



Effects of Ce additions on the age hardening response of Mg–Zn alloys



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ABSTRACT

The effects of Ce additions on the precipitation hardening behaviour of Mg–Zn are examined for a series of alloys, with Ce additions at both alloying and microalloying levels. The alloys are artificially aged, and studied using hardness measurement and X-ray diffraction, as well as optical and transmission electron microscopy. It is found that the age-hardening effect is driven by the formation of fine precipitates, the number density of which is related to the Zn content of the alloy. Conversely, the Ce content is found to slightly reduce hardening. When the alloy content of Ce is high, large secondary phase particles containing both Ce and Zn are present, and remain stable during solutionizing. These particles effectively reduce the amount of Zn available as solute for precipitation, and thereby reduce hardening. Combining hardness results with thermodynamic analysis of alloy solute levels also suggests that Ce can have a negative effect on hardening when present as solutes at the onset of ageing. This effect is confirmed by designing a pre-ageing heat treatment to preferentially remove Ce solutes, which is found to restore the hardening capability of an Mg–Zn–Ce alloy to the level of the Ce-free alloy.

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1. Introduction

Many recent research efforts on magnesium alloys have focused on improving the generally poor mechanical properties of magnesium, specifically alloy ductility/formability and strength. A promising method of alloy design, noted to improve the ductility of magnesium alloys, is the addition of rare earth (RE) elements. RE element additions to magnesium primarily result in an increase in ductility by promoting a random alloy texture following bulk deformation, instead of the strong textures typical of common Mg alloys [1–6]. While the specific origins of this effect remain the subject of continued study, reports have proposed that RE elements can modify texture by promoting non-basal slip during deformation [7–9], retarding dynamic recovery and recrystallization processes [10–12], or promoting growth of non-basal oriented grains [12,13]. It has been observed that this effect on texture also occurs for many different RE element additions, such as Gd [2], La [2], Nd [3,14], Y [2,3,7,9], and Ce [2–4]. Ce additions, in particular, have been noted to modify texture and enhance ductility when added at lower levels than most other RE elements [2,3]. This texture modifying potency for Ce makes it a desirable choice among RE elements for alloying with magnesium, as less overall RE may be used to produce the desired texture. Therefore, the cost associated with adding RE elements to modify texture can also potentially be minimized by using Ce.

Although texture improvement is a pre-requisite to achieve high ductility wrought alloys, thin gauge structural applications of magnesium will further require significant improvement in final material

strength. It is well known that precipitation hardening can be the most effective mechanism for achieving high strength products in metallic systems that do not undergo allotropic transformations [15]. Among magnesium alloys that have recently been the focus of extensive metallurgical analysis, Mg–Zn alloys have shown the potential to develop relatively high strength through precipitation hardening [16–22], as well as improved texture/ductility through Ce additions [13,23–27].

In a typical precipitation process for Mg–Zn alloys, two metastable precipitates, commonly denoted as β_1' and β_2' form during the decomposition of the supersaturated solid solution [18,20,28]. The β_1' precipitates commonly appear as rods parallel to the [0001] direction, and β_2' as thick plates along (0001) basal planes. However, β_1' may also form as blocky, cube-shaped precipitates, and β_2' as laths [18]. Reports from literature have identified the structure of these phases as the hexagonal MgZn₂ laves-C14 phase [28–32]. On the other hand, Gao and Nie [18] have reported the β_1' phase to be monoclinic Mg₄Zn₇, while more recently Rosalie et al. [33] have shown that within β_1' precipitates, both the MgZn₂ and Mg₄Zn₇ phases may be present. It is β_1' that is of most particular interest for the purposes of strengthening, as the orientation of the β_1' rods as perpendicular to the (0001) basal planes is suggested to be very efficient for strengthening, by impeding dislocations involved in both basal and prismatic $\langle a \rangle$ slip [21,34–36].

Despite established understanding on the precipitation phenomena in binary Mg–Zn alloys, the effects of Ce addition, as a potent texture modifier, on the precipitation hardening response of Mg–Zn alloys are yet to be fully examined. The objective of the current work is to fill this gap of knowledge and provide a basic understanding of the effects of Ce, in both alloying and microalloying levels, on the precipitation hardening capacity of the commercially promising Mg–Zn system. The

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precipitation hardening behaviour is investigated through microhardness measurement, transmission and scanning transmission electron microscopy (TEM and STEM), and thermodynamic analysis, which relate alloy chemistry to the phase transformation and hardening behaviour.

2. Experimental methodology

The series of Mg–Zn–(Ce) alloys examined in this work with their compositions are listed in Table 1. Binary Mg–Zn alloys, and alloys with 0.1 wt.% Ce additions, were prepared by gravity casting under an Ar atmosphere from pure Mg and Zn, as well as Mg–Ce master alloys. The two alloys with 1 wt.% Ce were supplied by CanmetMATERIALS in the as-cast condition. All cast ingots were homogenized for 3 h at 300 °C to eliminate possible regions of eutectic mixture, followed by 24 h at 400 °C to promote further microstructural uniformity. Samples of the homogenized ingots were then packed in MgO powder and solutionized for 6 h, water-quenched, and subsequently aged. The solutionizing temperatures were 520 °C for Z2 and ZE20, 420 °C for Z4 and ZE40, and 490 °C for ZE21 and ZE41 alloys. These temperatures were selected in order to maximize the amount of dissolved solute in the alloys, while not surpassing the solidus temperature. The solute concentrations and solidus temperatures for the alloys were calculated using the FactSage 6.3 software and VLGM database [37]. Artificial ageing for all alloys was conducted at 100 °C for times ranging up to 240 h. This ageing temperature was selected so as to be below the relatively low solvus temperature for 2 wt.% Zn in the binary Mg–Zn system (calculated to be 150 °C). Additionally, selected samples of the ZE20 alloy were also given a pre-ageing heat treatment. Pre-ageing was performed following solutionizing by immediately transferring the samples to a furnace at 300 °C, holding for 3 h, then water-quenching. This pre-ageing heat treatment was followed by artificial ageing at 100 °C. Hardness testing was conducted using an MHT series LECO 200 Vickers microhardness tester and a 100 g load. For each sample, a minimum of seven indentations were measured, with the reported values being the measurement averages after culling the maximum and minimum measurements from the data. Error bars presented on the data points represent one standard deviation, assuming a normal distribution.

Samples for optical microscopy were mechanically ground and polished. Polished specimens were then swabbed with a solution of 1% HNO₃ in ethylene glycol for approximately 1 min, which served to reveal secondary phases in the microstructure [38]. X-ray diffraction (XRD) examination was conducted on similarly polished samples, but without etching, using a Bruker D8 Advance instrument with Cu K α radiation over the range of 10–55°, at a rate of 0.4°/min and a step size of 0.04°. Foils for TEM analysis were prepared by mechanical grinding of aged samples to a thickness of less than 0.075 mm and then electropolishing at –50 °C and 50–90 V in a solution of 5.3 g lithium chloride (LiCl), 11.16 g magnesium perchlorate (Mg(ClO₄)₂), 500 ml methanol, and 100 ml butyl cellosolve. Foils were further thinned and cleaned by ion milling (2.5–3.5 keV and 4–7°) using a Gatan precision ion polishing system (PIPS) at liquid nitrogen temperature. TEM analysis was performed using a Philips CM12 microscope operating at 120 kV and a JEOL 2010F microscope at 200 kV, both equipped with an energy dispersive X-ray spectroscopy (EDS) detector. An FEI Titan-II

microscope at 300 kV was also used for scanning transmission electron microscopy.

3. Results & discussion

3.1. The as-quenched microstructures

Examination of the alloy microstructures in the as-quenched condition (i.e. directly following solutionizing) by optical microscopy and TEM, suggests that the Z2 and Z4 alloys are fully solutionized. For the ZE20 alloy, solitary particles are observed very rarely by TEM, appearing in such fine scale (~1 μ m) and low numbers as to not be apparent in optical microscopy observations. For the ZE21, ZE40, and ZE41 alloys, significant fractions of coarse secondary phase particles are observed in the as-quenched microstructures (Fig. 1). The formation of these secondary phase particles likely occurs during casting, with the quantity that remains in the as-quenched condition for each alloy dictated by their thermodynamic stability at the solutionizing temperature for that alloy. The volume fraction of the secondary phase particles is observed to scale with Ce content. This trend is most evident when comparing Z4 (no

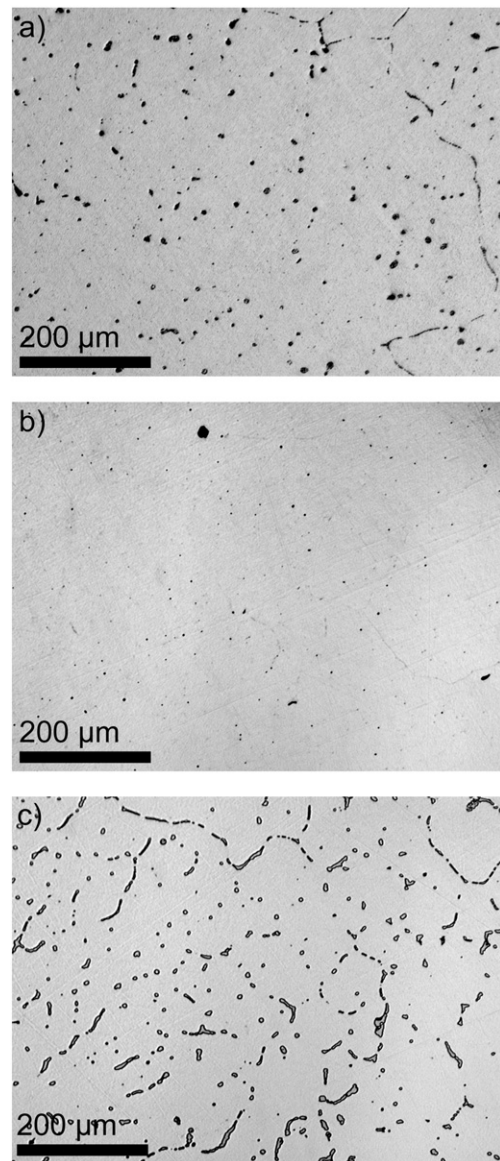


Fig. 1. Optical micrographs of a) ZE21, b) ZE40, and c) ZE41 alloys in the as-quenched condition, showing dark secondary phases.

Table 1
Mg–Zn–(Ce) alloy designations and compositions.

Alloy designation	Zn wt.% (at.%)	Ce wt.% (at.%)
Z2	1.6 (0.60)	–
Z4	4.0 (1.52)	–
ZE20	1.8 (0.68)	0.11 (0.019)
ZE40	4.0 (1.53)	0.10 (0.018)
ZE21	2.0 (0.76)	1.0 (0.177)
ZE41	4.0 (1.54)	1.0 (0.179)

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