Inhomogeneity of microstructure and mechanical properties of a 500 mm diameter heavy section semi-continuous cast AZ61 magnesium alloy ingot

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Abstract: For the large magnesium alloy ingot, there is a considerable difference in cooling rate of different parts in the ingot, which leads to non-uniform distribution of the secondary phases, solute segregation and tensile properties. In the present research, an heavy AZ61 alloy ingot with a diameter of 500 mm was made by semi-continuous casting. The microstructure and mechanical properties at different positions along the radial direction of the large ingot were investigated by using an optical microscope (OM), a scanning electron microscope (SEM), an energy dispersive spectroscopy (EDS), and a micro-hardness tester. The results indicate that the microstructure of the AZ61 ingot is non-uniform in different locations. It changes from equiaxed to columnar grains from the center to the edge; the average grain size gradually reduces from 1,005 μm to 763 μm, the secondary dendrite arm spacing reduces from 78 μm to 50 μm, and the Mg17(Al,Zn)12 phase is also refined. The micro-hardness value increases from 55.4 HV at the center to 72.5 HV at the edge of the ingot due to the microstructure differences, and the distribution of micro-hardness at the edge of the ingot is more uniform than that in the center. The tensile properties at room temperature show little difference from the center to the edge of the ingot except that the elongation at the edge is only 3.5%, much lower than that at other areas. The fracture mechanism is ductile fracture at the center and cleavage fracture at the edge of the ingot, and at the 1/2 radius of the ingot, a mixture of ductile and cleavage fracture is present.

Key words: AZ61 magnesium alloy; semi-continuous casting; microstructure; properties

Manganese alloys are widely used in the automotive and aerospace industries, and in 3C electronic equipment as well as in military equipment due to their high specific strength, high damping capacity, excellent shielding property against electromagnetic interference, and recyclability [1-6]. To satisfy the needs of engineering applications, the manufacture of heavy section parts made from magnesium alloys is necessary. But, in large ingots, the non-uniformity of the microstructure will lead to inhomogeneous mechanical properties, and therefore decrease the utilization ratio of the ingot.

In general, the grain size and microstructure of as-cast alloys are directly influenced by cooling rate [7].

Moreover, in the semi-continuous casting process, the cooling rate is mainly determined by casting parameters, such as withdrawal rate and circulation velocity of cooling water (cooling rate). For the large magnesium alloy ingot, there is a considerable difference in cooling rate of different parts in the ingot, which leads to non-uniform distribution of the secondary phases, solute segregation and tensile properties [8-9]. Hence, research on the microstructure inhomogeneity of different parts in large magnesium alloy ingots is significant, even though there are very few reports in this field. In this work, the differences of grain size, secondary dendrite arm spacing, micro-hardness and tensile properties at room temperature along the radial direction of a φ500 mm AZ61 ingot were discussed.

1 Experimental procedure

The AZ61 (Mg-6.5Al-1.03Zn, by wt.%) alloy ingot
with a diameter of 500 mm was prepared using the semi-
continuous casting process. The pouring temperature was
700 °C ± 10 °C, circulation velocity of cooling water was 220
L·min⁻¹ and the withdrawal rate was 35 mm·min⁻¹. Samples
for microstructure observation were cut into cubes, with
dimensions of 10 mm × 10 mm × 10 mm, along the radial
direction of the ingot, as shown in Fig. 1.

The samples were etched in a solution of 5 mL acetic acid
+ 5 g picric acid + 10 mL water + 100 mL ethyl alcohol for
20 s. Microstructure observations were carried out using an
NEOPHOT 30 optical microscope. The average grain sizes
were measured using the mean linear intercept method. The
fracture surface morphologies were observed using a TESCAN
VEGA II LMU scanning electron microscope (SEM) with
energy dispersive spectroscopy (EDS). Micro-hardness was
measured using a HXS-1000TAKY micro-hardness tester.
Tensile testing was performed on the CMT-5105 electronic
universal testing machine, with a rate of 4 mm·min⁻¹ at room
temperature. The calculation method of the secondary dendrite
spacing (λ) is shown in Fig. 2.

As the ingot surface was machined before further processing,
the microstructure and properties of the ingot at this area (at
250 mm) were not analyzed and discussed in this paper.

2 Results and discussion
2.1 As-cast microstructure
Optical microstructures of the AZ61 ingot at different positions
along the radial direction of the ingot are shown in Fig. 3. It
can be clearly seen that the as-cast microstructure consists of
an equiaxed grain zone, a columnar grain zone and a fine grain
zone in this ingot. The equiaxed grains formed at the areas no
more than 180 mm from the center of the ingot [Figs. 3(a) to
3(d)], the columnar grains formed at the edge [240 mm from
the center, Fig. 3(e)] and the fine grains at the surface [Fig.
3(f)] of the AZ61 alloy ingot.

The average grain size and secondary dendrite arm spacing
at different positions of the ingot are measured, as shown in
Fig. 4 and Fig. 5, respectively. The values of average grain size
reduces gradually from 1,005 μm at the center to 763 μm at
Fig. 3: Microstructures of AZ61 alloy ingot at different positions from center: (a) 0 mm, (b) 60 mm, (c) 120 mm, (d) 180 mm, (e) 240 mm, and (f) 250 mm

Fig. 4: Average grain size at different positions

Fig. 5: Secondary dendrite arm spacing at different positions

the edge of the ingot, and the secondary dendrite arm spacing reduces from 78 μm to 50 μm. This is because the cooling rate at the ingot edge is higher than that at the center, so the temperature gradient is small inside the ingot. The adsorption and clustering of atoms in the liquid alloy lead to the growth of the grains. The fast cooling rate near the surface of the ingot hindered the adsorption of the atoms, which resulted in a higher degree of solute segregation, leading to the finer grain size at the edge of the ingot.

It is well known that Mg-Al-Zn alloy is composed of the α-Mg matrix and the second phase β-Mg_{17}(Al,Zn)_{12} \[^{[10-13]}\]. Figure 6 shows the SEM images of β-Mg_{17}(Al,Zn)_{12} phase at different positions of the AZ61 alloy ingot. At the center [Figs. 6(a) and 6(d)] and 1/2 radius [Figs. 6(b) and 6(e)] of the ingot, the size of β-Mg_{17}(Al,Zn)_{12} phase is larger, and the β-Mg_{17}(Al,Zn)_{12} phase is mainly distributed at the grain boundaries. However, at the edge of the ingot [Figs. 6(c) and 6(f)] the β-Mg_{17}(Al,Zn)_{12} phase is fine and is uniformly distributed in the matrix or along the grain boundaries. In addition, a mass of lamellar structure is found around the thick β-Mg_{17}(Al,Zn)_{12} phase at the center and 1/2 radius of the ingot [Fig. 6(d) and 6(e)]. According to the EDS results (Table 1), the contents of Al and Zn in the granular phase (arrows A, B, and C in Fig. 6) are much higher than those in the lamellar structure phase (arrows D and E in Fig. 6).

Table 2 shows the EDS results for the α-Mg matrix of the ingot. The contents of Al are 2.88%, 3.35% and 3.87% in the positions 0, 120 mm and 240 mm from the center, respectively. However, the Zn is not found at the ingot center, and the content of Zn is 0.28% at the 1/2 radius and 0.64% at the edge of the ingot. The content of Al and Zn at the edge of ingot is far higher than that in the center. This is because the fast cooling rate at the edge of the ingot hindered the adsorption of the Al and Zn atoms, which resulted in a higher degree of solute segregation in the α-Mg matrix at the edge of the ingot. Conversely, at the center of ingot, a large number of Al and Zn elements form second phase compounds, resulting in the reduction of Al and Zn content in the α-Mg matrix. So, the Zn content at the center of ingot was too low to be measured.

Figure 7 shows the relative electric conductivity at different positions in the ingot. The relative electric conductivity can indirectly reflect the solid solubility of elements in the alloy; it has an inverse relationship with the solid solubility \[^{[4]}\]. Figure 7 shows that the value of relative electric conductivity gradually reduces from 14.81% at the center to 14.57% at the edge of the ingot. In other words, the values of solid solubility of the elements gradually increase from the center to the edge of the ingot. This is consistent with the SEM and EDS measurement results.

In the semi-continuous casting process, the microstructure is influenced by the cooling rate of the ingot \[^{[7]}\]. The cooling rate at the edge of the ingot was higher than that at the center, so that the edge of the ingot has a higher degree of undercooling (ΔT) than the center. During the solidification process, the nucleation and growth rate of grains mainly depend on the degree of undercooling. The relationships between the

Fig. 4: Average grain size at different positions

Fig. 5: Secondary dendrite arm spacing at different positions
nucleation rate, the growth rate and the degree of undercooling are given as follows:\(^\text{(14)}\):

\[
I = A \exp\left(\frac{-\Delta G_s + Q}{k\Delta T}\right)
\]

\[
R = \mu_1 \Delta T
\]

where \(I\) is the nucleation rate, \(A\) is a constant depending on the solidification structures, \(Q\) is the diffusion activation energy, \(\Delta G_s\) is the free energy change, \(k\) is Boltzmann's constant, \(R\) is the growth rate, \(\mu_1\) is a constant, \(\Delta T\) is the degree of undercooling. From equation 2 and equation 3, the \(I\) and \(R\) values increase with a rise in the degree of undercooling.

The nucleation rate and growth rate of AZ61 magnesium alloys increase gradually from the center to the edge due to the higher cooling rate at the edge. So, at the edge of the ingot, the adsorption of the atoms is hindered, and the solid solubility of the alloying elements Al and Zn is increased due to the higher cooling rate. The grain, secondary dendrite arm and \(\text{Mg}_17(\text{Al,Zn})_{12}\) phase were refined due to the higher nucleation rate. Conversely, the grain size, the secondary dendrite arm spacing and \(\text{Mg}_17(\text{Al,Zn})_{12}\) phase size are larger and the content of solute elements is smaller at the center of the ingot.

<table>
<thead>
<tr>
<th>Positions</th>
<th>Al</th>
<th>Zn</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>32.75</td>
<td>10.11</td>
<td>Bal.</td>
</tr>
<tr>
<td>B</td>
<td>33.59</td>
<td>10.12</td>
<td>Bal.</td>
</tr>
<tr>
<td>C</td>
<td>30.56</td>
<td>9.31</td>
<td>Bal.</td>
</tr>
<tr>
<td>D</td>
<td>15.30</td>
<td>4.70</td>
<td>Bal.</td>
</tr>
<tr>
<td>E</td>
<td>12.31</td>
<td>2.07</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Distance from the center (mm)</th>
<th>Al</th>
<th>Zn</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>2.88</td>
<td>0</td>
<td>Bal.</td>
</tr>
<tr>
<td>120</td>
<td>3.35</td>
<td>0.28</td>
<td>Bal.</td>
</tr>
<tr>
<td>240</td>
<td>3.87</td>
<td>0.64</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

Fig. 7: Relative electric conductivity of different positions in AZ61 alloy ingot
2.2 Mechanical properties

Figure 8 and Table 3 show the distribution of micro-hardness and the standard deviation of micro-hardness values at different positions within the ingot. From the center to the edge of the ingot, the micro-hardness of the alloy increases from 55.4 HV to 72.5 HV. In addition, the micro-hardness value of the $\alpha$-Mg matrix also increases. The micro-hardness of the as-cast AZ61 alloy is mainly influenced by the solid solubility of alloy solute elements, the secondary dendrite arm spacing and the distribution of second phase strengthening particles in the matrix [15]. From Figs. 7 and 8, it can be seen that the micro-hardness increases with an increase in the solid solubility of Al and Zn. In addition, combining with Fig. 5, it can be seen that the micro-hardness of the alloy also increases gradually with a reduction in the secondary dendrite arm spacing and $\text{Mg}_17(\text{Al,Zn})_{12}$ phase size. Table 3 shows that the standard deviation of micro-hardness of both the AZ61 alloy and $\alpha$-Mg matrix decreases from the center to the edge. These results indicate that the distribution of micro-hardness at the edge of the ingot is more uniform than that in the center.

The tensile properties of the ingot including ultimate tensile strength (UTS), yield strength (YS) and elongation at different positions are listed in Table 4. The UTS and YS values have little variation from the center to the edge of the AZ61 ingot. However, it can be seen that the elongation at the edge of ingot is only 3.5%, much lower than that at other positions. This is because in the equiaxed grain zone (Fig. 3), the elongations of the alloy are uniform due to the isotropic nature of the grains. However, because of the directivity of heat transfer during solidification, the columnar grain with anisotropy is formed at the edge of the ingot, which decreases the elongation significantly to 3.5%.

Table 4: Tensile properties at room temperature

<table>
<thead>
<tr>
<th>Distance from center (mm)</th>
<th>UTS (MPa)</th>
<th>YS (MPa)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>167</td>
<td>72</td>
<td>5.7</td>
</tr>
<tr>
<td>60</td>
<td>166</td>
<td>70</td>
<td>5.3</td>
</tr>
<tr>
<td>120</td>
<td>173</td>
<td>76</td>
<td>6.3</td>
</tr>
<tr>
<td>180</td>
<td>175</td>
<td>77</td>
<td>6</td>
</tr>
<tr>
<td>240</td>
<td>175</td>
<td>77</td>
<td>3.5</td>
</tr>
</tbody>
</table>

Figure 9 shows the SEM images of tensile fracture surfaces at different positions in the ingot. As shown in Fig. 9(a), at the center of the ingot, the fracture surface exhibits lots of dimples. However, at the edge of the ingot [Fig. 9(c)], a number of cleavage planes and steps are present, and some tearing ridges can also be observed in local areas of the tensile fracture surface. Figure 9(b) exhibits a mixture of cleavage and ductile fracture at the 1/2 radius of the ingot.

The influencing factors of tensile properties mainly include the average grain size, and the size and distribution of the second phase. Firstly, the average grain size from the center to the edge of the ingot decreases gradually as shown in Fig. 4, so the alloy near the edge exhibits better tensile performance. Secondly, the cooling rate decreases from the center to the edge of the ingot. At the center of the ingot, the size of the $\beta$-$\text{Mg}_17(\text{Al,Zn})_{12}$ phase is larger, and the $\beta$-$\text{Mg}_17(\text{Al,Zn})_{12}$ phase mainly distributes at the grain boundaries [Figs. 6(a) and 6(d)]. However, the $\beta$-$\text{Mg}_17(\text{Al,Zn})_{12}$ phase is fine and uniformly
distributes in the matrix or along the grain boundaries at the edge of the ingot. The second phase strengthening effect is more significant at the edge.

3 Conclusions

(1) A large AZ61 alloy ingot with 500 mm in diameter was successfully produced by the semi-continuous casting method. The as-cast microstructure consists of the equiaxed grain zone at the areas no more than 180 mm from the center of the ingot, the columnar grain zone at the edge and the fine grained zone at the surface of the ingot.

(2) The main compound in the alloy is Mg17(Al,Zn)12 phase near the grain boundaries at the center and 1/2 radius of the ingot. The β-Mg17(Al,Zn)12 phase is fine and non-uniformly distributes in the matrix or along the grain boundaries at the edge of the ingot. The average grain size is reduced from 1,005 μm at the center to 763 μm at the edge of the ingot. From the center to the edge, the secondary dendrite arm and Mg17(Al,Zn)12 phase are refined, and the solid solubility of alloy elements Al and Zn is increased.

(3) Microstructure differences lead to the different mechanical properties at the different positions within the ingot. The micro-hardness value increases from 55.4 HV at the center to 72.5 HV at the edge of the ingot and the distribution of micro-hardness at the edge of the ingot is more uniform than that in the center. The UTS and YS values have little variation from the center to the edge of the AZ61 ingot. However, the elongation at the edge of ingot is only 3.5%, much lower than that at other positions.

(4) The fracture mechanism is ductile fracture at the center and cleavage fracture at the edge of the ingot, and at the 1/2 radius of the ingot, a mixture of ductile and cleavage fracture is present.

References


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