition of 3Gd wt.% alloy at high magnification are shown in Figure-1a and Figure-1b respectively. Microstructure of both alloys consists of equiaxed  $\alpha$ -Mg matrix and second phase crystallized along the grain boundaries. It can be seen obviously that microstructure of alloy with Gd addition have smaller grain size. Average grain size of base alloy and modified alloy were 63 $\mu$ m and 45 $\mu$ m respectively. As reported, the Y containing alloy had finer grain size comparing with Mg-Zn-Zr (Luo *et al.*, 1995). Eutectic secondary phase was found distributed at grain boundaries of base alloy with a discontinuous network shown in Figure-2a of SEM observation. The presence of massive phase in this alloy is due to low solubility of Ce in Mg. The volume fraction of second phase was observed to slightly increase in alloy with Gd as seen in Figure-2b.



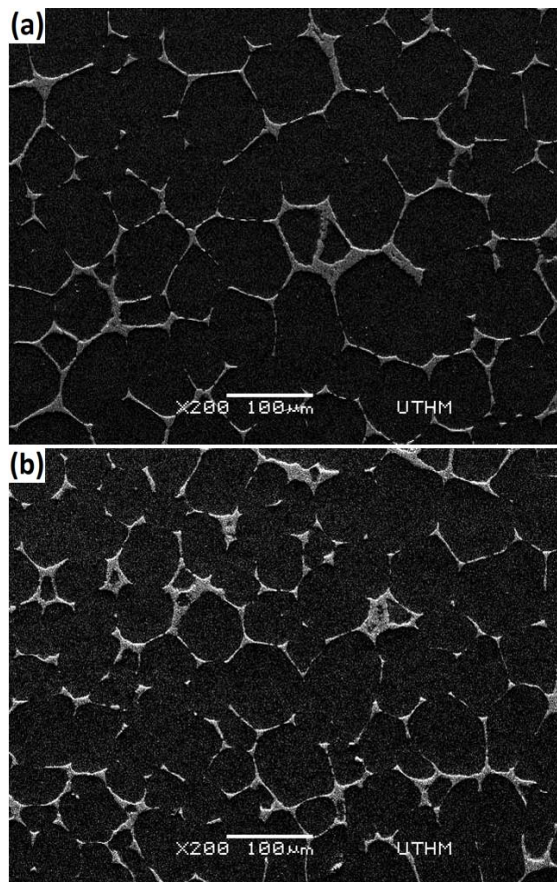
**Figure-1.** Microstructures of as cast (etched): (a) base alloy and (b) 3 wt.% Gd containing alloy.

EDS analysis of phase in base alloy (Figure-3a) shows that the second phases are highly enrich in Ce and also the presence of Zn. The matrix found without Ce and contains small Zn and Zr concentrations, as similar was





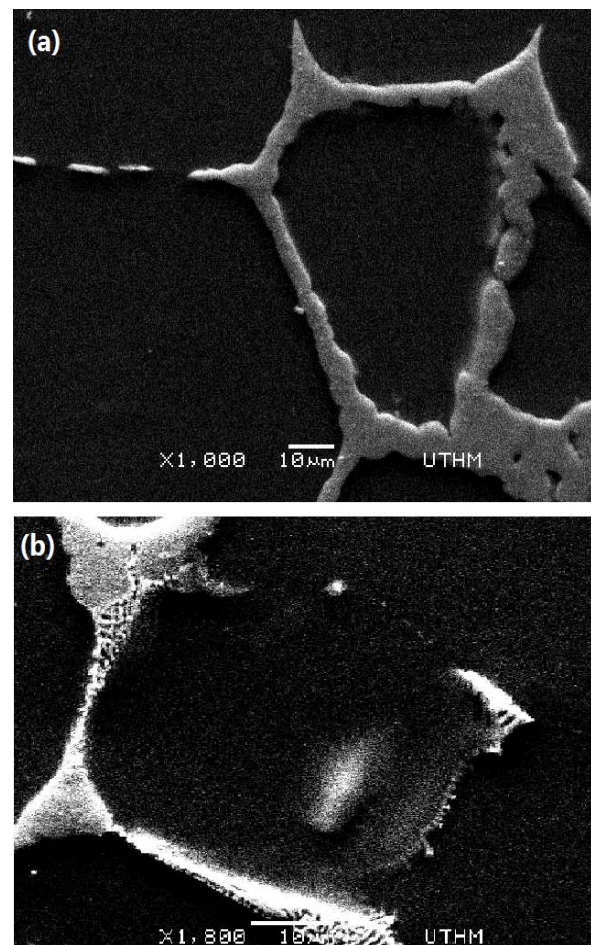
observed by (Rzychoń *et al.* 2012). The low Zn solutioned in  $\alpha$ -Mg matrix suggested that high residual Zn element formed compound with Ce in Mg-Zn-Ce phase, which concentrated in grain boundaries, thus suggested that the phase as the main strengthening mechanism in ZRE1 alloy (Rzychoń *et al.* 2012). In this study, Zr does not form any phases with any elements, contributes to grain refinement, which is in agreement with past studies in Mg-RE-Zn-Zr alloys (Luo *et al.* 1995, StJohn *et al.*, 2003, Rzychoń *et al.* 2012).



**Figure-2.** SEM micrograph of as cast: (a) base alloy and (b) 3Gd wt.% containing alloy.

Figure-3b and Table-2 show the high magnification of SEM micrograph and EDS element analysis of base alloy and base alloy with 3 wt.% Gd addition respectively. It can be seen that the as cast microstructure exhibited three phases, namely: The  $\alpha$ -Mg matrix which was supersaturated with Gd, Zn and Zr in solid solution, the bright  $(\text{Mg,Zn})_{12}\text{RE}$  phase and the network like phase merged with  $(\text{Mg,Zn})_{12}\text{RE}$  phase. The analysis results of XRD patterns for investigated alloys are shown in Figure-4a and 4b. It can be observed that typical  $\alpha$ -Mg peaks (Ref. code: 98-007-7908) are present in both base alloy and base 3 wt.% Gd alloys. The diffraction pattern of peaks which correspond to the  $\text{Mg}_{12}\text{Ce}$  phase (Ref. code: 98-062-1495) with slight deviation in  $2\theta$  from

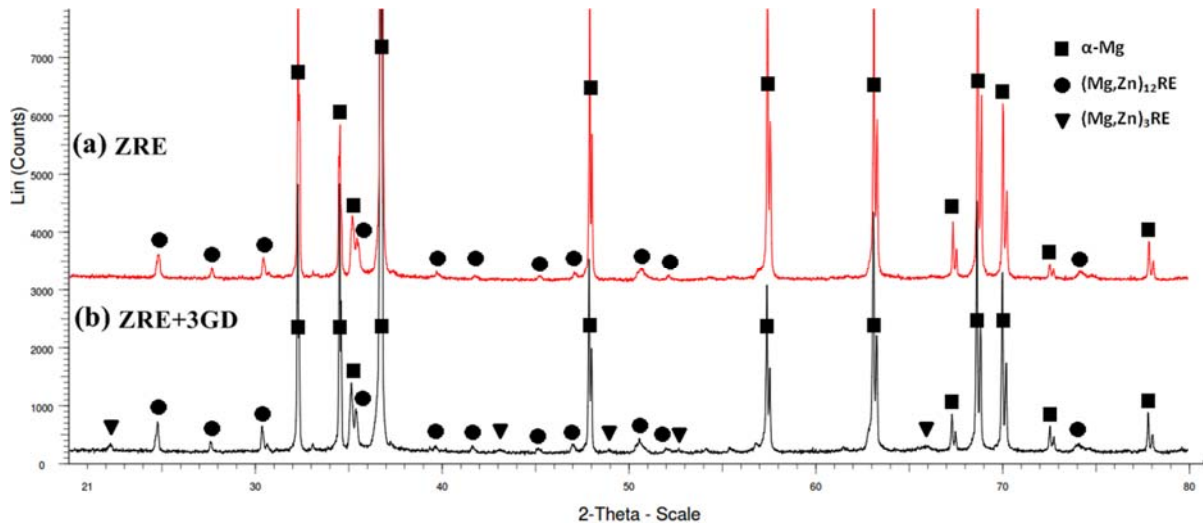
standard peaks also found in both alloys, due to the dissolution of Zn, which substitutes part of Mg due to small different between atomic radii of these elements. Therefore this phase could be identified as  $(\text{Mg,Zn})_{12}\text{RE}$ . This result is consistent with the result demonstrated in Ref (Rzychoń *et al.* 2012). As stated above, the  $\text{Mg}_{12}\text{Ce}$  phase type also corresponds with the peaks in base alloy with Gd addition. In addition, Gd element is dissolved in the phase as shown by EDS element analysis in Table-2 and Figure-3b. This is consistent with the observed phase, which was brightened due to the presence of Gd in the phase. Therefore this phase could be identified as  $(\text{Mg,Zn})_{12}(\text{Ce,Gd})$  or  $(\text{Mg,Zn})_{12}\text{RE}$  phase with Ce substituted by Gd without destroying the crystal structure, due to small different of atomic radius of Ce,  $r_{\text{Ce}} = 0.183$  and Gd,  $r_{\text{Gd}} = 0.178$ . Apart from  $\alpha$ -Mg and  $(\text{Mg,Zn})_{12}\text{RE}$  phase, an additional small peaks (Figure-4b) of  $\text{Mg}_3\text{Gd}$  type (Ref. code: 98-010-4068) are identified in the diffraction pattern of alloy with Gd addition, where small deviation in  $2\theta$  from the standard peaks, due to dissolution of high Zn and Ce elements. When the Zn concentration increases, the  $2\theta$  values shift to higher (Huang, Li *et al.*, 2009).



**Figure-3.** SEM micrograph of phases in as cast: (a) base alloy and (b) Base alloy with 3 wt.% Gd addition.

**Table-2.** Summary of EDS point analysis ( At.%) corresponding to intermetallics marked in Figure-3.

Symbol in Figure-3	Elements					Simplified composition
	Mg	Zn	Ce	Gd	Zr	
1	99.37	0.40	-	-	0.23	$\alpha$ -Mg with Zn & Zr (Mg,Zn) <sub>12</sub> RE
2	89.20	6.49	4.31	-	-	
3	99.05	0.25	-	0.09	0.61	$\alpha$ -Mg with Zn, Zr & Gd (Mg,Zn) <sub>12</sub> RE
4	90.49	5.12	3.90	0.49	-	
5	83.65	10.29	1.11	4.95	-	(Mg,Zn) <sub>3</sub> RE

**Figure-4.** XRD pattern of investigated alloys: (a) base alloy and (b) 3 wt.% Gd containing alloy.

This result is consistent with SEM micrograph at high magnification found in the presence of rich-Gd network like phase, merged with (Mg,Zn)<sub>12</sub>RE phase as shown in Figure-3b. EDS result of this phase in Figure-3b was summarized in Table-2. It can be seen that this phase contains highest Zn (10.29 at.%) and Gd (4.95 at.%) concentrations compared to other phases and contained more Zn than Gd. The result suggests that this new phase in Gd containing alloy is consumed high Zn & Gd elements, then reduce the amount of Zn & Gd concentration in  $\alpha$ -Mg matrix, which contributed to the reduction of the solid solubility of Gd in Mg. This similar behaviour was reported in Mg-Gd-Zn alloys (Srinivasan, Huang *et al.* 2014). Gd addition to Zn containing alloys possibly results in the formation of W phase (Mg<sub>3</sub>Gd<sub>2</sub>Zn<sub>3</sub>) and (Mg,Zn)<sub>3</sub>Gd. The Zn/Gd ratio for the W phase and (Mg,Zn)<sub>3</sub>Gd are 2 and 1.5, respectively. The morphologies of both phases found to be different, which is W phase presents the feather like shapes and (Mg,Zn)<sub>3</sub>Gd phase presents the network like or skeleton like shapes (Yang, Guo *et al.* 2013, Srinivasan, Huang *et al.* 2014). The new secondary phase in the base alloy with Gd addition was found to has Zn/RE(Gd and Ce) ratio of 1.69, which is close to Zn/Gd ratio 1.5 for (Mg,Zn)<sub>3</sub>Gd. In addition, the morphology of the phase is more similar to network like shapes. Based on the EDS result, analysis of

the corresponding XRD pattern and literatures, this phase could be suggested as (Mg,Zn)<sub>3</sub>(Gd,Ce) or (Mg,Zn)<sub>3</sub>RE phase. From EDS and XRD analysis, it can be concluded that the base alloy consisted of two phases, the  $\alpha$ -Mg and (Mg,Zn)<sub>12</sub>RE, while base alloy with 3 wt.% Gd addition consisted of three phases  $\alpha$ -Mg, (Mg,Zn)<sub>12</sub>RE contain Gd substitutes for Ce and new phase that was identified as (Mg,Zn)<sub>3</sub>RE.

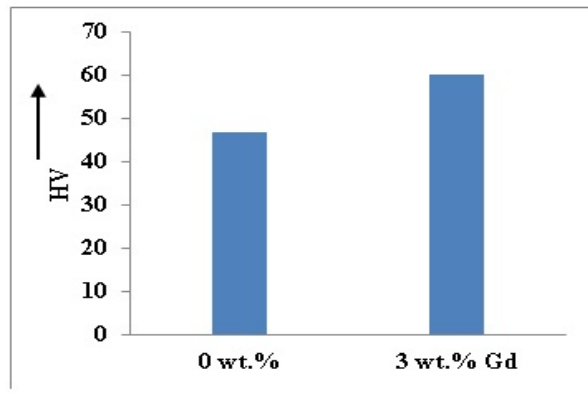
### Hardness

Vickers hardness (HV) testing indicated that HV of base alloy increased with the addition of Gd, from 47 HV to 60.3 HV as shown in Figure-5. The observed development of hardness in Gd-containing alloy, reflects three main hardening mechanisms: 1) grain refinement; 2) hardening by secondary eutectic phases, (Mg,Zn)<sub>12</sub>RE and (Mg,Zn)<sub>3</sub>RE; and 3) solid solution hardening by Gd and Zn dissolved in  $\alpha$ -Mg phase.

It is well known that the mechanical properties of as cast Mg alloys are greatly influenced by grain size, intermetallic phase and level of solute supersaturated in  $\alpha$ -Mg matrix (Celikin, Kaya *et al.* 2012, Ali, Qiu *et al.*, 2015). The grain refinement induces high concentration of incoherent grain boundaries and act as barriers to dislocation motion, which is inhibited a dislocation passing into the near grain and change its direction of



motion (Peng, Wang *et al.*, 2009). In addition, the grain boundaries are pinned with the thermally stable intermetallic phases, (Mg,Zn)12RE and (Mg,Zn)3RE inhibit the grain boundary sliding (Zhu, Gibson *et al.* 2010), therefore increase the hardness and strength of Gd-containing alloy.



**Figure-5.** Effect of Gd addition on hardness value of Mg-Zn-Ce-Zr alloy.

It is widely reported that Ce and La were more effective in refine the grain due to very low solid solubility limits and high melting temperature of the elements in Mg alloy in comparison with Gd and Y. During the solidification process, RE atoms are enriched at the solid-liquid interface, which will induce the expansion of a constitution supercooling area in front of the solid liquid interface, accelerating nucleation rate and restricting dendrite growth (Le, Zhang *et al.*, 2010, Golmakaniyoon and Mahmudi 2011). As stated earlier, Gd solid solubility was effectively reduced by Zn and the volume fraction of eutectic secondary phase of Gd-containing alloy increased. Therefore, Gd with the solid solubility restricted by Zn, and the high intention of Gd to form compound with Ce and Zn, resulting to the atoms concentrated at the solid liquid interface, resulting more effective undercooling area in front of the solid liquid interface, accelerating nucleation rate and restricting dendrite growth, reducing the grain size in Gd-containing alloy, hence strengthen the alloy.

Solid solution hardening by RE dissolved in  $\alpha$ -Mg matrix act as the main hardening contribution in binary Mg-Gd alloys and this is due to large Gd atoms inducing an internal elastic stress in the  $\alpha$ -Mg lattice. Mg-5Gd (wt.%) alloy contains even more than 20 wt.% of Gd in  $\alpha$ -Mg has been reported with hardness of 47HV (Kubásek and Vojtěch 2013). However in the presence of Zn, Gd solubility in Mg was found decreasing. In the present investigation, average concentrations of Gd and Zn in  $\alpha$ -Mg matrix are 0.60 wt% and 0.80 wt.% respectively. Relatively similar result was found in (Srinivasan, Huang *et al.* 2014) which was indicated approximately 1.0 wt.% and 1.90 wt.% of Gd and Zn concentrations, respectively in the Mg matrix for Mg-2Gd-2Zn alloy. Comparing to

Zn, the effect of solid solution strengthening by Gd is much higher due to the bigger atomic size of Gd and/or positive shear modulus misfits between Gd and Mg (Gao, Chen *et al.*, 2009). (Nie, Oh-Ishi *et al.*, 2008) conducted a detailed investigation on solute segregation suggested the co-segregation of Gd and Zn atoms in  $\alpha$ -Mg possibly provide a stronger pinning effect on the gliding dislocation than expected for single solute atoms. Therefore, it is suggested that the co-segregation of solid solution Gd and Zn has a contribution to the improvement of hardness of base alloy with 3 wt.% Gd addition.

## CONCLUSIONS

The effect of the addition of 3 wt.% Gd addition on microstructure and hardness of Mg-Zn-Ce-Zr (ZRE1) cast alloy was investigated. From an analysis of the results, the following can be summarized:

- Microstructure of Mg-Zn-Ce-Zr alloy consisted of two phases, the  $\alpha$ -Mg and (Mg,Zn)12RE, while 3 wt.% Gd containing alloy consisted of three phases  $\alpha$ -Mg, (Mg,Zn)12RE contain Gd substitutes for Ce, and (Mg,Zn)3RE.
- Gd addition to the Mg-Zn-Ce-Zr alloy clearly refined the grain size, which reduced the size of base alloy around 28%. In addition, Gd increased the volume fraction of eutectic secondary phase.
- Both grain refinement and hard secondary phases (Mg,Zn)12RE and (Mg,Zn)3RE are the main hardening mechanisms in development of the hardness, while the hardness of Mg-Zn-Ce-Zr cast alloy increased by 34%.

## ACKNOWLEDGEMENTS

This research is funded by Exploratory Research Grant Scheme (ERGS), vot number E023, Ministry of Higher Education, Malaysia.

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