Macro-Segregation and Microstructural Characteristics in Rheo-Diecasting of a High Strength Al–4.8 mass% Si–0.7 mass% Mg Alloy

Byoung-Hee Choi, Young-Soo Jang, Byung-Geun Kang and Chun-Pyo Hong*

Department of Materials Science and Engineering, Yonsei University, 134 Shinchon-dong, Seodaemun-ku, Seoul 120-749, Korea

Rheo-diecasting with electromagnetic stirring has been carried out to investigate macro-segregation and microstructural characteristics of a high strength Al–Si–Mg alloy under various process conditions. The amount of initial heterogeneous nucleation and the degree of temperature uniformity in the slurry are dependent on the superheat of the melt at pouring into the slurry making vessel. The formation of macro-segregation in rheo-diecasting was closely related to the microstructural characteristics of semi-solid slurries. Slurries which have fine and globular α-Al particles prevent the formation of macro-segregation throughout the rheo-diecast specimens. However, coarse and non-globular α-Al particles result in the formation of macro-segregation, leading to a non-uniform hardness distribution, dependent on the thickness of the product. Various casting process parameters were examined to prevent the formation of macro-segregation, and the optimal ranges of the process conditions for rheo-diecasting were determined as follows: the injection velocity of 0.3–1.0 m·s⁻¹ and the mold temperature above 150°C.

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1. Introduction

In recent decades, the need to improve the product quality in automotive industries has become imperative and led to the development of microstructural modification through novel casting processes.¹ High-pressure diecasting (HPDC) has been widely used to manufacture many automotive components due to its high productivity and economic benefits. However, there are some limitations in using this process to achieve high mechanical performance because of porosity defects resulting from turbulent die filling and shrinkage defects in thick-walled parts.² The rheo-diecasting process represents an alternative method for the fabrication of high-strength aluminum products.³,⁴ In rheo-diecasting, inner defects can be effectively eliminated due to the laminar feeding pattern and low gas content in semi-solid slurries, so blister defects do not occur during the T6 heat treatment. Recently, rheo-diecasting was successfully adopted to the manufacturing of engine blocks and suspension arms.⁵,⁶

For practical application of rheo-diecasting, a uniform mechanical property of complex near-net-shaped products is one of the most important aspects to be considered. In order to ensure the quality of the complicated products, it is essential to have a uniform microstructure without the formation of macro-segregation throughout the products. In recent research, the formation of segregation in various casting methods was reported.⁷–⁹ Most studies have focused on the microscopic segregation of the alloying elements or the formation of share band segregation in normal solidification processes,¹⁰–¹³ whereas no studies have been reported on the formation of macro-segregation in rheo-diecasting of Al-alloys.

In order to obtain high-quality rheo-diecasting products, the following two conditions must be satisfied: (1) proper alloy design for rheo-diecasting process and (2) optimization of slurry-making conditions and rheo-diecasting process parameters without having casting defects such as pore, misrun and macro-segregation. New alloys based on the Al–Si–Mg system for rheo-diecasting with high mechanical properties were developed.¹⁴,¹⁵ The characteristics of the slurry during the feeding process in the mold cavity and the mechanical properties of the rheo-diecast products depend on the quality of semi-solid slurries, which is determined by the size and uniformity of globular α-Al particles.¹⁶

In this study, the main goal is to investigate the effects of the slurry-making and rheo-diecasting process conditions on the formation of macro-segregation in thin and thick wall parts. Optimization of the slurry making conditions was carried out through cooling curve experiments and microstructural observations with various superheats of the melt at pouring and various solid fractions of slurries. Process parameters for rheo-diecasting were optimized with various injection velocities and mold temperatures. Hardness was measured at various thicknesses of rheo-diecast products.

2. Experimental Procedure

The chemical composition of the alloy used in the present study is shown in Table 1. In order to evaluate the exact temperatures corresponded with the solid fractions of the alloy from 0.0 to 0.5, the solid fraction-temperature relationship of the alloy was calculated, as shown in Fig. 1. The solid fraction in the present study was calculated by thermo-calc, which is based on the Scheil’s equation. However, in the rheo-casting process, the actual solidification of the alloy was proceeded at the non-equilibrium state. In order to validate the simulated data, the cooling curve experiment was carried out, and the results of the liquid and solidus temperatures show almost similar tendency with the calculated values. The

<table>
<thead>
<tr>
<th>Composition</th>
<th>Si</th>
<th>Mg</th>
<th>Cu</th>
<th>Fe</th>
<th>Mn</th>
<th>Zn</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>4.8</td>
<td>0.7</td>
<td>0.5</td>
<td>0.15</td>
<td>0.1</td>
<td>0.3</td>
<td>0.05</td>
<td>Bal.</td>
</tr>
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*Corresponding author, E-mail: neochaosmos@gmail.com
alloy was melted and degassed at 700°C using a chemical tablet (N₂). Before the slurry-making, the molten liquid of the alloy was kept at 700 ± 5°C for 30 min after degassing.

The schematic drawings of the rheo-diecasting system integrated with the slurry making system by electromagnetic (EM) stirring and HPDC used in the present study are illustrated in Fig. 2(a). The slurry making vessel made of stainless steel was used: 60 mm in diameter and 130 mm in length. The melt was poured into the slurry making vessel at various pouring temperatures from 625 to 670°C which have different superheats from 0 to 45°C. The EM stirring was carried out within 10 s from the pouring stage into the slurry making vessel. The EM stirring was adjusted with 10 A input AC current and 60 Hz frequency, and the electromagnetic field was induced at 0.03 tesla. The EM stirring was only activated in the liquid state, not in the mushy zone. Thus, the EM stirring improves heterogeneous nucleation of α-Al particles and does not affect the growth of α-Al particles in the mushy state. Heterogeneous nuclei of α-Al particles are generated in the inner vessel by the wall nucleation effect at an early stage of solidification. During the cooling stage in a slurry-making vessel, uniform distributions of the temperature and solute fields in the slurries are successfully generated by EM stirring, resulting in the growth of α-Al particles into a globular form. The semi-solid slurry was taken out of the slurry making vessel at various solid fractions from 0.2 to 0.5 and rheo-diecasting was carried out using a 85-ton high pressure diecasting machine. The mold and sleeve temperatures were changed from 15 to 250°C using a heating system and the injection velocity of the plunger was controlled from 0.1 to 3.5 m·s⁻¹ for optimizing the casting process parameters. To evaluate the formation of macro-segregation according to the thickness of the products, rheo-diecasting using a step-shaped mold which has various thicknesses from 2 to 10 mm was carried out. The schematic drawing of the step-shaped mold is shown in Fig. 2(b).

In order to observe the microstructures of the rheo-diecast specimens, the samples were polished with SiC paper and cloth with a 0.04 µm diamond suspension and etched in the Keller’s etchant. The microstructure was observed at the center position of the rheo-diecast specimen. To estimate the microstructural characteristics and macro-segregation in the rheo-diecast specimens, quantitative microstructural analysis using an optical microscope and an image analyzer were carried out, and the results were evaluated with various superheats of the melt and solid fractions. Hardness was measured after the T6 heat treatment using a Vickers hardness tester in various thicknesses of the specimens produced by rheo-diecasting. The T6 heat treatment conditions were set at a solid solution treatment of 520°C for 6 h and an aging treatment of 160°C for 5 h.

3. Results and Discussions

3.1 Microstructural characteristics of semi-solid slurry

In rheo-diecasting, microstructural characteristics such as the size, shape and density of α-Al particles are closely related to the quality of semi-solid slurries. A thermal analysis of liquid metal using a cooling curve experiment can be used for evaluating the nucleation and growth of α-Al primary particles during solidification of an alloy. In the present study, the superheat of the melt at pouring into a slurry making vessel was changed to evaluate the amount of heterogeneous nucleation of α-Al particles and to obtain high-quality semi-solid slurries. Figure 3 indicates the typical temperature-time curve from the cooling experiment during slurry making stage. The superheat of the melt at pouring, referred to as ΔT_{superheat}, is the temperature difference between the pouring temperature and the liquidus temperature of the alloy. The recalescence temperature which is caused by the latent heat of solidification
ification, referred to as $\Delta T_{\text{sur}}$, was measured in order to estimate the amount of heterogeneous nucleation in the early stage of solidification. The temperature difference between the center and the bottom surface of the slurry, indicated as $\Delta T_{\text{sur-cen}}$, was also measured for the evaluation of the temperature uniformity of the slurries. Figure 4 shows the typical microstructures of water quenched slurries obtained under various superheats. The quenching temperature of the slurries into water was set at 608°C with the solid fraction of 0.4 to observe slurry microstructures under the same solid fraction. To evaluate the microstructural morphology of the slurries quantitatively, the parameters such as the particle size and the form factor, referred to respectively as $D$ and $F$, were adopted using the following equations:

$$D = 4A/\pi$$
$$F = p^2/4\pi A,$$

where $A$ and $p$ are the area and the perimeter of $\alpha$-Al particles, respectively. The results of thermodynamic and microstructural analysis are summarized in Table 2.

Table 2: Quantitative analysis of thermodynamic and microstructural characteristics with various superheats at pouring into the slurry making vessel.

<table>
<thead>
<tr>
<th>Superheat (°C)</th>
<th>$\Delta T_{\text{spheat}}$ (°C)</th>
<th>$\Delta T_{\text{sur}}$ (°C)</th>
<th>$\Delta T_{\text{sur-cen}}$ (°C)</th>
<th>Form factor</th>
<th>Average size of $\alpha$-Al particles ($\mu$m)</th>
<th>Density of $\alpha$-Al particles ($/\text{mm}^2$)</th>
</tr>
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<tbody>
<tr>
<td>45</td>
<td>4.6</td>
<td>5.8</td>
<td>0.61</td>
<td>118</td>
<td>0.9·10²</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>5.2</td>
<td>3.6</td>
<td>0.69</td>
<td>88</td>
<td>1.4·10²</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>7.3</td>
<td>2.7</td>
<td>0.82</td>
<td>54</td>
<td>2.4·10²</td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>3.2</td>
<td>1.8</td>
<td>0.85</td>
<td>76</td>
<td>1.7·10²</td>
<td></td>
</tr>
</tbody>
</table>

Thus a few number of $\alpha$-Al nuclei could be formed because of small supercooling for heterogeneous nucleation, leading to the decreases of $\Delta T_{\text{sur}}$. It should be noted that heterogeneous nucleation can be effectively accelerated by the large amount of supercooling created from the contact of the melt with the vessel wall at the pouring stage. Moreover, this can only occur when the pouring temperature is higher than the liquidus temperature of the alloy. The value of $\Delta T_{\text{sur-cen}}$ decreases as the superheat decreases. In the microstructural analysis, uniform and globular $\alpha$-Al particles with an average size of 54 μm were obtained at the superheat of 15°C. As the superheat increases, coarsening of $\alpha$-Al particles occurs. The particle density reaches the highest value at the superheat of 15°C and decreases as the superheat increases. When the superheat of the melt at pouring was 0°C, the size of $\alpha$-Al particles increased and the density decreased compared to those with the superheat of 15°C.

In order to evaluate the relationship between the thermal characteristics and the microstructural characteristics of the slurries, the results of microstructural and thermal analyses of $\Delta T_{\text{sur}}$ and $\Delta T_{\text{sur-cen}}$ at various superheats are indicated in Fig. 5. When the superheat was 15°C, the finest and densest $\alpha$-Al particles were formed. Moreover, the size of the $\alpha$-Al particles increases and the density decreases as the superheat decreases. In the cases of the superheats of the melt at pouring from 15 to 45°C, the value of $\Delta T_{\text{sur}}$ increases as the superheat decreases. In the case of superheat of 0°C, the melt was cooled down to the liquidus temperature in the hot ladle.
Figure 5 Effects of superheat of the melt at pouring on the thermal and microstructural characteristics: (a) particle size, density and \( \Delta T_{\text{sur}} \) and (b) form factor and \( \Delta T_{\text{sur-cen}} \).

Figure 6 The effect of thickness of rheo-diecast specimens on the microstructures for various superheats at pouring stage.

decreases or increases from 15°C, as shown in Fig. 5(a). These changes in the particle size and density with the superheat of the melt at pouring are similar to the change of \( \Delta T_{\text{sur}} \) in the thermal analyses, which is related to the amount of heterogeneous nucleation. Furthermore, the form factor decreases as the superheat increases, which shows a similar curve that is inversely proportional to \( \Delta T_{\text{sur-cen}} \), as shown in Fig. 5(b). This suggests that the shapes of the \( \alpha \)-Al particles are closely related to the temperature difference within the slurries; it is considered that the \( \alpha \)-Al particles can grow in a globular form as the temperature distribution of the slurries becomes uniform.

3.2 Effects of slurry quality on formation of macro-segregation

For the rheo-diecasting of complex shaped product which has thick and thin wall parts, uniform microstructure without the formation of macro-segregation is important to obtain high quality of final casting products. In order to investigate the formation of macro-segregation, rheo-diecasting using a step-shaped mold with various thicknesses of 10, 6, 4, and 2 mm, which is illustrated in Fig. 2(b), was carried out with two melt superheats of 15 and 45°C. The solid fraction of slurries was held at 0.4, and the mold temperature and the injection velocity of rheo-diecasting were set at 150°C and 0.5 m s\(^{-1}\), respectively. The typical microstructures obtained are shown in Fig. 6. The non-uniform flow pattern was created during the injection of the slurry into the die cavity. The formation of the macro-segregation was arose when the remained liquid of the slurry was squeezed between solidified \( \alpha \)-Al particles. When the superheat was 15°C, the flow pattern of the slurry showed smooth and laminar flow characteristics due to the fine and uniform \( \alpha \)-Al particles. Thus, uniform distribution of \( \alpha \)-Al particles was obtained throughout the specimen without the formation of macro-segregation. However, in the case of the superheat of 45°C, as the thickness of the products decreases, the formation of the macro-segregation increases due to the irregular flow pattern at the thin section created by the interruption of coarse and non-uniform \( \alpha \)-Al particles.

The effects of the superheat on the volume fraction of \( \alpha \)-Al particles with various thicknesses of the specimen were examined for various superheats ranging from 15 to 45°C, and the results are shown in Fig. 7(a). The fraction of \( \alpha \)-Al particles decreased as the thickness of the specimen decreased for all superheats, and this tendency shows the minimum at a superheat of 15°C. As described in the previous section, fine and globular \( \alpha \)-Al particles can be obtained by a large amount of initial heterogeneous nucleation (high \( \Delta T_{\text{sur}} \)) and high temperature uniformity of the slurrys (low \( \Delta T_{\text{sur-cen}} \)). Therefore, a large \( \Delta T_{\text{sur}}/\Delta T_{\text{sur-cen}} \) value implies the creation of high-quality semi-solid slurries with fine and globular microstructures. In order to investigate the correlation between the quality of the slurry and the formation of macro-segregation, the differences in the fraction of \( \alpha \)-Al particles throughout the specimens with various \( \Delta T_{\text{sur}}/\Delta T_{\text{sur-cen}} \).
T_{sur-cen} values corresponding to the superheat of the melt at pouring were examined, and the results are shown in Fig. 7(b). As the $\Delta T_{sur}/\Delta T_{sur-cen}$ value increases, the difference in the fraction of $\alpha$-Al particles becomes smaller. This confirmed that high-quality semi-solid slurries with large amounts of initial heterogeneous nucleation and high temperature uniformity can lead not only to fine and globular $\alpha$-Al particles, but also to a uniform microstructure without macro-segregation.

In the semi-solid forming process, the microstructural characteristics of final products is largely affected by the solid fraction of the slurry.18,19) In order to analyze the effects of solid fraction on the formation of macro-segregation, the microstructures of the parts with the specimen thicknesses of 10 mm and 2 mm were observed for various solid fractions from 0.2 to 0.5. Two superheats of 15 and 45°C are used and the results are shown in Fig. 8. At the superheat of 45°C, the fraction of $\alpha$-Al particles decreases with a decrease of the specimen thickness from 10 to 2 mm, especially at the solid fraction of 0.2. However, at the superheat of 15°C, fine and globular $\alpha$-Al particles were uniformly distributed for all solid fractions. The differences in the fraction of $\alpha$-Al particles between the parts of 10 and 2 mm were analyzed, and the results are shown in Fig. 9. In the case of the superheat of 45°C, the difference in the fraction of $\alpha$-Al particles exhibits the largest value at the low solid fraction of 0.2, due to the non-globular $\alpha$-Al particles. However, at the low superheat of 15°C, the differences in the fraction of $\alpha$-Al particles are smaller than those at the superheat of 45°C for all solid fractions.

### 3.3 Mechanical property and process optimization

In rheo-diecasting, uniformity of mechanical performance is an important factor to ensure high product reliability. In order to investigate the effect of macro-segregation and microstructure on mechanical properties of rheo-diecasting products, hardness was measured under two superheat conditions at various thicknesses of products as described in Fig. 8, and the results are shown in Fig. 10. In the case of the superheat of 45°C, at which the formation of macro-
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seggregation occurs, the hardness value was non-uniformly distributed throughout the specimen for various solid fractions from 0.2 to 0.5, as shown in Fig. 10(a). In the case of the superheat of 15°C, at which fine and uniformly distributed α-Al particles arise, a uniform distribution of hardness was obtained, as shown in Fig. 10(b). In the solid fraction ranges of 0.3–0.4, the hardness value was highest with a uniform distribution. It is to be noted that high-quality semi-solid slurries with fine and uniform globular α-Al particles play a key role in preventing the formation of macro-segregation, leading to the uniformity of the mechanical properties of rheo-diecasting products.

The quality of semi-solid products is largely affected by the process parameters such as the mold temperature and the injection velocity. In order to investigate the effects of these parameters on the formation of casting defects such as misrun, porosity, and macro-segregation, rheo-diecasting was carried out at various mold temperatures in the range of 15–250°C and at injection velocities in the range of 0.1–3.5 m·s⁻¹. The optimization of process parameters by try-out experiments prior to mass production was introduced to prevent the casting defects, such as pore, mis-run, and macro-segregation. In the present study, macro-segregation is considered to occur when the maximum difference in volume fraction of α-Al phase in the specimen is larger than 10%. The superheat of the melt at pouring was maintained at 15°C, and the solid fraction of the slurry was set to 0.4. The differences in the volume fraction of α-Al particles between the parts of 10 and 2 mm were analyzed and the results are shown in Fig. 11. Casting defects such as misrun and porosity were also indicated in the figure. When the injection velocity exceeded 1.5 m·s⁻¹, porosity defects resulting from the turbulent die filling in the mold cavity were found in the thick-wall parts of the products. When the injection velocity was below 0.3 m·s⁻¹ or the mold temperature was below 150°C, misrun defect and the formation of macro-segregation were found because of the lack of castability. As a consequence, when the injection velocity and the mold temperature were appropriately controlled for rheo-diecasting, in the range of 0.3–1.0 m·s⁻¹ and above 150°C, respectively, uniform microstructures throughout the specimens without the formation of casting defects were obtained.

4. Concluding Remarks

In this study, a rheo-diecasting method coupled with EM stirring was applied to investigate the macro-segregation behavior and microstructural evolution of a high strength Al–Si–Mg based alloy under various process conditions. The main results are summarized as follows:

(1) The particle size and density of α-Al particles are greatly affected by the recalescence temperature at the initial solidification stage (ΔT_melt), and the shape of α-Al particles is related to the temperature differences within the slurry (ΔT_slurry). These two thermodynamic characteristics are dependent on the superheat of the melt at pouring into the slurry making vessel. The appropriate slurry-making condition for rheo-diecasting...
was obtained at a superheat of 15°C. When the superheat was higher than 15°C, coarse and non-globular α-Al particles were found.

2) It was found that the formation of macro-segregation in rheo-diecasting is closely related to the microstructural characteristics of semi-solid slurries. Slurries with fine and globular α-Al particles lead to uniform solidification microstructures throughout the rheo-diecast specimens. However, coarse and non-globular α-Al particles result in the formation of macro-segregation.

3) The uniformity of the mechanical properties was highly affected by the formation of macro-segregation. Upon the formation of macro-segregation, the hardness value was not uniform and dependent on the thickness of the products. With the optimum slurry which has fine and uniformly distributed globular α-Al particles, uniform hardness distribution was obtained throughout the products.

4) The optimum process parameters for rheo-diecasting without casting defects were as follows: the injection velocity in the range of 0.3–1.0 m·s⁻¹ and the mold temperature greater than 150°C. With inadequate process parameters, casting defects such as pores, misrun, and macro-segregation were generated.

REFERENCES