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Trans. Nonferrous Met. Soc. China 23(2013) 66-72

Transactions of Nonferrous Metals Society of China

www.tnmsc.cn

Influence of Ce addition on microstructure and mechanical properties of high pressure die cast AM50 magnesium alloy

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Received 9 April 2012; accepted 29 June 2012

Abstract: The influence of Ce addition on the microstructure and mechanical properties of AM50 magnesium alloy was investigated to improve its mechanical properties. The results show that the addition of Ce to AM50 alloy results in the grain refinement and the mechanical properties of the Ce-modified AM50 at room and elevated temperatures are remarkably improved. AM50 magnesium alloy containing 1% Ce (mass fraction) shows better refinement and mechanical properties compared with the AM50 magnesium alloy with 0.5% Ce and even AM50 alloy without any Ce.

Key words: AM50 magnesium alloy; cerium; die casting; microstructure; mechanical properties

1 Introduction

Weight reduction is very attractive for current applications in the automotive industry due to the fuel efficiencies and lower emission levels. Magnesium and its alloys are the lightest in engineering materials. High pressure die casting (HPDC) of magnesium alloys is the most preferred manufacturing process for automotive as well as for numerous other applications [1]. The most widely used commercial magnesium alloys in HPDC are based essentially on the Mg-Al system, such as AZ91, AM50 and AM60 [2]. These alloys have already been applied to automobile components, such as seat frames, steering wheels, instrument panels, brackets and fans [3]. On the other hand, some commercial applications of magnesium alloys for important structural components are limited due to their poor creep properties [4]. Mechanical properties of magnesium alloys decrease dramatically at high temperatures above 120 °C [5,6]. To improve the mechanical properties, novel processing methods, such as double-control forming technology (DCF), were firstly introduced for the production of the complex Mg alloy components with enhanced mechanical properties [7]. JIANG et al [8] reported that,

compared with die casting, the ultimate tensile strength of AM60B magnesium alloy was increased by 63.9% and elongation was increased by 3.3 times in DCF.

Nevertheless, the demand of magnesium applications in automotive parts increases, so that new magnesium alloys having low cost, high strength and excellent elevated temperature properties need to be developed. Therefore, additional efforts were dedicated to develop new magnesium alloys mainly based on Mg-Al-RE (RE is rare earth), Mg-Al-Ca/Sr and Mg-Al-Si systems, which have improved the elevated temperature performance [9]. Mg-Al-RE system shows a significant improvement among these heat-resistant alloys due to the formation of thermal stable Al₁₁RE₃ precipitates and complete suppression of the Mg₁₇Al₁₂ phase [10]. Some Mg-Al-RE alloys, such as AE42 and AE44, enhance properties at elevated temperatures [11]. However, these alloys are rarely applied because of poor castability and higher cost [12]. Moreover, the deterioration of the creep behavior of AE42 alloy at high temperature was reported and the related mechanism is still in discussion [13]. Another work showed beneficial effects of little amount RE elements on the microstructure and mechanical properties of AZ91 and AM60 magnesium alloys [14].

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In AE42/AE44 alloy, the RE is mainly introduced by the addition of mischmetal, which is either a Ce or a Nd-rich mixture or RE. The typical composition (mass fraction) of Ce-rich mischmetal added to AE42 and AE44 is 52%-55% Ce, 23%-25% La, 16%-20% Nd, and 5%-6% Pr [15]. The combined effect of these RE elements determines the microstructure and properties of AE42 and AE44 alloys. Although the influences of La, Pr and Nd on the microstructure and mechanical properties of HPDC AM40 alloy were investigated [16-18], not much studies on effects of individual Ce element addition on the microstructure and mechanical properties of HPDC AM50 alloy were reported in literatures. Therefore, the influence of different amount of Ce on the phase formation, microstructure development and mechanical properties at different temperatures were investigated in order to contribute for the development of new Mg-RE alloys.

2 Experimental

0.5% Ce and 1.0% Ce were added to AM50 alloy (referred as AM50Ce0.5 and AM50Ce1). The alloys were cast into step-shape plate with four different step thicknesses (2, 6, 10, 14 mm) with a width of 20 mm. Specimens were taken only from the 14 mm- section for the present study (Fig. 1). The Frech DAK 450 cold chamber HPDC machine was used for casting experiments. The process parameters involved were the melt temperature of 680 °C, the casting pressure of 40 MPa, the die temperature of 200 °C and the shot sleeve speed of 5 m/s. Moreover, a mixture of N₂ and 2% SF₆ (volume fraction) was used as protective atmosphere. For each alloy, at least 30 shots were made. Samples with size of 20 mm×10 mm×10 mm were used for the microstructural observation and hardness tests. These samples were ground with 800, 1200, 2000 and 3000 grit silicon carbide emery papers. Immediately after grinding,

 38
 38
 38
 38
 38

 160
 160

Fig. 1 HPDC step plates with different thicknesses (Unit: mm)

the specimens were washed with water, rinsed with ethanol and dried. The final polishing was conducted with OPSTM suspension (Struers). The chemical compositions of the alloys were analyzed using a spark emission analyser, SpectrolabM from SPECTRO (Kleve, Germany).

The specimens were chemically etched using a combination of acetic acid (7 mL), picric acid (25 g), ethanol (140 mL), and purified water (40 mL) to reveal the microstructure. The microstructure was investigated using a Leica DMI 5000 optical microscope and a Zeiss Ultra55 scanning electron microscope (SEM) equipped with an energy dispersive X-ray analyzer (EDX). Grain sizes were determined using the line intercept method according to Ref. [19]. XRD measurements were performed on a Siemens D5000 machine using Cu $K_{\alpha 1}$ radiation (wavelength λ =0.15406 nm) at an angle of 2 θ $(15^{\circ}-75^{\circ})$ for each sample. The increment was 0.04° and the step time was 6.4 s. The hardness tests were carried out by Vickers hardness on an Emcotest M1C010 automated hardness test machine; the test load, the dwell time and the repetition were 5 kg, 10 s and 10 times, respectively. Tensile and compression tests were performed at room temperature and elevated temperatures (100, 120, 150 °C) using a Zwick 050 testing machine according to Refs. [20] and [21]. Specimens with a gauge length of 30 mm, a diameter of 6 mm and threaded heads were used in tensile tests (Fig. 2(a)). The compression specimens were cylinders with a height of 16.5 mm and a diameter of 11 mm (Fig. 2(b)). Both tensile and compression tests were done at a strain rate of 1×10^{-3} s⁻¹. In order to obtain homogenous temperature, the samples were soaked at the test temperatures (except RT) for 10 min before test run. All tests were repeated at least three times.



Fig. 2 Sketches of tensile (a) and compression (b) test specimen (Unit: mm)

3 Results and discussion

3.1 Microstructures

The chemical composition of the investigated die casting magnesium alloys is given in Table 1.

Table 1 Chemical compositions of investigated alloys

A 11	W/%			
Alloy	Mg	Al	Mn	Ce
AM50	Bal.	4.78	0.43	-
AM50Ce0.5	Bal.	4.67	0.35	0.53
AM50Ce1	Bal.	4.49	0.32	1.02

According to the phase diagram of Mg–Al–Mn ternary magnesium alloy, constituents at room temperature are α -Mg, β -Mg₁₇Al₁₂ and Al₈Mn₅ intermetallics for Mg–Al–Mn alloy containing Mn with mass fraction lower than 1% [22]. For AM50 alloy, aluminum and manganese are two main alloying elements. The aluminum is partly in solid solution, and partly precipitates in the form of Mg₁₇Al₁₂ along grain boundaries as a continuous phase as well as part of lamellar structure. Manganese is usually added to suppress the hazardous influence of Fe on the corrosion resistance. Al₈Mn₅ can pick up Fe and remove it from the matrix [23].

The X-ray diffraction (XRD) patterns taken from the as-die cast investigated alloys are shown in Fig. 3. AM50 alloy mostly consists of Mg and Mg₁₇Al₁₂ phases. It can be seen in Fig. 3 that, two different phases are detected in Fig. 3(a), while three different phases are detected in Figs. 3(b) and (c) as a result of Ce addition. When Ce is added to AM50 alloy, the diffraction peaks of Mg₁₇Al₁₂ phase decrease and may be below the detection limit of XRD. The main secondary phase in Figs. 3(b) and (c) (with Ce) is γ -Al₁₁Ce₃ phase.



Fig. 3 XRD patterns of investigated alloys: (a) AM50; (b) AM50Ce0.5; (c) AM50Ce1

Figure 4 shows SEM morphologies of the investigated alloys. It can be clearly seen from Fig. 4(a) that α -Mg and intermetallic compound appear. The EDX study was carried out on three selected points as shown in Fig. 4(a). The results indicate that the P1, P2 and P3 refer α -Mg matrix, β -Mg₁₇Al₁₂ and Al–Mn intermetallic compounds, respectively. As seen in Fig. 4(b), when 0.5% Ce (mass fraction) is added to AM50 alloy, new γ -Al₁₁Ce₃ phase occurs and it appears as needle-shaped. As Ce amount increases from 0.5% to 1%, also the amount of y-Al₁₁Ce₃ phase obviously increases (Fig. 4(c)). Similar EDX study was done on three selected points as shown in Fig. 4(c). EDX results in combination with XRD findings show that three phases containing α -Mg, β -Mg₁₇Al₁₂ and γ -Al₁₁Ce₃ occur in AM50Ce1 alloy. Similar results can be seen in Fig. 4(b), but γ -Al₁₁Ce₃ phase is less compared with Fig. 4(c) because of the less Ce amount. As shown in Fig. 4, Ce addition decreases the fraction of β -Mg₁₇Al₁₂ phase and



Fig. 4 SEM images of investigated alloys: (a) AM50; (b) AM50Ce0.5; (c) AM50Ce1

facilitates to form γ -Al₁₁Ce₃. The β -Mg₁₇Al₁₂ phase in AM50 alloy distributes almost in bulk, but the distribution in AM50-Ce alloy is relatively dispersive.

The overall EDX results of AM50 alloy and with 0.5% Ce and 1% Ce are shown in Table 2. According to these results, it can be easily seen that the addition of Ce causes the formation of a new phase. In Table 2, P9 shows that new phase is most likely γ -Al₁₁Ce₃ according to its molar ratio of Al to Ce (n(Al)/n(Ce) = 11/3 = 3.67 for Al₁₁Ce₃ and 8.58/2.53=3.39 for P9). These results are in accordance with the results of ZHANG et al [24,25].

Table 2 EDX results of constituent phases of investigated alloys

Location	x/%			
in Fig. 4	Mg	Al	Mn	Ce
<i>P</i> 1	97.17	2.70	0.13	-
P2	61.85	26.89	0.09	-
<i>P</i> 3	_	54.41	36.59	_
<i>P</i> 4	_	41.67	-	13.52
<i>P</i> 5	96.84	3.22	-	_
<i>P</i> 6	53.46	34.13	0.15	_
<i>P</i> 7	72.18	27.41	_	_
P8	98.49	1.51	_	_
<i>P</i> 9	_	8.58	_	2.53

Figure 5 shows the optical microstructures of AM50 alloy with and without Ce addition. It can be seen that the microstructure of AM50 alloy mainly comprises of α -Mg and β -Mg₁₇Al₁₂ phases. Mg₁₇Al₁₂ phase appears in two forms: the first is massive, the second is like a lamellar structure. Also, it can be inferred that $Mg_{17}Al_{12}$ phase forms along grain boundaries as a network. When Ce is added to AM50 alloy, a new phase occurs. Since the solid solubility of Ce in Mg-matrix solid solution of AM50 alloy is low, this phase contains mostly Al and Ce [26]. The Al-Ce phase often appears between the Mg₁₇Al₁₂ phases, possibly providing an obstruction to the growth of Mg₁₇Al₁₂, breaking up its networked structure. Hence, Ce addition is proved to be beneficial to refining the grain size of AM50 alloy [27].

3.2 Mechanical properties

The relationship between the hardness, grain size and Ce addition is shown in Table 3. The hardness increases, and the grain size decreases significantly with increasing the amount of Ce.

Figure 6 shows the tensile properties of the investigated alloys tested at room and elevated temperatures. The alloys demonstrate the highest tensile yield strength (TYS) and ultimate tensile strength (UTS) at room temperature (RT). Typically, the TYS and UTS

-Mg17Al12 -Mg₁₇Al -Mg₁₇Al

Fig. 5 Optical microstructures of investigated alloys: (a) AM50; (b) AM50Ce0.5; (c) AM50Ce1

Table 3	Hardness	and grain	1 size of	investigated	l alloys	

Alloy	Hardness, HV	Grain size/µm
AM50	38±6	40±11
AM50Ce0.5	40±9	31±20
AM50Ce1	45±7	26±16

decrease while the elongation increases with the increase of temperature. It can be seen that the addition of Ce can remarkably improve the tensile properties of AM50 alloys at different temperatures.

From Fig. 6(a), it is obvious that AM50 alloy shows the lowest TYS, while with the addition of Ce, the TYS of the alloy is remarkably improved both at room and elevated temperatures. The TYS of AM50 alloy is 87 MPa at RT and 69 MPa at 150 °C, whereas for AM50Ce1 alloy the TYS is 98 MPa at RT and 80 MPa at 150 °C. The TYS increases by 12% at RT and 15% at



Fig. 6 Tensile properties of investigated alloys at different temperatures: (a) TYS; (b) UTS; (c) Elongation

150 °C with the addition of 1% Ce. The UTS has the same trend as TYS with the change of Ce content (Fig. 6(b)). The UTS of AM50 alloy is 145 MPa at RT and 120 MPa at 150 °C, while for AM50Ce1 alloy the UTS is 168 MPa at RT and 137 MPa at 150 °C. Thus, the UTS increases by 14% at RT and 16% at 150 °C with the addition of 1% Ce. The elongation of the alloys is also improved as the result of Ce addition. At all temperatures the highest value is acquired from AMCe1 alloy (Fig. 6(c)). For example, the elongation of AM50 alloy is 2.8% at RT and 8.3% at 150 °C, whereas for AMCe1 alloy the elongation is 4.8% at RT and 10.3% at 150 °C.

The elongation of the investigated alloys increases with the increase of temperature and reaches the highest value at 150 °C. In the previous studies, the effect of RE on the microstructure and mechanical properties of AM series magnesium alloys was investigated. These studies indicated that four RE elements (La, Ce, Pr, Nd) can improve the mechanical properties of AM series magnesium alloys and the strengthening effect increases with increasing RE content [16–18,24,28]. The present results are in a good agreement with the previous studies.

Figure 7 shows the compressive properties of the investigated alloys tested at room and elevated temperatures. The alloys demonstrate the highest



Fig. 7 Compressive properties of investigated alloys at different temperatures: (a) CYS; (b) UCS; (c) Compression

compressive yield strength (CYS) and ultimate compressive strength (UCS) at room temperature (RT). Typically, the CYS and UCS decrease, while the deformation in compression increases with the increase of the temperature. It can be seen that the addition of Ce can remarkably improve the compressive properties of AM50 alloy at different temperatures.

CYS and UCS have the same trends both at room and elevated temperature as TYS and UTS at different temperatures. From Fig. 7(a), it is obvious that AM50 alloy exhibits the lowest CYS, while with the addition of Ce, the CYS is remarkably improved both at room and elevated temperatures. The CYS of AM50 alloy is 104 MPa at RT and 88 MPa at 150 °C, whereas for AM50Ce1 alloy the CYS is 116 MPa at RT and 98 MPa at 150 °C. Therefore, the CYS is raised by 11% at RT and 12% at 150 °C with the addition of 1% Ce. Similarly, the UCS of the alloys also changes as CYS after the addition of Ce (Fig. 7(b)). The UCS of AM50 alloy is 296 MPa at RT and 270 MPa at 150 °C, while for AM50Ce1 alloy the UCS is 315 MPa at RT and 284 MPa at 150 °C. Hence, the UCS increases by 7% at RT and 5% at 150 °C with the addition of 1% Ce. The compression of the alloys is also developed as the result of Ce addition and at all temperatures, the highest value is obtained from AMCe1 alloy (Fig. 7(c)). For example, the compression of AM50 alloy is 12.6% at RT and 18.8% at 150 °C, whereas for AMCe1 alloy the compression is 15.0% at RT and 20.6% at 150 °C. The deformation in compression of the investigated alloys increases with the increase of the temperature and reaches the highest value at 150 °C.

With the addition of Ce, the grains of α -Mg matrix are refined and eutectic phase β -Mg₁₇Al₁₂ is suppressed and substituted by Al₁₁Ce₃ with the former being the dominant one. Al₁₁Ce₃ particles can refine the grain size significantly when Ce is added to AM50 alloy. Small grain size can effectively prevent the dislocation slip, so that the deformation of fine grain structural alloys needs higher stress. The main strengthening phase in AM50 alloy is β -Mg₁₇Al₁₂, which has a low melting point (with eutectic temperature of 437 °C) and a poor thermal stability. β -Mg₁₇Al₁₂ phase can readily soften and coarsen at the temperature exceeding 125 °C [29]. The addition of Ce to AM50 alloy results in the suppression of β -Mg₁₇Al₁₂ phase and the formation of Al₁₁Ce₃ phase. Al₁₁Ce₃ phase has a high melting point (>1200 °C), and then the softening temperature of the alloy containing $Al_{11}Ce_3$ phase can be increased. With the increase of Ce content in the alloys, the amount of Al₁₁Ce₃ particles increases significantly, and then the strengthening effect is also improved. Thermally stable Al₁₁Ce₃ phase forms with the addition of Ce, which is located at grain boundaries and suppresses the slippage of the grain boundary at elevated temperature, so that elevated temperature properties of the alloys are improved.

4 Conclusions

1) The addition of Ce can reduce the grain size of AM50 alloy.

2) The addition of Ce can remarkably improve the tensile and compressive properties of AM50 alloys at room and elevated temperatures. This is due to Al–Ce phases and the grain refinement.

3) The relationship between microstructure and mechanical properties was discussed. However, microstructure observations are not yet completed. Hence, further XRD and TEM measurement are planned for the future to identify all phases.

Acknowledgements

The authors want to express their gratitude to G. WIESE, J. BOHLEN, G. MEISTER, W. PUNESSEN and S. SCHUBERT for their technical support. Financial support from Turkey Council of Higher Education (YÖK) Scholarship for Faruk's PhD Study in Helmholtz-Zentrum Geesthacht HZG is also appreciated.

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添加铈对高压压铸 AM50 镁合金 显微组织和力学性能的影响

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摘 要:研究添加铈对高压压铸 AM50 镁合金显微组织和力学性能的影响。结果表明:添加铈能使 AM50 镁合金 晶粒细化,并使其室温和高温力学性能得到显著改善。相对于未添加 Ce 和添加 0.5%Ce(质量分数)的 AM50 镁合金,添加 1%Ce(质量分数)的镁合金的晶粒更细,力学性能更优。 关键词: AM50 镁合金; Ce; 压铸;显微组织;力学性能

(Edited by Wei-ping CHEN)

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