Original Article

The effects of annular electromagnetic stirring parameters on microstructure evolution of rheocast AZ91 magnesium alloy

N. Farzam Mehr\(^a,\)\(^*,\) H. Aashuri\(^b\)

\(^a\) Institut für Metallurgie, TU Clausthal, Robert-Koch-Straße 42, 38678 Clausthal-Zellerfeld, Germany
\(^b\) Department of Materials Science and Engineering, Sharif University of Technology, Azadi Ave., Tehran 1458889694, Iran

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ABSTRACT

In the present study, an annular electromagnetic stirrer was used to fabricate the semi-solid slurry of AZ91 magnesium alloy under various conditions of electromagnetic intensities and times. The fabricated semi-solid slurry was injected into a step-die cavity, using a hydraulic jack with ram speed of 1 m/s. The effects of electromagnetic intensity, stirring time and step thicknesses on the microstructure evolution, shape factor, globules equivalent diameter, solid fraction and microhardness of samples were investigated. The experimental results showed that obtained optimal parameters of electromagnetic stirring were 55 (A) stirring current and 30 (s) stirring time. In this conditions, the value of shape factor, was \(\sim0.85\). By increasing the thickness of the steps, the shape factor and micro-hardness value was decreased, whereas, the equivalent diameter of globules and solid fraction were increased.

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1. Introduction

As the lightest metal material, magnesium alloys have low density, high specific strength which benefits to save energy, high specific stiffness, excellent machinability and castability, good weldability and damping characteristic, and so potentially low cost [1–3]. AZ91 alloy is the most extensively used magnesium casting alloy in the field of transportation, chemical, construction and energy industries, due to the combination of good comprehensive casting and mechanical properties [4,5]. This alloy is constituted with two principle phases; (1) solid solution of Al in Mg (\(\alpha\)-Mg) and (2) \(\mathrm{Mg_7Al_12}\), which presents in an eutectic structure (\(\alpha + \beta\)) [6].

Semi-solid metal (SSM) processing in which alloys are isothermally treated between solidus and liquidus temperature, and the primary dendritic microstructure transforms into globular structure, is one of the promising technologies capable of producing high integrity components because of improved mechanical properties and the added bonus of being a near net shape route, less energy consuming, high dimensional precision, and longer die life [1,7–9]. The semi-solid
techniques use the slurry-molten metal, which included of a certain solid metal volume fraction, while, the conventional die-casting methods are based on the injection of a thoroughly liquefied metal into the cavities of a die [10]. Mainly, the most globular shape factor of the primary phase with the most refined structure in the semi-solid range, shows the best semi-solid behavior [11,12]. Three predominant mechanisms responsible for structural evolution during the SSM process, are dendrite arm fragmentation, dendrite arm root remelting, and nucleation controlled mechanisms [7]. Two main SSM processing routes are thixocasting and rheocasting [13]. The rheocasting process, is one-step processing technique which involves the application of shearing during solidification to produce a non-dendritic semi-solid slurry that can be transferred directly into a mold or die to give a final product [8,14]. There are many various methods to achieve non-dendritic microstructures [11,12,15]. Among these methods EMS is the most efficient method to produce feedstock materials in industrial scale. In this case, stirring process breaks up the dendrites, which would normally be present, thus, the microstructure in the semi-solid state consists of spheroids of solid surrounded by liquid. Annular EMS (A-EMS), is the most efficient technique between EMS methods to obtain refine and spheroidize grains, which can be used to produce semi-solid slurries for rheocasting. Compared with the ordinary EMS, A-EMS has some advantages as follows: high productivity and quality, simple equipment and lower cost [12]. The aim of this work is to investigate the microstructure evolution, variations of shape factor, globules equivalent diameter, solid fraction and micro-hardness of samples in order to the variations of A-EMS parameters.

### 2. Experimental procedure

#### 2.1. The fabrication of AZ91 magnesium alloy ingots

To produce the AZ91 magnesium alloy ingots, pure Mg, Al, Zn and Mn were melted under the protection of flux with chemical composition of 50% MgCl₂, 20% KCl, 15% MgO and 15% CaF₂ in a steel crucible in an electrical furnace with a power of 85 kW. Then, the obtained melt was poured into a metallic mold with 150 mm height and 30 mm diameter. The nominal chemical composition of the AZ91 alloy ingots fabricated for the present work which was determined by atomic absorption method (±0.01) was as follows: 9.1 wt.% Al, 0.7 wt.% Zn, 0.26 wt.% Mn and Mg as balanced.

#### 2.2. The production of AZ91 magnesium semi-solid slurry

The fabricated ingots were melted at 750 ± 1 °C in a silicon carbide crucible. To protect the melt against reaction with the environment as well as to ignition proof events during melting because of the magnesium high chemical activity, 1 wt.% MAGREX 36 flux (Fars Rizan Mavad., Iran) and 1 wt.% Ca (wrapped in an Al foil) was added to the melt. After remelting of AZ91 alloy ingots, the melt was poured into the cylindrical mild steel crucible which set in the EM-stirrer. A cylindrical low carbon steel with 5 cm diameter and 13 cm height was set in the middle of the EMS steel crucible to eliminate the middle area effect. This is named an annular electromagnetic stirrer (A-EMS) method. The A-EMS parameters were applied on the different thickness of steps according to Table 1. Note that; stirring time of AZ91 semi-solid slurry more than 35 (s) did not fill-in the step-die due to an intense reduction in a liquid fraction. Thus, the maximum stirring time was chosen as 35 (s). Due to the use of steel as an EM-stirrer crucible, since this steel affected the strength of the applied magnetic field, the maximum amount of applied current under the nominal power of EM-stirrer reduced to 55 (A).

#### 2.3. Rheocasting system

To provide the semi-solid slurry injection force into the step-die cavity, a one-way hydraulic jack (applying force from one side) with high ram speed (1 m/s) was employed. This rheocasting system was included of: (1) a furnace with 1 kW power for mold pre-heating up to 500 °C equipped with a chromel alumel thermocouple and (2) the set of mandrel and a step-die cavity with 5, 7 and 9 mm step thicknesses. The mandrel with 29.5 mm diameter and 11.5 cm height was made up of H13 hot work tool steel. Also, the step-die cavity was made up of H13 hot work tool steel.

#### 2.4. Morphology and microstructure characterizations

For the microstructure characterization, the cross-sections of samples were polished mechanically; grinding with SiC paper followed by polishing with diamond paste using mixtures containing 0.05 and 0.3 μm of Al₂O₃ particles, and etched in a solution of 60 ml ethylene glycol, 20 ml acetic acid, 1 ml nitric acid and 19 ml distilled water.

The optical microscopy, OM (OLYMPUS, U-MSSPG, Japan) was used to study the microstructure of the specimens. The field emission scanning electron microscopy, FESEM (MIRA 3, TESCAN) was applied to investigate the morphology of the samples. The X-ray diffractometer (BRUKER advance D8, Germany, Cu Kα = 1.54) was used to evaluate the phase identification. At least 10 representative areas were studied in each sample through metallographic evaluations. The effects of stirring current, stirring time and thickness of the steps, on the shape factor, globules equivalent diameter, and solid fraction of globules for each step of the specimens were performed.

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**Table 1 – Applied A-EMS parameters work.**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Stirring current intensity (A)*</th>
<th>Stirring time (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>1</td>
<td>30</td>
<td>25</td>
</tr>
<tr>
<td>2</td>
<td>30</td>
<td>30</td>
</tr>
<tr>
<td>3</td>
<td>30</td>
<td>35</td>
</tr>
<tr>
<td>4</td>
<td>45</td>
<td>25</td>
</tr>
<tr>
<td>5</td>
<td>45</td>
<td>30</td>
</tr>
<tr>
<td>6</td>
<td>45</td>
<td>35</td>
</tr>
<tr>
<td>7</td>
<td>55</td>
<td>25</td>
</tr>
<tr>
<td>8</td>
<td>55</td>
<td>30</td>
</tr>
<tr>
<td>9</td>
<td>55</td>
<td>35</td>
</tr>
</tbody>
</table>

* Stirring current was measured to indicate the electromagnetic intensity.
Fig. 1 – Optical microstructure of AZ91 semi-solid alloy which stirred at the conditions of $I_s = 30$ (A) and $t_s = 25$ (s), then injected to step-die cavity. Step with the thickness of (a) 5 mm, (b) 7 mm and (c) 9 mm.

Fig. 2 – Optical microstructure of AZ91 semi-solid alloy which stirred at the conditions of $I_s = 40$ (A) and $t_s = 25$ (s), then injected to step-die cavity. Step with the thickness of (a) 5 mm, (b) 7 mm and (c) 9 mm.

using Clemex Vision image analysis software. The shape factor of globules was calculated from the following equation:

$$F = \frac{4\pi A}{P^2}.$$  (1)

where $A$ and $P$ are the area and the perimeter of the primary particles, respectively ($F = 1$: sphere; $F \to 0$: needle). The equivalent diameter of primary particles ($\alpha$-Mg) was calculated using the following equation:

$$D = 2\sqrt{\frac{A}{\pi}}.$$  (2)

where $A$ is the area [15]. For these measurements the magnification of OM images was selected in a way that at least 50 numbers of primary $\alpha$-Mg grains are presented. At least 3 specimens were processed for each investigation and the average value was chosen as the final result.

2.5. Micro-hardness

To determine the specimens micro-hardness, the Vickers micro-hardness (Buehler, 1600-6100, United Kingdom) was used and 25 g force at the 15 (s) dwell time was applied. The final value was the mean value of 5 number of micro-hardness measured for each step. AZ91 magnesium alloy is consists of primary magnesium particles ($\alpha$-Mg) and eutectic structure. Thus, the value of total hardness is calculated by measuring each phase hardness according to the following equation:

$$H_{\text{total}} = H_s f_s + H_{l} f_{l}, \quad f_s + f_{l} = 1,$$  (3)

where $H_{\text{total}}$ is the total value of hardness, $H_s$ is the primary phase hardness, $H_{l}$ is the eutectic structure hardness, $f_s$ is the solid phase fraction and $f_{l}$ is the liquid phase fraction [16].

3. Results and discussion

3.1. Microstructure

The typical optical microstructures of AZ91 magnesium semi-solid alloy under various conditions of EMS ($I_s$: stirring current intensity and $t_s$: stirring time) are presented in Figs. 1–4. As shown in Fig. 3, dendrite branches of $\alpha$-Mg (Mg rich phase) are separated from each other which was due to liquid phase existence. Owing to the rotation of solid particles in the melt, the roundness of these particles is increased. On the other hand, longer stirring time resulted in the growth of $\alpha$-Mg globules that is not considered as a desirable result. Dendritic branches are apparent in Fig. 2 which indicates that the electromagnetic field power was not enough to crush the dendritic arms and formation of globular particles.

Table 2 and Fig. 5(a)–(d) present the values and variations of shape factor in different stirring currents (electromagnetic intensity) and times at various step thicknesses, respectively.

By increasing the stirring current intensity at constant values of stirring time, the applied electromagnetic field intensity was increased, which resulted in the separation of more secondary dendrite arms. These arms act as nucleuses, so, more grains are formed and the shape factor was increased. At the constant value of current intensity, by increasing the amount of stirring time up to 30 (s), the sphericity increased, but after 30 (s) it was decreased, since long stirring time led to the crash of globules and agglomerated them to each other, so coarse and rosette-shaped particles were achieved. By increasing the thickness of step, the shape factor was decreased. This was
due to the fact that, when semi-solid slurry was injected into the mold, the step with less thickness will fill-in later. On the other hand, the grains which had more sphericity could be easily slipped on each other in the liquid phase of slurry. These grains flow faster than the grains with less sphericity. Thus, the step with less thickness possessed globules with more sphericity.

Fig. 6(a) and (b) shows the variation of equivalent diameter with different step thicknesses at the various stirring current intensity and time. Since the cooling rate of melting is non-equilibrium, by increasing the stirring time, particles became coarse. As it is mentioned, long stirring time was led to crashing and joining of globules to each other. So, coarse and rosette-shape particles were achieved. Another significant point was that, the distribution of particles size became more uniform and fine particles were eliminated by increasing the stirring time. This was due to the particles growth based on Ostwald Ripening mechanism. Meanwhile, by increasing current intensity at constant values of stirring time, equivalent diameter of particles was decreased due to the finer crushed dendrites as a result of higher electromagnetic field. It seemed that with the improvement of shape factor, the average diameter of globules was increased. So, particles became more spherical at the expense of increasing globules diameter. At the stirring time of more than 30 s, shape factor was decreased and equivalent diameter of particles was increased. The equivalent diameter of globules in a step with 5 mm thickness was the smallest and in a step with 9 mm thickness was bigger than the others. This was due to the cooling rate which was the highest for step with 5 mm thickness and lowest for step with 9 mm thickness.

Fig. 7(a) and (b) presents the variation of solid fraction against different stirring currents and times. By increasing the stirring current, stirring agitating was carried-out more drastically and fluid temperature reduced more rapidly. So, the reduction of temperature was more significant at higher currents subsequently the value of solid fraction was increased. By increasing the stirring time, the temperature of fluid decreased and let the solid particles growth in longer times. Thus, the solid fraction was increased.

<p>| Table 2 – The shape factor of step samples in various conditions of ( I_s ) and ( t_s ). |
|----------------------------------|----------------------------------|----------------------------------|</p>
<table>
<thead>
<tr>
<th>Sample</th>
<th>5 mm thickness of step</th>
<th>7 mm thickness of step</th>
<th>9 mm thickness of step</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ref.</td>
<td>0.32</td>
<td>0.31</td>
</tr>
<tr>
<td>2</td>
<td>30 A – 25 s</td>
<td>0.38</td>
<td>0.36</td>
</tr>
<tr>
<td>3</td>
<td>30 A – 30 s</td>
<td>0.44</td>
<td>0.44</td>
</tr>
<tr>
<td>4</td>
<td>30 A – 35 s</td>
<td>0.41</td>
<td>0.40</td>
</tr>
<tr>
<td>5</td>
<td>40 A – 25 s</td>
<td>0.51</td>
<td>0.48</td>
</tr>
<tr>
<td>6</td>
<td>40 A – 30 s</td>
<td>0.70</td>
<td>0.69</td>
</tr>
<tr>
<td>7</td>
<td>40 A – 35 s</td>
<td>0.62</td>
<td>0.57</td>
</tr>
<tr>
<td>8</td>
<td>55 A – 25 s</td>
<td>0.75</td>
<td>0.73</td>
</tr>
<tr>
<td>9</td>
<td>55 A – 30 s</td>
<td>0.83</td>
<td>0.85</td>
</tr>
<tr>
<td>10</td>
<td>55 A – 35 s</td>
<td>0.80</td>
<td>0.79</td>
</tr>
</tbody>
</table>
Fig. 5 – The shape factor of samples in various conditions of stirring according to (a) different current intensity for 5 mm thickness of step (b) different stirring time for 5 mm thickness of step, (c) different thickness of steps at $t_s = 25$ (s) with different amounts of $I_s$ and (d) different thickness of steps at $I_s = 40$ (A) with different amounts of $t_s$.

Fig. 6 – Equivalent diameter of globules in different step thickness at (a) $t_s = 30$ (s) with various amounts of $I_s$ and (b) $I_s = 55$ (A) with various amounts of $t_s$.

According to the above mentioned results, to obtain the best globular structure, the optimum values of $t_s$ and $I_s$ was achieved at 30 (s) and 55 (A), respectively. Fig. 8(a) illustrates the as-cast (reference sample) dendritic microstructure of AZ91 magnesium alloy and Fig. 8(b) shows the globular microstructure of the rheocast AZ91 alloy which was fabricated in EM-stirrer under optimal conditions. Both structures had primary rich solid particles of Mg surrounding by eutectic matrix, but had a remarkable difference in the size and shape of particles. Stirring process and creation of turbulence in the slurry resulted in the crash of primary particles through breaking dendrites. By using AEM-stirrer, the growth
Fig. 7 – Solid fraction of samples in different step thicknesses at (a) $t_s = 30$ (s) with various amounts of $I_s$ and (b) $I_s = 40$ (A) with various amounts of $t_s$.

Fig. 8 – Microstructure of (a) cast AZ91 magnesium alloy and (b) AZ91 alloy which was fabricated in electromagnetic stirrer in the optimal conditions and then rheocasted.
of primary dendritic arms is stopped or equaled with the growth of secondary dendrites, which led to the production of the much more rosette-shaped dendrites from primary phase, as Mao et al. reported in their study [17].

XRD analysis of rheocasted AZ91 semi-solid alloy (Fig. 9) showed that, it consisted of α-Mg, Mg₁₇Al₁₂ and Al₂Ca phases. Three different phases were identified in the SEM micrograph of AZ91 semi-solid alloy as illustrated in Fig. 10. From the XRD analysis, and morphology of area A, it could be concluded that this area was the primary spherical α-Mg phase. The lamellar β-phase (Mg₁₇Al₁₂) was manifested in the form of separated islands in the grain-boundaries because the volume percentage of β-eutectic structure was much less than α-phase. After solidification process, due to the microscopic separation, β-phase surrounding fields and grain-boundaries had much more concentration of Al atoms. Probably, this over-saturated regions of Al would supply the necessary motivated force to create β-lamellar phase after solidification. Mg₁₇Al₁₂ intermetallic compound in AZ91 alloy may be changed to Mg₁₁₇.₅Al₁₁₅Zn₁₅ or Mg₁₁₇(Al,Zn)₁₂ compounds due to the presence of Zn [18,19]. XRD analysis revealed that area B in the optimal sample was Mg₁₁₇.₅Al₁₁₅Zn₁₅ phase. Islam et al. investigated the white regions in eutectic packets and described that rich particles of Al and Ca play a key role in the formation of primary α-Mg phase, thus, these particles acted as places for nucleation and growth of magnesium [20]. XRD analysis showed that this area (C in Fig. 10.) was Al₂Ca phase.

3.2. **Micro-hardness of step-die cavity samples**

Fig. 11(a) and (b) shows the variation of step-die cavity samples micro-hardness at the various conditions of stirring current and time. The results demonstrated that the thickness of the steps is an impressive factor in obtaining the micro-hardness value. Generally, the hardness of eutectic structure was higher than primary α-phase. By reducing the thickness of the steps, the solid fraction was decreased, which led to the enhancement of eutectic structure amount. So, the micro-hardness value was increased. On the other hand, by decreasing the thickness, cooling rate was increased. Thus, two important parameters which resulted in enhancement of total micro-hardness were: increasing the amount of eutectic structure...
(or reducing the value of solid fraction) and enhancement of cooling rate. For example, for a sample which stirred for 30 (s) under 55 (A) current intensity, for a step with 9 mm thickness, the hardness of primary α-phase, eutectic structure and total hardness were 61.4, 137 and 91.5 Vickers, respectively. However, for a step with 5 mm thickness, these amounts were 65.72, 143 and 102.4 Vickers, respectively. Finally, as can be seen from Fig. 11, the hardness of globular samples was much more than the hardness of dendritic structures.

4. Conclusion

By increasing the stirring current intensity, the shape factor of globules was enhanced and the equivalent diameter of globules was decreased. By increasing the stirring time up to 30 (s), the sphericity increased, but after 30 (s) it was decreased, however the solid fraction was increased. According to the achieved results, to obtain the best globular structure, the optimal values of \( I_0 \) and \( t_0 \) and were 55 (A) and 30 (s), respectively. By reducing the thickness of the steps, the value of micro-hardness was increased. Also, it can be found that, globular structures acquired much more hardness than the dendritic samples. X-ray analysis and SEM micrograph were showed that rheocasted AZ91 semi-solid alloy consisted of α-Mg, Mg17Al12 and Al2Ca phases.

Conflicts of interest

The authors declare no conflicts of interest.

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REFERENCES


Fig. 11 – The step-die cavity samples micro-hardness (a) at \( t_0 = 40 \) (A) with various stirring times and (b) at \( t_0 = 30 \) (s) with various stirring currents.

