Effect of homogenizing treatment on microstructure and conductivity of 7075 aluminum alloy prepared by low frequency electromagnetic casting

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Abstract: The heat treatment process has great effects on microstructure and conductivity of ingots. In this study, the ingots of high strength 7075 aluminum alloy were prepared by low frequency electromagnetic casting (LFEC), and the effect of different homogenization processes (single-step homogenization at 465 °C for different holding times and three-step homogenization) on the microstructure and conductivity of 7075 aluminum alloy were studied by means of metallographic microscopy, electrical conductivity test, differential thermal analysis and X-ray diffraction phase analysis. For comparison, the ingot by conventional direct casting (DC) under the same conditions was also prepared. Results show that the non-equilibrium eutectic phases with low melting point in the ingot dissolve continuously into the matrix as the holding time of single-step homogenization increases. The endothermic peak of non-equilibrium phases can not be completely eliminated through a 24 h single-step homogenization, but can be eliminated after a three-step homogenization (200 °C/2 h + 460 °C/6 h + 480 °C/12 h). Meanwhile, the homogenization has a better effect on the LFEC ingot than the conventional DC ingot. Under the same homogenizing conditions, the grains of LFEC ingot are characterized by a lower content of low melting point phases and the ingot shows higher electrical conductivity than DC ingot.

Key words: LFEC; heat treatment; 7075 aluminum alloy; microstructure

Recently, the considerable commercial and military interest in Al-Zn-Mg-Cu alloys has been developed significantly. The Al-Zn-Mg-Cu alloys have high element contents and wide solidification range. However, the structure with more precipitates at the grain boundary and larger size grains is obtained than other aluminum alloys when the conventional DC casting is used to produce the ingot. Moreover, such structure cannot be completely eliminated in the subsequent processing and heat treatment, resulting in the degradation of performance in toughness and corrosion resistance [1-5]. Based on the principle of Vive’s CREM (Casting, Refining, Electromagnetic) [6], low frequency electromagnetic casting (LFEC) was developed by professor Cui [7-9] and his research team with an AC induction coil arranged around the conventional DC casting mold and the application of a low frequency current (<50 Hz). This new technique has been applied to prepare both round and rectangular ingots of aluminum alloys. The LFEC process showed better grain refining effect than the CREM process [6]. Improved surface quality and reduced macrosegregation were also achieved with the application of LFEC process [10-11].

However, the previous research was mainly focused on the effect of LFEC process on the as-cast microstructure and conductivity. The research on the evolution of microstructure and conductivity of the ingot prepared with LFEC process in the subsequent heat treatment process [12-14] is quite limited. It is necessary to carry out a deeper research on this subject, because the heat treatment process has big effect on microstructure and conductivity of the ingots, and the conductivity is a good way to judge corrosion-resistance. In this study, the ingots of high strength 7075 aluminum alloy were prepared by LFEC casting, and for comparison, the ingots by conventional DC casting were also prepared under the same conditions. The homogenizing treatment...
was carried out for the ingots and the evolution of microstructure and conductivity was systematically studied.

1  Experimental procedure

The 7075 aluminum alloy (Al-5.6%Zn-2.5%Mg-1.6%Cu-0.23%Cr-0.1%Ti, by wt.) ingots with 150 mm in diameter were fabricated with conventional DC and LFEC casting, respectively. The specific casting parameters are listed in Table 1. The disc-shaped samples with 30 mm in thickness were cut along the cross section of the ingot, and then put into the homogenizing furnace to conduct single- and three-step homogenization.

Table 1: Specific parameters of casting 7075 aluminum alloy ingot

<table>
<thead>
<tr>
<th>Casting temp.</th>
<th>Withdrawal rate</th>
<th>Water flow rate</th>
<th>Field coils current</th>
</tr>
</thead>
<tbody>
<tr>
<td>720 °C</td>
<td>100 mm-min⁻¹</td>
<td>60 L·min⁻¹</td>
<td>120 A (20Hz)</td>
</tr>
</tbody>
</table>

Figure 1 shows the DSC curves of the as-cast microstructure of the ingots with two casting processes. The first peak on the curve appears at 478 °C indicating the melting point of the phases. Therefore the temperature of homogenization cannot exceed 478 °C in order to avoid the overheat during the homogenization process. The temperature of single homogenization is set at 465 °C with the consideration of the measurement error and control precision of the furnace. So the single-step homogenization was carried out at 465 °C for 6, 16 and 24 h, respectively, and the three-step homogenization was achieved by 200 °C/2 h + 460 °C/6 h + 480 °C/12 h.

The metallurgical microstructure was observed using a Leica DMI5000 optical microscope (OM). The phase analysis was conducted by means of SISCIA58.0 metallographic analysis software to qualitatively and quantitatively investigate the degree of solubility of the dendrite network and the area fraction of residual phases. Using Sigmascope conductivity instrument made in Fisher Company, the average value of electrical conductivity was obtained from 5 tests for each sample. The precipitates were analyzed by Pw3040/60 XRD from Netherlands, with a scanning rate of 6°·min⁻¹ and a measurement range of 5° to 110°. DSC analysis was performed using MDSC Q100 differential scanning calorimeter made in U.S.A, with a heating rate of 10 °C·min⁻¹.

2  Results

2.1  As-cast microstructure

Figure 2 shows the microstructure of 7075 aluminum alloy ingots fabricated by the two casting processes. It can be seen that many non-equilibrium eutectic phases present in the as-cast microstructure, and the DC ingot has more coarse non-equilibrium eutectic phases than the LFEC ingot. The LFEC ingot was characterized by fine and homogeneous grains. The dendrite arm spacing of DC alloy was about 35 μm, which is larger than that of LFEC alloy (25 μm). The area percentage of non-equilibrium eutectic phases at the grain boundary is 3.16% for LFEC ingot and 3.8% for DC ingot.

Figure 3 shows the area percentage of the residual phases after different homogenization treatment. After single-homogenization, the area fraction of the residual phases decreases as the homogenizing time prolongs. The area of residual phases is the smallest after the three-step
The endothermic peak at 480 °C disappears gradually after homogenizing for 16 h, and a new endothermic peak at about 490 °C appears. Therefore, all the non-equilibrium eutectic phases with low melting point dissolve into their matrix. Moreover, there are new endothermic phases separated out, and the new endothermic peak exists even after 24 h homogenization. All the endothermic peaks disappear after the three-step homogenization, indicating again the higher efficiency of the three-step homogenization.

2.4 X-ray diffraction analysis

Figure 6 illustrates the X-ray diffraction curves of as-cast and homogenized alloys. The Al₂MgCu phase appears only after single-step homogenization. Since the cooling rate of semi-continuous casting is fast, before Zn and Cu element separate out, they have solved into the non-equilibrium solid phases. Thus, the non-equilibrium solid phases can be expressed as Mg(Al, Zn, Cu)₂ phase. During homogenizing process, Zn diffuses from Mg(Al, Zn, Cu)₂ phase to the matrix and leads to the transformation of Mg(Al, Zn, Cu)₂ to Al₂CuMg(S). Meanwhile, LFEC ingot exhibits a broader peak of η-(MgZn₃) phase than DC ingot.

2.5 Effect of homogenization on conductivity

Figure 7 shows the histogram of conductivity of the ingots vs different homogenization treatments. The as-cast ingot shows the lowest conductivity. The conductivity of samples increase with extending the homogenizing time for single-step homogenization, indicating again the higher efficiency of the three-step homogenization. Compared the homogenized microstructure of DC ingot with LFEC ingot, the LFEC ingot shows lower area fraction of residual phases with the same homogenizing time.

2.3 DSC curve analysis after different homogenization

Figure 5 shows the DSC curves of the ingots after different homogenizations. The enthalpy of endothermic peak decreases gradually as the homogenizing time increases. The enthalpy of endothermic peak of LFEC ingot is smaller than that of DC ingot. The endothermic peak at 480 °C disappears gradually after homogenizing for 16 h, and a new endothermic peak at about 490 °C appears. Therefore, all the non-equilibrium eutectic phases with low melting point dissolve into their matrix. Moreover, there are new endothermic phases separated out, and the new endothermic peak exists even after 24 h homogenization. All the endothermic peaks disappear after the three-step homogenization, indicating again the higher efficiency of the three-step homogenization.
Fig. 4: Microstructures of 7075 aluminum alloy ingot after different homogenization treatment: 465 °C/6 h, DC (a); 465 °C/6 h, LFEC (b); 465 °C/16 h, DC (c); 65 °C/16 h, LFEC (d); 465 °C/24 h, DC (e); 465 °C/24 h, LFEC (f); three-step homogenization, DC (g); three-step homogenization, LFEC (h)
Fig. 5: DSC curves of 7075 aluminum alloy after different homogenization: 465 °C/6 h (a), 465 °C/16 h (b), 465 °C/24 h (c), 200 °C/2 h + 460 °C/6 h + 480 °C/12 h (d)

(a) Peak: 480.26 °C
Onset: 478.17 °C
Enthalpy: 4.8216 J/g

(b) Peak: 495.26 °C
Onset: 490.46 °C
Enthalpy: 1.304 J/g

(c) Peak: 494.31 °C
Onset: 491.26 °C
Enthalpy: 0.5492 J/g

(d) Peak: 495.18 °C
Onset: 493.73 °C
Enthalpy: 0.3062 J/g
eutectics at the grain boundary. Thus, LFEC ingot acquires less low-melting point eutectics at the grain boundary than DC ingot.

The refinement of original microstructure has effect on the structure change and heat treatment in the following process. The dissolution rate and dissolubility of the second phase of LFEC ingots was higher than those of DC ingot under the same conditions during homogenization, which was ascribed to the less non-equilibrium second phase in the original as-cast microstructure of LFEC ingot.

The homogenization process is mainly based on the atomic diffusion process within the grain. The solute atoms diffuse from a higher content at the grain boundary to the grains. The homogenization process has almost come to an end when the composition becomes uniform. The dynamic equation of homogenization is as follows:

\[ \frac{1}{T} = P \ln \left( \frac{t}{G L^2} \right) \]  

where, \( T \) is the absolute homogenization temperature, K; \( P=R/Q \), \( R \) is the gas constant, J·(mol·K)\(^{-1} \), \( Q \) is the diffusion activation energy, kJ·mol\(^{-1} \); \( G = 4.6/ (4\pi^2 D_0) \), \( D_0 \) is the coefficient which is unrelated to the temperature, cm\(^2\)·s\(^{-1} \); \( t \) is the homogenizing time, h; \( L \) is the dendrite arm space, \( \mu \)m.

As shown in Equation (1), when the temperature of homogenization is constant, the time for homogenization is shortened as the dendrite arm space reduces, meaning that the refinement of original as-cast microstructure can improve the diffusion speed of solute atoms during homogenization. The LFEC ingot acquires finer grains, and the atom diffusion rate of LFEC ingot is higher than that of DC ingot, which makes the LFEC ingot better homogenization.

\[ \frac{1}{T} = P \ln \left( \frac{t}{G L^2} \right) \]  

3 Discussion

During LFEC casting process, under the influence of electromagnetic field, a forced convection is formed in the melt. The forced convection makes the aluminum melt with low temperature near to the wall of the mold flows to the center of the mold during the LFEC casting. Therefore under the influence of forced convection, the temperature field becomes more uniform. The relatively uniform melt temperature is beneficial to forming plenty of homogeneous grain nuclei in the melt, and therefore reduces grain remelting caused by local overheating and increases the number of effective grain nuclei. The grains are closely connected with each other before the growth stage of dendrite, and then they form a fine and homogeneous microstructure with a globular shape.

During solidification of DC ingot, the low-melting-point eutectics will form at the grain boundary due to the high content of alloying element of the 7075 aluminum alloy and the non-equilibrium solidification of DC casting process. The application of electromagnetic field can somehow improve the solid solubility \(^{[7]} \) and then lead to the decrease of the fraction of low-melting point eutectics at the grain boundary. Thus, LFEC ingot acquires less low-melting point eutectics at the grain boundary than DC ingot.

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4 Conclusions

(1) The as-cast microstructure of LFEC ingot is characterized by finer grains, more homogeneous distribution and smaller area percentage of non-equilibrium phases at the grain boundary compared with
the DC ingot.

(2) The three-step homogenization has a better effect than single homogenization, as it can completely eliminate the endothermic peak of non-equilibrium phases. Many MgZn\(_2\) phases present in the ingot with three-step homogenization.

(3) The LFEC ingot acquires more uniform microstructure than the DC ingot, and obtains better homogenizing effect under the same homogenization conditions. The enthalpy of low-melting point phases of the LFEC ingot is smaller than that of DC ingot after different homogenization.

(4) The conductivity of the LFEC ingot is consistently higher than that of the conventional DC ingot under the same homogenization condition. The ingot has higher conductivity after three-step homogenization than single homogenization.

References


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