Materials Research Express

PAPER

An investigation on the precipitates in T5 treated high vacuum die-casting AE44–2 magnesium alloy

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Keywords: HVDC-AE44-2 alloy, aging, precipitation, microstructure, mechanical properties, dislocations

Abstract

The AE44-2 magnesium alloy was fabricated via die-casting under high vacuum (denoted as HVDC-AE44-2 alloy). The microstructures and mechanical properties of the aged HVDC-AE44-2 alloys were investigated. It was found that age strengthening can be achieved in the HVDC-AE44-2 alloys. The hardness, the yield strengths (YS) and the ultimate tensile strengths (UTS) of the HVDC-AE44-2 alloys after aging at 200 °C for 12 h (T5) increased by 8.8 HV, 30.5 MPa and 22.6 MPa, respectively, while the elongations remained almost at the same level (The elongation was 19.6% in the die casting. The elongation was 19.1% in aged at 200 °C for 12h). It was also found that the improved age hardening ability was closely related to the Al4Mn and Al11Mn4 precipitation during aging. Both the α-Mg and the Al11RE3 have crystallographic orientation relationships with the Al4Mn. The slipping of dislocations was impeded by the precipitates during tensile deformation at room temperature, and the dislocations were accumulated around the precipitates, which is the reason for the increase of the HVDC-AE44-2 alloy strength.

1. Introduction

Magnesium alloys have potential applications in aerospace, transportation, and other fields due to their low density, good electromagnetic shielding and machining ability [1, 2], which is beneficial for reducing weight, energy saving and emission reduction [3–6]. The application of magnesium alloys in different situations requires materials to exhibit different properties, such as creep resistance, high temperature mechanical properties and corrosion resistance [7]. However, the applications of the most magnesium alloys are limited as the structural components due to their low strengths [8]. The properties of magnesium alloys are closely related with the casting method, the casting method determines the microstructure, and the microstructure determines the properties of the magnesium alloy. The casting methods of magnesium alloys usually include gravity casting, high pressure die casting, squeeze casting, semi-solid metal casting, and other casting processes [9]. Compared with other die-cast magnesium alloys, Mg-4Al-4RE Mg alloys have attracted attention owing to its applications at elevated temperatures [6]. AE42 and AE44 are two main Mg-Al-RE alloys. The magnesium alloys flow easier when 4%Al is added in alloy and can be formed to the die casting parts with complex shapes. The addition of rare earth elements (RE) can improve the performance of the alloy at elevated temperatures and refine the grains significantly during the casting process. In addition, adding 0.3% of manganese (Mn) can remove iron from the magnesium alloy and improve the corrosion resistance of the alloy. The creep resistance of the AE42 degrades rapidly at temperatures above 150 °C, adding more rare earth elements (RE) to AE42 improves casting performance, which results in the development of AE44 [10, 11]. Recently the die-cast AE44 alloys added with La + Ce misch metal were investigated as a result of its cheap cost [10, 11]. Therefore, a cheap and excellent die-casting magnesium alloy has attracted a lot of attention.
2. Experimental procedures

The Mg alloys were strengthened obviously by both the precipitation hardening and the dispersion strengthening after aging [12–14]. In die-cast Mg-Al-RE alloys, the results of Nie et al. showed that the mechanical properties could be enhanced considering the dynamic nanoscale Al-Mn precipitates during aging at 200 °C. The hardness of the Mg-6Zn-4Sn-1Mn alloy reached a peak of 74 HV during the aging at 180 °C for 12 h [15]. Wang et al. found that the artificial aging with low temperature and short time can remarkably improve the mechanical properties of the high pressure die casting Al-Si-Cu-Mg-Zn alloy, and as the aging temperature increases from 160 °C to 220 °C, the θ’ nanoscale particles was sufficiently precipitated and improved the strength of the alloys significantly. The results by Su et al. showed that the β phase was precipitated during the aging, and the strength of the Mg-Gd(-Y)-Zn-Mn alloys were significantly improved by ~100 MPa. The Al8Mn5 particles were precipitated within α-Mg grains after a heat treatment (at 350 °C for 16 h) in the die-cast Mg-4Al-4RE-0.3Mn [18]. In our previous work, the AE44-2 alloys are fabricated via gravity casting, high pressure die casting without high vacuum and high pressure die casting with high vacuum, respectively. The microstructures and mechanical properties of the AE44-2 alloys by the three different fabrication routes were compared. The results showed that the properties of the alloys fabricated by high pressure die casting with high vacuum are better than those of the Mg alloys fabricated by the gravity casting route and the high pressure die casting without high vacuum route. In addition, it was found that the high pressure die-cast alloys without high vacuum had many trapped gas pores [19]. In the current work, high vacuum die-cast route was developed to fabricate the HVDC-AE44-2 alloy, and the HVDC-AE44-2 alloys had fewer micro trapped gas pores than those of the high pressure die casting without high vacuum, which avoided cracks nucleation and propagation during both the aging and the deformation. The properties of the aged HVDC-AE44-2 alloys were improved and thus the strengthening mechanisms of the Al4Mn and Al11Mn4 were discussed.

A lot of researches have been done on the aging of die casting magnesium alloys. However, most of the researches focus on high pressure die casting Mg-Al-RE alloys without high vacuum, and the Mg-Al-RE alloys of the high pressure die-cast without high vacuum are not heat treatable because the trapped gas pores can expand during the aging treatment, which leads to the difficulty of the further improving the properties of the alloy. In addition, there are few studies on the Mg-Al-RE alloys fabricated via high pressure die casting with high vacuum route, and the precipitates of those alloys were not characterized in detail and the crystallographic orientation relations between the phases in those the alloys were not clear after aging at 200 °C. In the previous studies [10, 11, 18], which resulted in the strengthening mechanisms remaining unclear. In this work, after aging, the precipitates Al4Mn and Al11Mn4 that increase the strength of the HVDC-AE44-2 alloys were identified and the crystallographic orientation relationships between the phases were investigated using high resolution transmission electron microscope (HRTEM). Furthermore, the strengthening mechanisms of the as-aged HVDC-AE44-2 alloys are analyzed.

2. AE44-2 (RE = La + Ce misch metal) Mg alloy purchased from Magontec Xi’an Co. Ltd was used as the raw material. The mixed gas of SF6 and N2 was used for protection during AE44-2 alloy melting, and the DM300 (a precision horizontal cold chamber die casting machine) was used to fabricate die-cast AE44-2 alloy under high vacuum. A melting temperature of 680 °C was pre-set. The die was equipped with an oil heating system and the temperature of the oil heating element was set to 280 °C. The mold temperature of the HVDC-AE44-2 alloys was 190 °C. The HVDC-AE44-2 alloys was aged at 200 °C (0 h, 4 h, 8 h, 12 h and 16 h) and 250 °C (0 h, 4 h, 8 h, 12 h and 16 h), respectively. The rod-shaped specimens of the HVDC-AE44-2 alloys were used in the tensile tests.

The chemical composition of the alloys were analyzed by inductively coupled plasma emission spectrometry (ICP-OES), and the results are shown in table 1 [19]. The hardness tests were carried out with a Wolpert-Wilson Vickers Hardness tester. The phases of the die-cast sample were examined by a Bruker XD8 ADVANCE A25 x-ray diffractometer (XRD) with a copper target Kα line at a scan rate of 0.02° s−1 and 2θ range from 20° to 90°. The microstructures of the HVDC-AE44-2 alloys were observed by a scanning electron microscope (ZEISS-6035) equipped with energy dispersive x-ray spectroscopy. The phases and the crystallographic orientation relations between the phases were indexed by a high resolution transmission electron microscope (JEM-2100F). The electron backscatter diffraction data were obtained and analyzed by the Zeiss ZEISS-6035 equipped with

| Table 1. Chemical composition of die-cast AE44-2 alloys (in wt%). |
|-----------------|-----|----|---|----|---|
| Alloy           | Al  | Ce | La | Mn | Mg |
| HVDC-AE44-2     | 4.32| 2.17| 1.11| 0.23| Bal |

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TSLMSC (the probe equipped on EBSD, and the model is TSMLSC). The die-cast rod-shaped tensile samples were 60 mm in gauge length and 6.5 mm in diameter. The tensile tests were performed by a universal testing machine (Instron-5982) at room temperature under a tensile rate of 0.5 mm min$^{-1}$.

3. Results

3.1. The microstructure and the phase analysis of the alloy

Figure 1 shows the XRD patterns (JPCDS No.35-0821) of the as-die-cast HVDC-AE44-2 alloys, as-aged HVDC-AE44-2 alloys at 200 \degree C for 12 h and at 250 \degree C for 12 h. The $\alpha$-Mg, $\text{Al}_3\text{RE}$, $\text{Al}_{11}\text{RE}_3\text{Mn}_7$, and $\text{Al}_{19}\text{RE}_2\text{Mn}_7$ were found in all of them. Figure 2 is a SEM image of the as-die-cast HVDC-AE44-2 alloy. It can be seen that the lamellar and granular second phases exist in the as-die-cast HVDC-AE44 alloys and the lamellar phases are uniformly distributed in the $\alpha$-Mg. The grains are refined in the as-die-cast HVDC-AE44-2 alloys due to the high cooling rate [20], and rare earth elements and aluminum atoms preferentially segregated at the solid-liquid interface to form Al-RE compounds, thereby inhibiting the growth of crystal grains [21].
Figure 3 (a) is a BF image of the as-die-cast HVDC-AE44-2 alloys, in which Al$_{11}$RE$_3$ is identified by HRTEM image as shown inset. Figure 3 (b) is another BF image of the HVDC-AE44-2 alloy, in which Al$_2$RE and Al$_{10}$RE$_2$Mn$_7$ are identified with EDS analysis as shown in figures 3 (c) and 3 (d), respectively, and the rare earth elements are found to be La and Ce. The $\alpha$-Mg is surrounded by the lamellar Al$_{11}$RE$_3$ and the granular Al$_2$RE. It is consistent with the results in literatures [11, 21]. Figure 3 show that the Al$_{11}$RE$_3$, Al$_2$RE and Al$_{10}$RE$_2$Mn$_7$ are the second phases in the as-die-cast HVDC-AE44-2 alloys, which are consistent with the XRD results as above mentioned.

In order to investigate the phase’s transformations of the HVDC-AE44-2 alloys before and after aging, the HRTEM was used to analyze the phases and microstructures of the HVDC-AE44-2 alloys aged at 200 °C for 12 h and 250 °C for 12 h. Figure 4 is a TEM image of the HVDC-AE44-2 alloys aged at 250 °C for 12 h. It was found that the morphology, size, and distribution of the layered Al$_{11}$RE$_3$ and the granular Al$_2$RE do not change significantly, which is consistent with the results in literature [11, 21]. However, the granular precipitate was found in the TEM images and HRTEM after aging (as shown in figures 4 (a) − (d)), and the lattice spacing of the precipitate was 0.225 nm, which proved to be Al$_4$Mn. The Al$_{11}$RE$_3$ is indexed by SAED (inset in figure 4 (b)) along the zone axis $[001]$. The $\alpha$-Mg is indexed by SAED (inset in figure 4 (d)) along the zone axis $[−12−16]$. The red dashed line is the interface between Al$_{11}$RE$_3$ and Al$_4$Mn in figure 4 (b). From the HRTEM image as shown in figure 4 (b), it can be seen that the lattice spacing of Al$_{11}$RE$_3$ is 0.223 nm, the lattice spacing of Al$_4$Mn is 0.225 nm. Figure 4 (c) is the TEM BF image including Al$_4$Mn, $\alpha$-Mg and Al$_{11}$RE$_3$. Figure 4 (d) is the HRTEM image including Al$_4$Mn and $\alpha$-Mg. The red dotted line is the interface between Al$_4$Mn and $\alpha$-Mg. As shown in figure 4 (d), the lattice spacing of $\alpha$-Mg is 0.241 nm, the lattice spacing of the precipitated Al$_4$Mn is 0.227 nm. The lattice mismatch between the maternal phase and the new phase used in this paper proposed by Turnbull–Vonnegut [22], as expressed in equation (1):

$$\delta = \frac{\alpha_m - \alpha_n}{\alpha_n} \times 100\%$$

where $\alpha_m$ and $\alpha_n$ are the lattice spacings of the maternal phase and new phase, respectively. The d($200$) Al$_{11}$RE$_3$ = 0.223 nm, dAl$_4$Mn = 0.225 nm and d($101$) Mg = 0.241 nm in this work as mentioned above. The coherent boundary is when the atoms at the boundary are located at the nodes of the two-phase lattice at the same time. The characteristic of the semi-coherent boundary is that an edge dislocation is generated at certain distances along the phase boundary, and all atoms except the atoms on the edge dislocation line are coherent [23, 24]. The lattice mismatches for the ($200$) Al$_{11}$RE$_3$/Al$_4$Mn and ($101$) Mg//Al$_4$Mn are 0.89% and 7.1%, respectively. It can be seen from figure 4 (b) that the atomic arrangement of the Al$_{11}$RE$_3$ and Al$_4$Mn at the interface are paralleled, and the lattice mismatch is 0.89%, thus there is a coherent crystallographic orientation.
The relationship between Al$_4$Mn and Al$_{11}$RE$_3$. In Figure 4 (d), the atomic arrangement of the Al$_4$Mn and α-Mg at the interface are nearly paralleled, and the lattice mismatch is 7.1%, so there is a semi-coherent crystallographic orientation relationship between the Al$_4$Mn and α-Mg [22–24]. And the coherent precipitates Al$_4$Mn can improve the strength of the alloys [25].

The precipitation of Al$_4$Mn was also found in the HVDC-AE44-2 alloys aged at 200 °C for 12 h as shown in Figures 5 (a)–(c) show the EDS analysis of the Al$_4$Mn and the Al$_{11}$RE$_3$, respectively. It can be seen from the figure 5 (a) that the morphology of the Al$_4$Mn is granular. In addition, as shown in figure 5 (d), the granular Al$_{11}$Mn$_4$ and Al$_2$RE were also observed and determined by EDS analysis in figures 5 (e) and (f), respectively. During aging, the Al$_4$Mn and Al$_{11}$Mn$_4$ are precipitated. It can be known from [11, 16, 17] that the precipitation strengthening can improve the properties of the alloy.

3.2. The aging effect on mechanical properties of the HVDC-AE44-2 alloys

Figure 6 shows the hardness versus time curves of the HVDC-AE44-2 alloys aged at 200 °C and 250 °C, respectively. The hardness test has been done more than three times, and the experimental results show that the data is stable, as shown by the error bars in figure 6. With the increasing of the aging time, the hardness of the alloy increases first and then decreases. After aging at 200 °C for 12 h and at 250 °C for 8 h, the hardness of the HVDC-AE44-2 alloys reaches a peak of 71.7 HV and 70.6 HV, respectively. The increase in hardness is related to the precipitation phases during the aging process, and the strengthening mechanisms are mainly the Orowan bypass mechanism and the Friedel cut mechanism [26].

Previous studies [27–30] have proved that there is a certain relationship between hardness and strength, and that hardness has a linear relationship with the yield strength and tensile strength of the sample. Figure 7 shows the yield strength, tensile strength, and elongation of the as-die-cast and as-aged HVDC-AE44-2 alloys at different temperatures. Moreover, each step of the tension was repeated more than three times to ensure the accuracy of the experimental data. The experimental results show that the data is stable, as shown by the error bars in figure 7 (b). It can be seen from figure 7 that the best comprehensive properties of the aging was obtained at 200 °C for 12 h. Compared with the as-die-cast HVDC-AE44-2 alloy, the yield and the tensile strengths of the as-aged HVDC-AE44-2 alloy increase by ~30.5 MPa (~20.4%) and ~22.6 MPa (~9.1%), respectively.
alloy reaches the ageing peak, the precipitated particles are fine and dispersed, and the precipitated particles may be cut by dislocations. After reaching the aging peak, the size of the precipitates increases, which is a non-cutable obstacle. In addition, the energy barrier required for the Friedel cut mechanism is higher than the Orowan bypass mechanism when the particle spacing of the precipitates increases. The mechanism of precipitates hindering dislocations changes from Friedel cut to Orowan bypass \cite{31–33}. For the aged HVDC-AE44-2 alloys at 250 °C for 12 h, the sizes of the precipitates grow coarser and the dislocation slip mechanisms may change from the Friedel cut mechanism to the Orowan bypass mechanism, which leads to the strength of the aged HVDC-AE44-2 alloy at 200 °C for 12 h being higher than that of the aged HVDC-AE44-2 alloy at 250 °C for 12 h.

### 3.3. The microstructure evolution

Heat treatment can change the grain size \cite{34}. According to the Hall-Petch formula, the change of the grains sizes affects the properties of the alloy \cite{35, 36}. In order to understand whether the aging causes the changes in the grain distribution, size and orientation of the HVDC-AE44-2 alloys, the microstructure of the as-die-cast and as-aged HVDC-AE44-2 alloys at different temperatures were analyzed by EBSD. Figure 8 shows the IPF
maps, grains sizes distributions and corresponding misorientation angle distributions of the HVDC-AE44-2 alloys under different conditions. As shown in figures 8(a), (d) and (g), the grains distributions in as-die-cast and as-aged HVDC-AE44-2 alloys at different temperatures remain almost unchanged. It can be seen from figures 8(b), (e) and (h), the average grains size of the alloys as-die-cast, as-aged at 200 °C for 12 h and aging at 250 °C for 12 h are 7.56 μm, 7.64 μm and 7.74 μm, respectively. The grain size basically remains unchanged before and after aging. The average grain size before and after aging remained nearly the same. Figures 8 (c), (f) and (i) show the misorientation angle distributions of the alloys as-die-cast, as-aged at 200 °C for 12 h and at 250 °C for 12 h. The low-angle grain boundaries (LAGBs, < 10°) of the alloys as-aged and as-die-cast also remain nearly the same. Both misorientation angle distributions of the as-aged alloys at 200 °C for 12 h and at 250 °C for
Before the hardness peak is reached, the corresponding precipitation strengthening effect of the HVDC-AE44-2 alloy is obviously improved due to the coherent precipitates. The strengthening mechanisms of the dislocations obstructed by the particles spaces of the precipitates are increased, the energy barrier required for the Friedel cut mechanism is higher than that of the Orowan bypass mechanism, and the mechanisms of the dislocations obstructed by precipitates changes from the Friedel cut mechanism to the Orowan bypass mechanism. Due to the lower critical shear stress required by the Orowan bypass mechanism, the energy barrier that the Friedel cut mechanism needs to overcome becomes higher and it turns to the Orowan bypass mechanism. When the precipitate sizes increased with the increasing of aging time, which is a non-cutable obstacle. After the hardness peak is reached, the precipitate sizes become coarser, the accumulation of the dislocations can still be seen. However, in figure 9(c), when g = 000–2, these dislocations disappear. According to the values of g · b, the dislocation type can be determined as (a) dislocation.

3.4. The dislocation observation by TEM
To further investigate the effect of the precipitates on the dislocation slip, figure 9 presents the TEM bright field, dark field images and SAED (inset), in which the dislocations accumulated by precipitates were observed. The types of dislocations are usually distinguished by the values of g · b (g: Diffraction vector, b: Burgers vector) [38–41]. The dislocation is invisible if g · b = 0, the dislocation is visible and if g · b=0. Figure 9(a) is the bright field image along the zone axis [2–1–10]. It can be seen that dislocations are accumulated. Figures 9(b) and (c) are the bright field image and dark field image under g = 01–10 and g = 000–2, respectively. In figure 9(b), when g = 01–10, the accumulation of the dislocations can still be seen. However, in figure 9(c), when g = 000–2, these dislocations disappear. According to the values of g · b, the dislocation type can be determined as (a) dislocation.

4. Discussion

4.1. The crystallographic relationships between the precipitates and the pre-existing phases in as-die-cast alloy
The precipitates play a key role on improving the properties of the alloys [42–45]. It was found that the precipitates of the Al4Mn and the Al11Mn4 are the main precipitation strengthening phases in the as-aged HVDC-AE44-2 alloys based on the TEM and HRTEM analysis. The coherent precipitates have a better strengthening effect [22]. The lattice mismatch for the (2 0 0) Al11RE3 // Al4Mn and (1 0 1) Mg // Al4Mn is 0.89% and 7.1%, respectively. Combined with figure 4, there is a coherent crystallographic orientation relationship between Al4Mn and Al11Mg, and there exist a semi-coherent crystallographic orientation relationship between the Al4Mn and α–Mg. The grain size basically remains unchanged before and after aging. Therefore, the strength of the HVDC-AE44-2 alloy is obviously improved due to the coherent precipitates.

4.2. The strengthening mechanisms
Before the hardness peak is reached, the corresponding fine precipitate particles were dispersively distributed, which may be cut off by dislocation due to the obstructed precipitate particles. After the hardness peak is reached, the precipitate sizes increased with the increasing of aging time, which is a non-cutable obstacle. When the particles spaces of the precipitates are increased, the energy barrier required for the Friedel cut mechanism is higher than that of the Orowan bypass mechanism, and the mechanisms of the dislocations obstructed by precipitates changes from the Friedel cut mechanism to the Orowan bypass mechanism [31–33, 46]. The average size of the Al4Mn in figure 5(a) is about 100 nm. Compared with figure 4(a), the average precipitate sizes of the alloy aged at 250 °C are coarser than that of the aged at 200 °C. When the precipitate sizes become coarser, the energy barrier that the Friedel cut mechanism needs to overcome becomes higher and it turns to the Orowan bypass mechanism. Due to the lower critical shear stress required by the Orowan bypass mechanism, the strength of the alloy aged at 250 °C for 12 h is lower that of the alloy aged at 200 °C for 12 h, which shows the feature of the over aging [47]. It is worth noting that the increase in strength after aging is not accompanied by a significant loss in ductility (The elongation was 19.6% in the die casting. The elongation was 19.1% in the aged at 200 °C for 12h).
4.3. The dislocations are impeded by the precipitates

According to the Hall–Petch formula, the grain sizes affect the properties of the alloy significantly. However, it was found that the grain size remained almost unchanged before and after aging. When the tensile specimen is stretched, the dislocations slipping are impeded by the precipitates, and the \((a)\) dislocations accumulated around the precipitates, thereby enhancing the strength of the HVDC-AE44-2 alloys.

5. Conclusion

This work mainly studies the effect of aging on the microstructures and properties of the HVDC-AE44-2 alloys. The following conclusions can be drawn from the results:

1. As the aging time increases, the hardness of the HVDC-AE44-2 alloys first increases and then decreases, and reached the aging peak after aging at 200°C for 12 h.
2. The comprehensive properties of the HVDC-AE44-2 alloys aged at 200°C for 12 h are the best one. It is worth noting that the increase in strength after aging was not accompanied by a significant loss in ductility (The elongation of the HVDC-AE44-2 alloys changed from 19.6% to 19.1%, which is similar to the conclusion of Nie et al.).
3. The crystallographic orientation relationship between the Al₄Mn and the Al₁₁RE₃ was found to be a coherent relationship, and there exist a semi-coherent crystallographic orientations relationship between the Al₄Mn and \(\alpha\)-Mg.
4. The as-aged HVDC-AE44-2 alloys are strengthened by both the granular Al₄Mn and the Al₁₁Mn₄.
5. The \((a)\) dislocations were accumulated around the precipitates due to the dislocation slipping impeded by the precipitates.

Acknowledgments

The authors would like to express their sincere thanks to Dr Yin Fan, P. Eng. of National Hydrological Service, Meteorological Service of Canada, Environment and Climate Change Canada, for her proofreading. The work was financially supported by the Qinghai Provincial Science and Technology Key Program (No. 2018-GX-A1).

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