Research on Homogenization Treatment of Mg-8wt.% Sn-1.5 wt.% Al Alloy

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Abstract: The microstructure and morphology of Mg-8wt.%Sn-1.5wt.%Al alloy during homogenization were investigated by optical microscope (OM), scanning electron microscope (SEM), energy dispersive analysis (EDS) and HCT-3 comprehensive thermal analyzer. The results show that plenty of dendrite segregation arise in the as-cast alloy, and non-equilibrium eutectic phases distribute along the grain boundaries; at the same time, a large number of Sn and Al atoms segregated. The differential scanning calorimetry (DSC) curve shows that only one endothermic peak ($549 \degree C$) exists, which is caused by the redissolution of Mg$_2$Sn phase; according to the homogenization empirical formula, 460 $\degree C$ to 500 $\degree C$ is the appropriate heat period. However, surface oxidation appears when treated at 500 $\degree C$; so atmosphere (Ar$_2$) protect was used, after treating for 500 $\degree C \times 48$ h, ideal resolution effect has been achieved.

1. Introduction
Magnesium alloy has the advantages of low density ($1.738 g/cm^3$), high specific strength, high specific stiffness, and good shock absorption performance. It is widely used in automotive, electronics, aviation, aerospace and other fields, and has important application values and broad application prospects [1-3], which is a “green engineering material”. The casting-state microstructure of magnesium alloy is easy to grow in the form of dendrites, and the segregation of main alloy elements is serious, which reduces the plastic workability of the alloy. To improve the uniformity of the alloy structure and composition, the ingot needs to be homogenized heat treatment before plastic deformation of the alloy [4,5]. The Mg-Sn series magnesium alloy is a series of heat-resistant magnesium alloys with great application prospects.

The eutectic temperature of Mg-Sn is 562$\degree$ C. At the eutectic point, the solid solubility of Sn in Mg is 14.85wt%. As the temperature decreases below the eutectic point, the solid solubility of Sn in Mg decreases rapidly, and it drops to 0.45wt.% at 200$\degree$C. Such a large solid solubility and solid solubility change provide a good basis for the solid solution strengthening and aging strengthening of Mg-Sn series magnesium alloys [6-9].

However, there are many coarse dendrites in the as-cast microstructure of the casting-state Mg-Sn-Al magnesium alloy. To give full play to the effects of solid solution strengthening and aging strengthening of the alloy elements, the alloy needs to be homogenized heat treatment. Homogenization treatment is to dissolve and diffuse the non-equilibrium eutectic phase at a higher temperature, so as to
eliminate the non-equilibrium eutectic structure, improve dendrite segregation, improve the plastic deformation ability of the alloy, and improve the solid solubility of the alloy elements in the matrix at the same time, which can prepare the structure for the hot deformation process and aging precipitation of the alloy \cite{10-12}. During the homogenization process, the re-dissolution of the Mg2Sn phase is the key to control the structure and properties of the alloy. This paper takes the Homogenization treatment process optimization of Mg-Sn-Al magnesium alloy as the goal, solves the problems of surface oxidation in the homogenization process of the alloy, and derives a reasonable Homogenization treatment system.

2. Experimental materials and methods

2.1. Experimental materials
The alloy in this experiment was Mg-8wt.% Sn-1.5wt.% Al magnesium alloy, and the main raw materials were pure Mg (99.95wt.%), pure Sn (99.98wt.%) and pure Al (99.95wt.%). An intermediate frequency induction melting furnace was used for smelting, using a mixed gas of argon (Ar): tetrafluoroethane (R134a) = 10:1 as the protective gas. First the crucible was preheated to 300°C, adding pure magnesium, and then increasing the temperature to 750°C. After the pure magnesium is completely melted, a certain proportion of pure Al was added. After holding for 5 minutes, a certain proportion of pure Sn was added. After holding for 5 minutes, MgO filter was added to filter the melt. The temperature was controlled at about 700°C. Finally, the crucible was taken out of the melting furnace and cooled. After the ingot being taken out, it was cut into Φ10cm×10cm specimens along the concentric circle of the ingot by wire cutting for heat treatment and microstructure analysis.

2.2. Experimental methods
The actual composition of the alloy was tested using ARL4460 inductively coupled plasma emission spectrometer; the metallographic sample was ground to 5000# sandpaper and polished, then corroded with 3wt.% picric acid solution, and finally observed on the Carl Zeiss Axiovet 2000MAT metallographic microscope; the JEM-2010 scanning electron microscope with an energy spectrometer was used to analyze the microscopic morphology and micro-area components of the sample.

3. Results and discussion

3.1. Casting-state microstructure and analysis
As shown in Figure 1, the uniformity of the structure of the casting-state alloy is poor, the grain shape is irregular, and the grain shape is "cell-like" dendrites, and the average grain size is larger, ranging from 250μm to 300μm. The second phase is mainly distributed at the grain boundary, with obvious fish-scale characteristics, and forms a non-equilibrium eutectic structure together with the α-Mg matrix to segregate at the grain boundary.

![Fig. 1 Microstructure of the as-cast alloy](image)

From the results of scanning electron microscopy and EDS energy spectrum analysis of the alloy (Figure 2), it can be seen that the content of alloying elements inside the grains is less, the content of Sn
element is only about 3wt.%, and the content of Al element is less, which is almost impossible to detect. This is because in the process of non-equilibrium solidification, α-Mg preferentially nucleates and grows, while the solute atoms are continuously pushed to the solid/liquid interface, and gather at the solid/liquid interface, and part of the alloying elements and the matrix form a non-equilibrium eutectic structure through the non-equilibrium eutectic reaction, which is distributed at the grain boundary, and part of it is segregated at the grain boundary in the form of atoms.

Figure 2 Scanning electron microscopy and EDS analysis of the as-cast alloy

Figure 2 Scanning electron microscope image and EDS energy spectrum analysis of casting-state alloy

After analyzing the casting-state structure of Mg-8wt.% Sn-1.5wt.% Al alloy, it can be found that there is a large amount of non-equilibrium structure in the casting-state structure of the alloy, and a large number of alloying elements segregate at the grain boundaries. The alloying elements are not evenly distributed in the matrix. To provide a good organization basis for the subsequent thermoplastic deformation, the alloy must be homogenized heat treatment.

3.2. DSC curve

Figure 3 shows the DSC curve of the casting-state Mg-8wt.% Sn-1.5wt.% Al alloy. The results show that there is only one endothermic peak in the alloy, which is 549.0°C, and it is the melting of the high melting point eutectic phase in the alloy. There is no endothermic peak of low-melting eutectic phase due to the extremely low content of Mg17Al12 phase in the alloy, which has been re-dissolved during heating. Refer to the DSC curve and the empirical formula of Homogenization treatment (Equation 1): the reasonable Homogenization treatment temperature is 466°C~507°C, so 460°C, 480°C, and 500°C are selected for Homogenization treatment of the alloy.
\[ T_c = (0.9 \sim 0.95) T_m \]  \hspace{1cm} (1)

Among them, \( T_m \) is the melting point of the alloy.

![DSC curve of casting-state alloy](image1)

**Figure 3** DSC curve of casting-state alloy

3.3. Homogenizing the microstructure during heat treatment

![Microstructure evolution](image2)

**Figure 4** Microstructure evolution during Homogenization treatment

(a) 460°C×4h; (b) 460°C×8h; (c) 460°C×16h; (d) 480°C×4h; (e) 480°C×8h; (f) 480°C×16h;
(g) 500°C×4h; (h) 500°C×8h; (i) 500°C×16h

The Homogenization treatment of the alloy was carried out at 460°C, 480°C, and 500°C respectively, and the heating time was 4h, 8h, and 16h respectively. As shown in Figure 3, when the Homogenization treatment was carried out at 460°C, the remelting effect of the alloy was not ideal. There are still more non-equilibrium eutectic structures in the structure; when the temperature rises to 480°C, with the
extension of time, the number of non-equilibrium eutectic structures begins to decrease, but there are still more second phases remaining. When performing Homogenization treatment at 500°C, after 16h, the alloy has achieved good heat treatment effect. Most of the non-equilibrium phases have been re-dissolved, but a small amount of second phases still exists at the grain boundaries, and the heating time needs to be further extended to proceed for re-dissolved. However, when the alloy was homogenized at 500°C for 16h, the surface oxidation phenomenon appeared, which made it impossible to carry out further heat treatment.

Fig.5 Microstructure after treating at 500°C for 48h under argon atmosphere

Since there is no endothermic peak of the low-melting eutectic phase in the DSC curve of the alloy, and the oxidation phenomenon only exists on the surface of the sample, it can be inferred that the alloy is not oxidized due to over-firing. To avoid further heat treatment of the alloy on the premise of avoiding oxidation on the surface of the alloy, the conditions of argon atmosphere protection were selected. Under argon protection, the sample can be subjected to 500°C×48h Homogenization treatment. The metallographic structure after heat treatment is shown in Figure 5. It can be seen from the figure that after the alloy is heat treated at 500°C×48h, the non-equilibrium eutectic phase in the alloy has almost completely re-dissolved, and only a small amount of the second phase is distributed in the crystal, which has achieved a good uniform heat treatment effect.

Fig.6 Scanning electron microscope and EDS analysis after treat at 500°C for 48h in argon atmosphere

The SEM image and EDS energy spectrum analysis result of the alloy is shown in Figure 6. It can
be seen that after the Homogenization treatment at \(500^\circ\text{C} \times 48\text{h}\), the segregation of alloy elements at the grain boundary has been well improved. Both the element and the Al element are uniformly distributed in the matrix. The Sn element content in the matrix is about 8.1wt.\%, and the Al element content is about 1.6wt.\%, indicating that the homogenization effect of the alloying elements has been achieved.

4 Conclusion

1) There is severe dendrite segregation in the casting-state structure of Mg-8wt.% Sn-1.5 wt.% Al alloy, and there are lots of Mg2Sn non-equilibrium eutectic phases on the grain boundaries, and Al elements mainly exist in the form of atoms in the alloy, a large number of Sn atoms and Al atoms segregate at the grain boundaries at the same time.

2) Due to the surface oxidation phenomenon of Mg-8wt.% Sn-1.5wt. % Al alloy during homogenization, it needs to be heat treated under the protection of argon atmosphere. The reasonable homogenization system for this alloy is \(500^\circ\text{C} \times 48\text{h}\), after processed with this homogenization system, the non-equilibrium eutectic phase in the alloy is basically completely decomposed, and the alloy elements are also distributed uniformly.

Acknowledgements

The authors would like to acknowledge the Projects No.51871195 by National Natural Science Foundation of China for the financial support of this study.

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