Effect of different casting techniques on the microstructure and mechanical properties of AE44-2 magnesium alloy

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Abstract

AE44-2 magnesium (Mg) alloys were fabricated by gravity casting (GC), high pressure die casting (HPDC), and high vacuum assisted high pressure die casting (HVHPDC). The effect of these three different casting techniques on the microstructure evolution, texture, and mechanical properties of the AE44-2 alloy was investigated. The results showed that the different cooling rates in these three different casting techniques led to the different distribution and morphology of the precipitated phases, and rapid cooling contributed to a dense network distribution of the phases as well as grain refinement. In addition, the faster cooling rate resulted in a decrease of the dislocation accumulation. The addition of vacuum assistance in the HPDC process increased texture strength. The average grain size of the HPDC alloy was reduced by 90.4% compared to the GC alloy and the yield strength increased by 85.7 MPa due to rapid cooling. The elongation of the HVHPDC alloy increased by 2.3% compared to the HPDC alloy due to vacuum assistance. Moreover, the mechanical properties improved for the alloys in the order of GC < HPDC < HVHPDC because of grain refinement caused by the faster cooling rate. Based on the analysis of the strengthening mechanisms, the rapid cooling process of the HPDC alloy led to better strengthening compared to the GC alloy. In addition, grain refinement contributed to 82.1% of the strengthening mechanism.

1. Introduction

Magnesium (Mg) alloys are the lightest structural metal material [1], and are specially used for energy saving and environmental protection applications, where the benefits of lightweight have allowed for a significant reduction in fuel usage and its corresponding environmental benefits [2, 3]. More specifically, Mg alloys with high specific strength and recyclability are the most promising structural metals used in the aerospace and automotive industries [1–3]. However, the potential applications of Mg alloys require the material to exhibit different key properties, such as creep resistance, high-temperature mechanical properties, and corrosion resistance [2–4]. It is, therefore, crucial to understand the relationship between the casting method of such alloys, their microstructure and their performance: the casting method affects the microstructure, which determines the performance. Mg alloys have good fluidity and pouring properties, which can be used to manufacture complex parts, making casting the main manufacturing method for magnesium alloy parts [1, 4, 5]. In addition, the as-cast conditions or parameters influence the performance [5, 6].

Commonly used casting methods for Mg alloys include gravity casting (GC), high pressure die casting (HPDC), squeeze casting, semi-solid metal casting, and other casting processes [7]. In the traditional process of GC, the molten metal solidifies under the influence of gravity. On the other hand, the molten metal in HPDC is injected into the mold at a high speed and pressure and solidifies rapidly under these conditions, which comprises a precision casting process. Compared with GC, HPDC is the main process used for Mg alloy production, with high productivity, reduced mechanical secondary processing, and rapid cooling, which can effectively refine α-Mg master matrix and improve the mechanical properties of the alloys [5–7]. Bai et al [8]
observed the difference in microstructure, tensile strength, and creep properties of a Mg-4Al-(1-4)La alloy obtained by GC and HPDC. Shastri H et al [9] also reported that the HPDC AZ91 alloy has better tensile and creep properties compared with the GC analog. The die-casting process is only suitable for metals with good fluidity and forming properties. Pores are formed in the castings when using the above-described methods and heat treatment is not possible [10]. However, high vacuum assisted high pressure die casting (HVHPDC) has been developed as a new type of casting process and a special variant of the HPDC process in which die-cast alloys are heat treatable [1, 11]. The porosity of the alloy prepared by HVHPDC is significantly reduced and the density of die castings is improved [1, 11, 12]. Li S et al [11] proved that HVHPDC can reduce the gas volume of the mold cavity, reduce the number of pores, and improve the mechanical properties of the Mg-6Gd-3Y-0.5 Zr alloy compared with the alloy developed by HPDC. Therefore, HVHPDC is one of the most promising emerging casting processes.

A new type of die-casting AE44 (Mg-4Al-4RE) alloy was developed by Hydro magnesium, which exhibited good high-temperature mechanical properties and creep resistance [13, 14]. Studies on the microstructure, intermetallic compound stability, mechanical properties and creep properties of the AE44 alloy in different casting processes have proven it to be the most promising die-casting Mg alloy for automobile engine front brackets [14–18]. For example, Zhang et al [2] reported that the AE44-2 (Mg-4Al-4Ce/La-0.4Mn) alloy exhibited excellent tensile properties and corrosion resistance, which were mainly attributed to the grain refinement and precipitation of the intermetallic compounds at the grain boundaries, and the existence of a dense Al-La/Ce phase film, respectively. Rzychon et al [15] also reported that the formation of the Al11RE3 and Al2RE phases and the suppression of the Mg17Al12 phase were the main factors that improved the creep resistance and high-temperature mechanical properties of the AE44 Mg alloy, and it was found that the thermostable Al11RE3 phase at the grain boundary could suppress the sliding. Moreover, Zhu et al [19] proved that the addition of Mn element has a significant impact on the creep resistance of the Mg-Al-RE alloys, which is related to the dynamic precipitation of nano Al–Mn phase. The AE44 alloy is mostly reported as obtained by the HPDC method, and GC and HVHPDC AE44 alloys have rarely been reported. Although the grains of die-casting seem acceptable, no report on the casting texture and the grain size of the AE44 alloy using the three different casting techniques is available.

Therefore, the microstructure evolution, casting texture, and mechanical properties of AE44-2 Mg alloys fabricated by the three different casting techniques, namely GC, HPDC, and HVHPDC, were studied to determine the most suitable casting method for mass production. In addition, the strengthening mechanisms of the alloys were discussed.

2. Experimental procedure

The AE44-2 magnesium alloy provided by Magontec Xi’an Co. Ltd (Shanxi Province, China) was used as the raw material. As shown in figure 1, after the AE44-2 Mg alloy was re-melted in a furnace under a mixed protective gas composed of SF6 and N2, the AE44-2 Mg alloy melt was prepared by gravity casting, high pressure die casting,
and high vacuum assisted high pressure die casting. The GC and die casting (HPDC and HVHPDC) processes were carried out using a traditional metal mold and a DM300 precision horizontal cold chamber die casting machine, respectively. A melting temperature of 680 °C before casting was used. 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 GC, HPDC, and HVHPDC alloys was 190 °C by temperature gun measurement.

The chemical composition of the alloys was determined by inductively coupled plasma emission spectrometry (ICP-OES), and the results are shown in table 1. The microstructure of the casting specimen was observed using an optical microscope (OM, LEICA DMI3000M) and a scanning electron microscope (SEM) (Zeiss ZEISS-6035 field-emission emission) equipped with energy-dispersive x-ray spectroscopy (EDS). The second phase of the casting sample was examined using a Brooke 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°. EBSD data were

| Alloy | Al  | Ce  | La  | Mn  | Mg  |
|-------|-----|-----|-----|-----|-----|
| AE44-2| 4.32| 2.17| 1.11| 0.23| Bal.|

Figure 2. (a) HPDC and HVHPDC structures and (b) AE44-2 casting from test specimen.

Figure 3. Optical micrographs of the (a) GC, (b) HPDC, and (c) HVHPDC alloys.
obtained from a Zeiss ZEISS-6035 field-emission emission scanning electron microscope (SEM) equipped with TSLMSC. Tensile tests were carried out using a universal testing machine (Instron-5982) at room temperature with a tensile rate of 1 mm min$^{-1}$ and using the Vickers microhardness to test the hardness of the casting sample. Figure 2(a) shows HPDC and HVHPDC structures. Figure 2(b) shows AE44-2 casting from test specimen. The metallographic samples were 3 mm in height and 3.2 mm in diameter. The GC tensile samples were 15 mm in gauge length, 4 mm in gauge width, and 3 mm in gauge thickness. The die-casting tensile samples were 60 mm in gauge height and 6.4 mm in gauge diameter.

### 3. Results and discussion

#### 3.1. Microstructure

The cross-section OM micrographs of the AE44-2 alloys prepared by the three different casting techniques are shown in figure 3. As observed in figure 3(a), the intermetallic compound phase in the GC alloy is relatively homogeneously distributed in the Mg matrix, and no obvious defects were observed. As shown in figures 3(b) and (c), a few pores can be identified in the HPDC and HVHPDC alloys on the surface of the cross-section. Compared with the GC alloy, defect bands appeared in the HPDC and HVHPDC alloys. Narrow bands could be observed in the HPDC and HVHPDC alloys, which follows a profile parallel to the surface of the casting. This type of defect is induced by shear stress, whereby the viscosity of the melt filling the mold increases until structural defects appear [20, 21]. In addition, the defect band appeared in the ‘core’ region, which is rich in intermetallic phases. Relevant studies have shown that the formation of these defect bands has been observed in the die casting of magnesium and aluminum alloys, which can be controlled with change to the casting methods [21–23]. The defect bandwidth in the HPDC and HVHPDC alloys was 0.32 mm and 0.26 mm, respectively. Therefore, the defect bandwidth of the die casting alloys is reduced by the vacuum-assisted process.

Figures 4(a)–(c) and (d)–(f) show the OM and SEM cross-section microstructures near the surface of the GC, HPDC, and HVHPDC alloys. It can be seen from figures 4(a) and (b) that the distribution of the intermetallic phase is different due to the different cooling rates. In contrast to the GC alloy, the intermetallic phase of the HPDC alloy is significantly dense due to the rapid cooling. In the HPDC and HVHPDC alloys (figures 4(b) and (c)), the narrow band, rich intermetallic phases are found in the ‘skin’ region, and a dendritic microstructure is observed in the outer layer. This is consistent with previously reported results corresponding to the ZAE244 alloy [21]. Moreover, the HPDC and HVHPDC alloys exhibit a small amount of externally solidified crystals (ESCs). However, the band in the HVHPDC alloy is reduced as well as the dendritic microstructure in the outer layer, likely because vacuum can accelerate liquid flow and reduce the viscosity of the melt in contact with the mold.

It can be observed from figure 4(d) that the distribution of the intermetallic phase is relatively scattered, which mainly appears as needle-like and particle shaped. As shown in figures 4(e) and (f), these intermetallic phases gradually form a continuous network distribution as needle-like, rod-like, polygonal, and blocky shapes. Combined with the analysis from figures 3(b) and (c), the pores in the HVHPDC alloy is less than that in the HPDC alloy. The intermetallic phase in the HVHPDC alloy is relatively homogeneously distributed. Since the
formation of a second phase is mainly considered a thermodynamic process or a non-equilibrium solidification process, the different distribution of the solute atoms and phase composition are mainly controlled by the cooling rate [8]. The results reveal that the intermetallic compounds present different forms depending on the cooling rate. Intermetallic phase will be further discussed below. This inevitably affects the corresponding mechanical properties.

3.2. Strengthening phases

The XRD patterns obtained from the three different casting techniques of the AE44-2 alloys are provided in figure 5. Employing XRD analysis, the α-Mg, Al₁₁RE₃, Al₂RE, and Al₁₀RE₂Mn₇ intermetallic phases were observed in the AE44-2 alloys obtained by the three casting techniques, and no significant change in the intensity of the diffraction peaks was observed. The main precipitate in the three casting conditions corresponds to the Al₁₁RE₃ phase.

An EDS analysis was performed on the AE44-2 alloy samples to further observe the morphology and distribution of the Al₁₁RE₃, Al₂RE, and Al₁₀RE₂Mn₇ phases. As shown in figure 6, the precipitates were mainly distributed in the grain boundaries and the inside of the grains, in which the main elements were Al and RE, and a small amount of Mn. In addition, combining the XRD phase analysis and the corresponding EDS mapping images, it was determined that the precipitates (intermetallic phase) corresponding to the Al₁₁RE₃ phase.

As illustrated in figures 7(c), (f), (i), the misorientation angles of the GC, HPDC, and HVHPDC alloys are concentrated at the low-angle grain boundaries (LAGBs) and the high-angle grain boundaries (HAGBs). The latter phenomenon can be inferred from the texture of the alloy. This may be related to the dislocation accumulation caused by the growth of the crystal grains during the solidification process [26]. As
shown in (figures 7(f) and (i)), the misorientation angle distributions of the HPDC and HVHPDC alloys present two spikes around $60^\circ$-$70^\circ$ and $85^\circ$-$90^\circ$, which may be the occurrence of ESCs in the cold chamber. During injection high-speed filling, the ESCs particles are re-melted and broken, which affects their shape [27].

Theoretically, the grains grow randomly during the solidification process of the molten metal. However, in practice, there is a temperature gradient when the grains are cooled, and the nucleation grows along the direction of heat loss, which inevitably leads to the formation of the casting texture [28, 29]. Figure 8 shows the \{0001\}, \{11-20\}, and \{10-10\} pole figures of the GC, HPDC, and HVHPDC alloys. TD and ND represent the transverse and normal direction of the cast ingot, respectively. In figure 8(a), the pole density points of the pole figures in the GC alloy are distributed along the center and the edge, and the maximum texture intensity is at 48.23. Analyzing figure 7(a), it can be observed that the grains in the GC alloy are relatively coarse and small in number, which results in high texture intensity. In figures 8(b) and (c), the \{0001\}, \{11-20\}, and \{10-10\} pole figures of the HPDC and HVHPDC alloys have a scattered distribution of pole density points, and the texture intensity is 3.99 and 4.77, respectively. The addition of vacuum assistance in the HPDC process promotes the change of the texture intensity. This is likely because vacuum helps the liquid flow and affects the direction of the crystal growth and temperature gradient during rapid cooling. In the solidification process, different temperature gradients will form different random textures. The orientation of the grains changes due to the relative motion between the melt and the mold, so the directional solidification texture obtained by the casting texture growth of
the hexagonal close-packed structure is \{001\} \langle210\rangle [29, 30]. However, the HPDC and HVHPDC alloys produced a random texture, which is called die-casting texture.

The dislocation accumulation is caused by the relative motion of the crystal plane during grain growth due to the difference in the temperature gradient and the solidification pressure. Figure 9 displays the kernel average misorientation (KAM) images of the GC, HPDC, and HVHPDC alloys. The growth and relative motion of the grains in the GC alloy, and the accumulation of residual stress results in a high KAM value (0.56) due to the small temperature gradient in the solidification process. The average KAM value of the HPDC alloy is 0.09 less than that of the GC alloy because the temperature gradient increase reduces the relative motion of grain growth. The average KAM value of the HVHPDC alloy is 0.04 less than that of the HPDC alloy, since vacuum assistance accelerates the liquid flow, promotes the rapid solidification of the grains, and reduces the growth time of the crystal grains. The rapid cooling of pressurized casting mismatch the cooling rate of \(\alpha\)-Mg and the second phase, increasing the dislocation density around the particles, that is, the stress of the die-cast alloy cannot be released [31]. However, the faster cooling rate can reduce the dislocation accumulation in the alloy.

3.4. Mechanical properties

Figure 10 shows the tensile properties of the AE44-2 alloy developed using the three different casting techniques. The faster cooling rate has a significant effect on the tensile properties of the AE44-2 alloy. The tensile yield strength (TYS) and ultimate tensile strength (UTS) of the alloys decrease in the order of HVHPDC > HPDC > GC. The TYS of the HPDC alloy is 85.7 MPa higher than that of the GC alloy, and the UTS of the HVHPDC alloy is 12.2 MPa higher than that of the HPDC alloy. The elongation (EL) of the HVHPDC alloy increased by 2.3% compared to that of the HPDC alloy. The relevant strengthening mechanism for the above alloys has two factors to be considered. Firstly, according to the Hall-Petch relationship, the die-casting process can greatly improve alloy strength through a high cooling rate, which could result in grain refinement compared to the GC process. Secondly, fine Al–RE phases at the grain boundaries can effectively
fortify the grain boundary in the AE44-2 alloys. In addition, the Al11RE3 phase may hinder grain boundary sliding and dislocation motion [32].

Figure 11(a) shows the Vickers hardness variation of the AE44-2 alloys using the different casting techniques. The average Vickers hardness of the alloys is presented in figure 11(b). It can be observed that the Vickers hardness is low for the GC alloy, intermediate for the HPDC alloy, and high for the HVHPDC alloy. The difference in hardness range of the HVHPDC alloy is smaller than that of the HPDC alloy. For both the HPDC and the HVHPDC alloy, the hardness of the edge area is 2 ~ 3 HV higher than in the center area. This shows that the grain size difference between the central region and the edge region is small. Compared with the GC alloy, the average hardness of the HPDC and HVHPDC alloys is higher than 12 ~ 16 HV. This contributes to the large reduction of grain size. Therefore, the Vickers hardness is greatly increased due to the fine grain strengthening effect.

3.5. Strengthening mechanism

Based on the experimental results, it was determined that the cooling rate of the casting process has a significant impact on the grain size and precipitated phases. Compared with the GC alloy, the HPDC alloy has finer grains and second-phase network distribution. Therefore, the strengthening mechanism of the HPDC alloy is mainly refinement and second phase strengthening. The contribution of the grain refinement on the mechanical properties of the Mg alloy can be calculated using the Hall–Petch equation [33, 34]:

\[
\sigma_{HP} = \sigma_0 + kd^{-1/2}
\]  

where \(\sigma_{HP}\) is the yield strength due to grain refinement, \(\sigma_0\) is the original yield strength of the material, \(d\) is the average grain size, and \(k\) is the strengthening coefficient closely related to the crystal structure. Based on equation (1), the contribution of rapid cooling to the fine grain strengthening of the AE44-2 alloy can be derived as follows:
where $\Delta \sigma_{HP}$ is the increment in yield strength due to grain refinement, and $d_{HPDC}$ and $d_{GC}$ are the average grain size of the HPDC and GC alloys, respectively. When $d > 1 \, \mu m$, the $k$ value of the Mg alloy is constant ($280 \sim 320 \, MPa/\mu m$) [35]. $\Delta \sigma_{HP}$ is calculated to be $69.9 \sim 79.8 \, MPa$.

The network distribution of the precipitated Al-RE phases plays an important role in the strength of the Mg-Al-RE alloys since the Al$_2$RE phases could effectively hinder dislocation motion [32]. Therefore, the second-phase strengthening is mainly due to the Orowan strengthening mechanism. Zhang et al [36] proposed an improved Orowan equation for magnesium alloy, shown below:

$$
\Delta \sigma_{HP} = k \left( \frac{1}{d_{HPDC}} - \frac{1}{d_{GC}} \right)
$$

Figure 9. Kernel average misorientation map of the (a) GC, (b) HPDC, (c) HVHPDC alloys, and (d) average KAM value.

Figure 10. Tensile properties of the GC, HPDC, and HVHPDC alloys: (a) tensile stress-strain curve, (b) corresponding TYS, UTS, EL.
\[ \Delta \sigma_p = \frac{0.13Gb}{d_p} \left( \frac{1}{2b} \right)^{\frac{1}{2}} \ln \left( \frac{d_p}{2b} \right) \]  

where \( \Delta \sigma_p \) is the yield stress of precipitation strengthening, \( G \) is the shear modulus \((=1.66 \times 10^4 \text{ MPa for Mg alloys})\), \( b \) is the magnitude of the Burgers vector \((3.21 \times 10^{-10} \text{ m})\), and \( f_v \) and \( d_p \) are the volume fraction and the average particle size of the Al-RE phase, respectively. The calculated value is \( \Delta \sigma_p = 15.3 \text{ MPa} \).

The sum of the yield stress of the fine grain strengthening and the second phase strengthening is calculated to be \( 85.2 \sim 95.1 \text{ MPa} \), which is very close to the experimental data \( (\Delta \sigma_{YTS} = 85.7 \text{ MPa}) \). However, compared with the GC alloy, the rapid cooling of the HPDC alloy leads to strengthening, in which fine grain strengthening accounts for 82.1%.

### 4. Conclusions

The following conclusions are drawn from the experimental results of this work.

1. The AE44-2 alloy in all the three casting conditions consists of \( \alpha \)-Mg, Al_{11}RE_{3}, Al_{2}RE, and Al_{10}RE_{2}Mn_{7} \) phases. The rapid cooling results in a denser network distribution of the second phase and grain refinement. The intermetallic phase is relatively homogeneously distributed in the HVHPDC alloy.

2. During the solidification process, the faster cooling rate can result in a decrease of the dislocation accumulation. The addition of vacuum assistance in the HPDC process increased texture strength. HVHPDC was the most suitable casting method for AE44-2 alloy among the three different casting techniques.

3. The mechanical properties of the GC alloy are the lowest, for the HPDC alloy they are intermediate, and for the HVHPDC alloy, they are the highest. The poor mechanical properties of the GC alloy were attributed to the coarse grains and the dispersion of the second phase. The presence of a continuous network of intermetallic phase and the finer grain size contributed to superior mechanical properties in the HVHPDC alloy.

4. Based on the strengthening mechanism analysis, the rapid cooling of the HPDC alloy leads to strengthening compared to the GC alloy, the \( \Delta \sigma_{YTS} \) and \( \Delta \sigma_p \) is calculated to be \( 69.9 \sim 79.8 \) and \( 15.3 \text{ MPa} \), respectively. Among them, fine grain strengthening has the biggest contribution of 82.1%.

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