Microstructural evolution of thixomolded AZ91D magnesium alloy with process parameters variation

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Abstract

A prototype system of Thixomolder® was used to manufacture the AZ91D magnesium alloy. The effect of various processing parameters on the microstructure was investigated using an image analyzing system. The solid particle size was found to be reduced with increasing barrel temperature while the particle morphology was not essentially affected. The size reduction and the increase of the solid particle sphericity followed the elevated screw rotation speed because of the accelerated solute diffusion. Furthermore, the refinement of the solid particles occurred because of the high cooling rate induced by low injection velocity or low mold temperature. The mechanism of nucleation and growth of the solid particle was put forward to explain the generation and evolution of non-dendritic microstructure including the particle with entrapped liquid, or with rosette shape in semisolid slurry under forced convection. It was believed that there was a critical solid fraction about 10–20%, at which the particle grew intensively, considered as the change point of coalescence and Ostwald ripening.

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1. Introduction

Thixomolding®, combining conventional die casting with plastics injection molding, is a relatively new process for production of near-net-shape parts from magnesium alloys in a single integrated machine [1–3], as shown in Fig. 1. Compared with traditional casting techniques and other semisolid metal (SSM) processing technologies, it effectively combines the slurry making and components production into a one-step process, the forming process is an environmentally friendly, high-speed injection molding process with high efficiency and energy management, the products of which is with low porosity and high dimensional stability [4–6]. With the distinct advantages in weight savings and mechanical properties, thixomolded magnesium alloys are presently applied in consumer electronics and, to a lesser degree, the automotive industry [7–10]. In practical application, the main process parameters during the thixomolding process include the barrel temperature, the screw rotation speed, injection velocity and mold temperature, which have a significant effect on the filling and solidification of semisolid slurry, and the quality and mechanical properties [6,7]. Since the microstructure of the product is the primary factor to influence properties of the final components, its control by process parameters is of importance [4,5,11]. Therefore, an effort is made in this paper to investigate the effects of various processing parameters on the microstructure by the phenomena and the generation mechanism of the non-dendritic morphology in order to improve the quality and properties of thixomolded AZ91D magnesium alloy components.

2. Experiment

A JSW JLM220-MG prototype Thixomolding® machine, equipped with a mobile phone housing mold, was used for this experiment. The raw material was the commercial alloy AZ91D with the chemical composition of 8.3% Al, 0.54% Zn, 0.14% Mn, 0.011% Si, Fe, Cu and Ni below 0.01%, and Mg balance by the ICP spectral analyzer. The solidus and liquidus temperature were obtained of 502 °C and 595 °C from the DSC, using differential scanning calorimetry, as shown in Fig. 2. Cross-sections for optical microscopy observations were rough ground, fine ground and polished, and then chemically etched in a 4% solution of nitric acid in ethanol. Self-programmed software was used for quantitative metallographic analysis of microstructure.
images, including the volume fraction, size and morphology of solid phase. Barrel temperature, screw rotation speed and injection velocity and mold temperature were modified, respectively, for the present study.

3. Results and discussion

3.1. General microstructure characteristics

The typical features of thixomolded microstructure of the AZ91D alloy are shown in Fig. 3, which can be described as a suspension of solid particles α-Mg dispersed in a liquid matrix with the lack of dendrites [1,4–7,11]. In general view, three types of morphology of the solid particles may be found in the micrographs, one is the particle with entrapped liquid, and one is with pure solid, the last is with rosette shape.

The eutectic component is shown as a mixture of α-Mg and an intermetallic compound Mg17Al12. It can be seen that the intermetallics are distributed between the spaces of the α-Mg grains with the thickness of about 1 μm. In addition, the content of Al and Zn is rich in the intermetallic compounds, but decrease towards the interface with the solid grains, confirmed by Czerwinski et al. [7,12,13].

3.2. Microstructure evolution for the different processing conditions

3.2.1. Microstructure evolution for different barrel temperature

According to the solidus and liquidus temperatures, barrel temperature was adjusted into four different values: 580 °C, 590 °C, 600 °C and 610 °C to acquire reasonable solid fraction.
for the thin wall components with the injection velocity, screw rotation speed and mold temperature of 2.09 m/s, 168 rad/min and 230 °C. A general view of the microstructure evolution in different solid fraction is shown in Fig. 4(a–d). Furthermore, in the conditions of this study, the solid fraction and morphology of solid particles, based on the quantitative and statistical analysis are listed in Fig. 5, together with some of the recent results [7].

During the process, barrel temperature was crucial to the solid fraction, the increase of barrel temperature was accompanied by the reduction of solid fraction and the particle size (Fig. 4). Surprisingly, there seemed to be a critical solid fraction at which the particle growth was particularly intense (Fig. 5), confirmed by Czerwinski [14]. In addition, it could be investigated that the particle density (Fig. 4) with entrapped liquid was diminished for a high barrel temperature. Furthermore, increasing barrel temperature did not essentially affect the particle sphericity (Fig. 5(b)).

### 3.2.2. Microstructure evolution for different screw rotation speed

To achieve the required morphology of the solid phase, a series of components were manufactured in three different screw rotation speeds for this study. The three screw rotation speeds were composed of 105 r/min, 143 r/min and 168 r/min with the barrel temperature, injection velocity and mold temperature of 600 °C, 2.09 m/s and 230 °C. The microstructure evolution with various shear rates is shown in Fig. 6(a–c) and the statistical results are plotted in Table 1.

It was seen that the solid fractions were nearly constant in different screw rotation speed for the same barrel temperature. With increasing the screw rotation speed, the particle morphology changed from dendrite to sphere via rosette (Fig. 6), coupled with the reduction of average particle size and the enhancement of morphology, as listed in Table 1, which was similar to the observation of Fan and co-workers [4,15,16].

### 3.2.3. Microstructure evolution for different injection velocity

Injection velocity had strong effect on the quality of the components by influencing the slurry flow mode, injection time and particle size as well. A variety of specimens were prepared for this study at different injection velocities of 1.52 m/s, 2.09 m/s and 2.28 m/s with the barrel temperature, screw rotation speed

<table>
<thead>
<tr>
<th>Screw rotation speed (r/min)</th>
<th>Solid fraction (%)</th>
<th>Average diameter (μm)</th>
<th>Sphericity</th>
</tr>
</thead>
<tbody>
<tr>
<td>105</td>
<td>8.45</td>
<td>34.29</td>
<td>0.674</td>
</tr>
<tr>
<td>143</td>
<td>8.23</td>
<td>33.83</td>
<td>0.677</td>
</tr>
<tr>
<td>168</td>
<td>8.33</td>
<td>28.71</td>
<td>0.709</td>
</tr>
</tbody>
</table>
Fig. 5. The solid particle size (a) and morphology (b), plotted as a function of the solid fraction. Sphericity calculated as $SC = \frac{4\pi A}{P^2}$, $A$ is a particle area and $P$ is its perimeter.

and mold temperature of 600 °C, 143 r/min and 230 °C. Table 2 shows the effect of injection velocity on the size and morphology of the solid particles.

It was apparent the solid fraction had little change in different injection velocity for the same barrel temperature, listed in Table 2. It was seen that the solid particles were thinner in a low injection velocity than in a high velocity, while the morphology was less influenced. Although the low injection velocity is favorable to refine the structure, practically, the injection velocity should be high in order to fill the cavity in a shorter time and prevent solidification of the slurry during the filling process. Furthermore, it seemed that high injection velocity would be much more suitable to the form of products with thinner walls of this study.

Table 2
The quantitative analysis of the solid particles for different injection velocity

<table>
<thead>
<tr>
<th>Injection velocity (m/s)</th>
<th>Solid fraction (%)</th>
<th>Average diameter (µm)</th>
<th>Sphericity</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.14</td>
<td>8.63</td>
<td>32.09</td>
<td>0.747</td>
</tr>
<tr>
<td>1.52</td>
<td>8.84</td>
<td>32.35</td>
<td>0.723</td>
</tr>
<tr>
<td>1.9</td>
<td>8.59</td>
<td>33.07</td>
<td>0.738</td>
</tr>
<tr>
<td>2.09</td>
<td>8.23</td>
<td>33.83</td>
<td>0.677</td>
</tr>
<tr>
<td>2.47</td>
<td>8.57</td>
<td>34.20</td>
<td>0.707</td>
</tr>
</tbody>
</table>

3.2.4. Microstructure evolution for different mold temperature

Mold temperature was set between 180 °C and 210 °C for this study with the barrel temperature, screw rotation speed and injection velocity of 600 °C, 143 r/min and 2.09 m/s. The size and morphology of solid particles are presented in Table 3.

Although the barrel temperature was set invariably, the results exhibited the reduction tendency expressed by both the content and the size of the solid particles for a low mold temperature. Furthermore, the morphology of the particles preserved consistent with high regularity, which was seemed to not follow the variation of mold temperature. The morphology was seemed to have strong dependence on the barrel temperature and screw rotation speed.

3.3. Microstructure evolution mechanism

At present, the major effort of all the semisolid technologies is focused on preparing the ideal microstructure with fine and spherical solid particles uniformly suspended in a liquid matrix. During the forming process, the slurry is achieved by partial remelting of AZ91D chips under forced convection induced by screw rotating. To explain the generation of non-dendritic morphology in semisolid slurry under forced convection, several mechanisms have been proposed, reviewed by Fan [4] and Kirkwood et al. [17]. Based on the former mechanisms and the experiment of this study, the scheme of microstructure evolution from dendritic to globular under the forced convection for this study is explained in Fig. 7.

The mechanism can be divided into two stages where, at first, in a solid state, an equiaxed structure would form due to recrystallization of chips controlled by the plastically deformation induced during the mechanical fragmentation of chips during the very initial stages of heating [14,15], as shown in Fig. 7(a and b). At the same time, Al diffuses from Mg$_{17}$Al$_{12}$ into $\alpha$-Mg, which makes the enrichment of the solute along the primary grains boundary (Fig. 7(b)). Along with the increase of the barrel temperature, the high concentration region of solute will begin to melt at the eutectic temperature and liquid phase appear at the primary and recrystallized grains boundaries. In addition, chemical segregation of Al and Zn within the particles would cause some small entrapped liquids inside the particles (Fig. 3(a), marked by arrow b) [7,14,15], as illustrated in Fig. 7(c). Furthermore, some secondary dendrite arms can detach at their roots because of melting resulted from solute enrichment and thermosolutal convection (Fig. 7(c)) [11,13]. Then the grain
boundaries created by recrystallization are disintegrated by melting during the stirring above the solidus temperature (Fig. 7(c)). After the first stage, in a semisolid state, Ostwald ripening and coalescence, driven by the reduction of the interfacial energy between particles and liquid, and controlled by solutes diffusion [4], are superimposed on the alloy melting. Coalescence usually brings the instantaneously formation of larger particle by contact of smaller particles. It appears that the existence of the particles with entrapped liquid (Fig. 3(a), marked by arrow a) or rosette shape (Figs. 3(b) and 4(b and c), marked by arrows) are caused by the coalescence coarsening of the particles, especially coalescence of dendritic arms in complex shaped particles, rather resulted from a mechanical fragmentation. However, Ostwald ripening, resulted from the Gibbs–Thomson effect, and limited here due to the very short processing times, would lead to a loss of entrapped liquid, because the small particles with entrapped liquid will dissolve and the entrapped liquid will join the bulk liquid (Fig. 7(d)) [4].

In addition, some particles would be broken down into smaller particles under the influence of forced convection, coupled with the spheroidization of the solid particles (Fig. 7(e)) due to the shearing and abrasion among particles [18]. Finally, the typical semisolid microstructure forms after the series of processes above.

Generally, the coarsening rate increases with increasing barrel temperature for the faster solute diffusion. However, growth by coalescence coarsening makes a major contribution to the total microstructural coarsening at high solid fraction because of high contacting probability of the particles, while Ostwald ripening is the dominant mechanism at low solid fraction because of
the reduced opportunity of the coalescence between the particles [6–16,18]. The critical solid fraction with the particle coarsening intensively, mentioned above, is considered as the change point of coalescence and Ostwald ripening. The plotted data in Fig. 5 indicates that the point is about 0.1–0.2 of solid fraction for this study. The growth is particularly intense above the point, while is not significantly affected under the point, shown in Figs. 4 and 5.

Furthermore, both the growth and the breakdown of the particles are accelerated with increasing the screw rotation speed for the enhanced mass transport, but the breakdown is prevalent for a high screw rotation speed for the other process parameters preserve constant, which is responsible to the particle refinement (Fig. 6).

In addition, associated with the nucleation and growth of the solid phase, the refinement of the solid particles occurs because of the high cooling rate induced by low injection velocity or low mold temperature irrespective of barrel temperature and screw rotation speed.

4. Conclusions

The effects of the processing parameters on the microstructure of thixomolded AZ91D were investigated. The microstructure is composed of \( \alpha \)-Mg surrounded by an intermetallic compound of \( \text{Mg}_{17}\text{Al}_{12} \). The results show that:

1. The mechanism of nucleation and growth of the solid particle is proposed, and proved to be effective to explain the generation and evolution of non-dendritic microstructure including the particle with entrapped liquid, or with rosette shape in semisolid slurry under forced convection.

2. The size of the solid particles reduces with increasing barrel temperature while the morphology is not essentially affected because coalescence coarsening is prevalent at high solid fraction while Ostwald ripening is dominant at low solid fraction.

3. The size reduction and the increase of sphericity of the solid particles follow the elevated screw rotation speed because both the breakdown and growth of the particle are accelerated with increasing the screw rotation speed.

4. The refinement of the solid particles occurs because of the high cooling rate induced by low injection velocity or low mold temperature.

5. It is believed that there is a critical solid fraction about 10–20%, at which the particle coarsens intensively, considered as the change point of coalescence and Ostwald ripening.

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