Effect of Temperature Ranges of Alternating Current Imposition on Modification of Primary Mg₂Si Crystals in Hypereutectic Mg-Si Alloy

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An alternating current (AC) of 60 A with a frequency of 1 kHz was imposed on the hypereutectic Mg-4.8 mass% Si melt during solidification in order to modify the primary Mg₂Si crystals. The liquidus and eutectic temperatures of the Mg-4.8 mass% Si alloy are 761°C and 638°C, respectively. In order to investigate the effect of temperature ranges with application of AC on the modification of the primary Mg₂Si crystals, six temperature ranges from 770°C to 740, 700 and 630°C, and from 700°C to 680, 650 and 630°C were adopted. The temperature ranges examined had an obvious influence on the modification of the primary Mg₂Si crystals. For a starting temperature of 770°C, the average size of the primary Mg₂Si crystals could be significantly reduced with further decrease in the ending temperature to 700 and 630°C, with agglomeration of the refined primary Mg₂Si crystals in these two samples was observed. For a starting temperature of 700°C, the average sizes of the primary Mg₂Si crystals could also be reduced, although no obvious agglomeration of the refined primary Mg₂Si crystals was observed. The sample treated in the temperature range between 700 and 630°C had primary Mg₂Si crystals with the lowest average size and the highest uniformity of size. [doi:10.2320/matertrans.MRA2008366]

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1. Introduction

Magnesium alloys, the lightest structural metal materials, have been given much attention for applications in the automobile industry during the past two decades, in order to reduce fuel consumption and lower exhaust gas emissions.^{1,2)} Among the broad range of magnesium alloys, those containing Mg₂Si particles have excellent creep resistance and have been extensively studied,²⁻⁴⁾ because Mg₂Si exhibits a high melting temperature, high hardness and high compression strength.⁵⁾ In particular, the hypereutectic Mg-Si alloys have high potential as structural materials for elevated temperature applications.²⁾ However, the hypereutectic Mg-Si alloys prepared by conventional ingot metallurgy processes have very low ductility and strength, due to the large primary Mg₂Si crystal size and the brittle eutectic phase.⁶⁾ Therefore, refinement of the primary Mg₂Si crystals is very important to achieve the desired mechanical properties of hypereutectic Mg-Si alloys.

For hypereutectic Mg-Si alloys prepared by ingot metallurgy processes, the microstructures were traditionally refined by the addition of refiners, such as the rare earth element yttrium⁷⁾ and the boron-containing compound KBF₄.⁸⁾ However, the material compositions become complex upon such additions, making recycling of the materials difficult. Compared with the addition of refiners, a route for microstructure refinement induced by electromagnetic vibration (EMV) has an excellent advantage in that there is no change in the composition of the treated alloys. It has long been established that the EMV technique is a very effective route to refine the microstructures of various metals prepared by ingot metallurgy processes.⁹⁻²²⁾

Unfortunately, few studies have been performed on modification of the primary and eutectic Mg₂Si crystals in

primary Mg-Si alloys by EMV. In a preliminary study,²³⁾ we showed that the Mg₂Si crystals in a hypereutectic Mg-Si alloy could be effectively refined by weak EMV induced by imposing an alternating current (AC) with a high frequency of 1 kHz. In the present study, AC was imposed on the hypereutectic Mg-Si melt over different temperature ranges to clarify the correlation between the temperature range during application of the AC and the effect on modification of the primary Mg₂Si crystals.

2. Experimental Procedure

The hypereutectic Mg-Si alloy used in the present study was the same as that used in the previous study,²³⁾ in which the silicon content was approximately 4.8 mass%, and the liquidus and eutectic temperatures were determined as 761 and 638°C, respectively.

The samples treated by application of AC were prepared as follows. Approximately 25 g of the hypereutectic Mg-Si alloy was melted at 800°C in a mild steel crucible using an electric resistance furnace under a protective flux cover (45 mass% MgCl₂, 35 mass% KCl, 5 mass% CaF₂, 15 mass% NaCl). The melt was poured into an Al₂O₃ tube (21 mm inner diameter, 25 mm outer diameter, 70 mm length) that was preheated to 700°C using a small in-house built electric resistance furnace. The Al₂O₃ tube was held with a clamp fixed on a bracket, as shown in Fig. 1.

After the melt was poured into the Al₂O₃ tube, a couple of tungsten electrodes (3 mm diameter) were quickly inserted into the melt. The distance between the two tungsten electrodes was 12 mm. The tungsten electrodes were covered with an Al₂O₃ tube (3 mm inner diameter, 5 mm outer diameter, 60 mm length). The end of tungsten electrodes with a length of 5 mm was not covered, so as to apply AC into the melt. The distance between the end of the tungsten electrodes and the bottom of the Al₂O₃ tube was 10 mm. A

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Table 1 Statistical results of the sizes of the primary Mg₂Si crystals in the hypereutectic Mg-Si alloy treated by imposing the alternating current in the different temperature ranges.

Process No.	Temperature ranges of the alternative current imposition	Statistical average size of primary Mg ₂ Si crystals (μm)	Standard deviation (μm)	Total ratio of Mg ₂ Si crystals with sizes over than 400 μm
1	Without alternative current treatment	403	241	40.0%
2	770°C~740°C	322	153	24.0%
3	770°C~700°C	237	92	6.0%
4	770°C~630°C	202	96	4.0%
5	700°C~680°C	220	89	4.5%
6	700°C~650°C	199	79	2.5%
7	700°C~630°C	196	68	0%

Fig. 1 Schematic view of the experimental apparatus.

K-type thermocouple was fixed to one of the two tungsten electrodes and the temperature of the melt was recorded using a digital recorder (Keyence, GR-3500).

The small in-house built electric resistance furnace was turned off after the temperature of the melt was decreased to approximately 770°C. A brick (as shown in Fig. 1) was then moved away and the small furnace was removed. The hypereutectic Mg-Si melt contained in the Al₂O₃ pipe was then air-cooled, with application of AC at 60 A with a frequency of 1 kHz over the different temperature ranges. Six temperature ranges (process Nos. 2 to 7, Table 1) were examined to investigate the correlation between the duration of AC and the effect of modification on the primary Mg₂Si crystals. For Nos. 2 to 4, the starting temperature of AC application was fixed to 770°C with ending temperatures of 740, 700, and 630°C, respectively, and for Nos. 5 to 7, the starting temperature was fixed to 700°C with ending temperatures of 680, 650, and 630°C, respectively. Process No. 1 in Table 1 indicates the sample prepared without AC treatment.

After solidification, the cylindrical ingots were cut longitudinally along the middle plane parallel to the

Fig. 2 Observed area in the sample (unit: mm).

electrodes. They were then cut at the horizontal plane 20 mm from the bottom of the ingots. Samples for microstructural observations were prepared by a standard procedure with final polishing using a 0.05 μm alumina suspension. The samples were then etched with 3 vol% HF solution for 1 min. The etched samples were observed using a scanning electron microscope (SEM; Keyence, VE-7800) under different magnifications. The middle area between the two tungsten electrodes was selected for SEM observations, as shown in Fig. 2. The size of the observed area was 15 × 15 mm².

To evaluate the effect of AC, the sizes of the primary Mg₂Si crystals for all samples were measured from SEM images with 40× magnification, using the longest length of the primary trunk of the dendritic Mg₂Si crystals. All Mg₂Si crystals present in one SEM image area were measured. Mg₂Si crystals from other SEM images were also measured until 200 primary Mg₂Si crystals were obtained for every sample, and the 200 data sizes were then analyzed using statistical methods. The average values and standard deviations obtained for the 200 primary Mg₂Si crystals of each sample were then used to evaluate the effect of AC on the primary Mg₂Si crystal in the hypereutectic Mg-Si alloy.

3. Results

3.1 SEM observations of primary Mg₂Si crystals

To clearly understand the distributions of refined primary

Fig. 3 Low magnification (20 $\times$) SEM images of the hypereutectic Mg-Si alloy treated through process No. 1 (a), process No. 2 (b), process No. 3 (c), process No. 4 (d) and process No. 7 (e).

Mg₂Si crystals, low magnification (20 $\times$) SEM images of the hypereutectic Mg-Si alloy treated through process Nos. 1, 2, 3, 4 and 7 were observed, and are shown in Fig. 3. Large amounts of long and coarse dendritic primary Mg₂Si crystals were present in the sample without AC treatment, as shown in Fig. 3(a). The features of the long and coarse dendritic primary Mg₂Si crystals under high magnification are discussed later with regard to their formation mechanisms. The primary Mg₂Si crystals were refined by application of AC, except for the No. 2 sample. For the No. 3 and 4 samples, it was interesting that some large primary Mg₂Si crystal-free areas could be observed, as indicated by the white lines in the Figs. 3(c) and 3(d). The refined Mg₂Si crystals were agglomerated locally in these two samples. However, for

the sample with a starting temperature of 700°C, large primary Mg₂Si crystal-free areas were not observed and the refined Mg₂Si crystals had relatively uniform distribution, as shown in Fig. 3(e).

To observe the features of the Mg₂Si crystals more clearly, a relatively high magnification SEM image of the sample without AC treatment was obtained, as shown in Fig. 4. Two typical morphologies can be observed for the primary Mg₂Si crystals in this sample. One is a coarse dendritic morphology, such as the crystals denoted by A and B. The other is a polygonal morphology, such as the crystals denoted by C.

Figure 5 shows 40 $\times$ SEM images of the hypereutectic Mg-Si alloys treated through process Nos. 2 to 4. For the sample treated through process No. 2, some coarse primary Mg₂Si

Fig. 4 Middle magnification ($40\times$) SEM image of the hypereutectic Mg-Si alloy without alternative current treatment.

crystals with complex dendritic morphology were observed; however, they were not observed in the samples treated through process Nos. 3 and 4. Compared with the sample without AC treatment, the primary Mg_2Si crystals in samples No. 3 and 4 were significantly refined, were non-uniformly distributed and agglomerated.

Figure 6 shows $40\times$ SEM images of the hypereutectic Mg-Si alloy treated through process Nos. 5 to 7. For these three samples, the primary Mg_2Si crystals were refined compared with the sample without AC treatment (Fig. 4). Small amounts of coarse primary Mg_2Si crystals with complex

dendritic morphology were observed only in the sample treated through process No. 5, such as the Mg_2Si crystal denoted by A in Fig. 6(a). Agglomerations of the refined primary Mg_2Si crystals were not observed in these three samples.

3.2 Statistical analysis of the primary Mg_2Si crystal sizes

Statistical histograms of the 200 data sets of primary Mg_2Si crystal sizes are shown in Figs. 7, 8 and 9 for all samples, and the results for average size and standard deviation are listed in Table 1. The total ratios of the numbers of coarse ($>400\mu\text{m}$) Mg_2Si crystals to the 200 primary Mg_2Si crystals for all samples are also listed in Table 1.

Figure 7 shows the statistical histogram of the primary Mg_2Si crystals sizes in the hypereutectic Mg-Si alloy without AC treatment. The size interval for counting in this histogram was $100\mu\text{m}$. For this sample, the primary Mg_2Si crystal sizes were distributed in a very wide range, with an average size and standard deviation of $403\mu\text{m}$ and $241\mu\text{m}$, respectively.

Figure 8 shows the statistical histograms for the primary Mg_2Si crystal sizes in the hypereutectic Mg-Si alloy treated through process Nos. 2 to 4. The size interval for counting in these histograms was $50\mu\text{m}$. For the sample treated through process No. 2, the primary Mg_2Si crystal sizes were distributed in a relatively wide range, as shown in Fig. 8(a). The average size and standard deviation were decreased to $322\mu\text{m}$ and $153\mu\text{m}$, respectively. For the sample treated through process No. 3, the distribution of primary Mg_2Si

Fig. 5 Middle magnification ($40\times$) SEM images of the hypereutectic Mg-Si alloy treated through process No. 2 (a), process No. 3 (b) and process No. 4 (c).

Fig. 6 Middle magnification (40 $\times$) SEM images of the hypereutectic Mg-Si alloy treated through process No. 5 (a), process No. 6 (b) and process No. 7 (c).

Fig. 7 Statistical histogram of the primary Mg₂Si particles sizes in the hypereutectic Mg-Si alloy without alternative current treatment. The size interval for counting was 100 μm.

crystal sizes was in a narrow range, as shown in Fig. 8(b). The average size and standard deviation were further decreased to approximately 237 and 92 μm, respectively. For the sample treated through process No. 4, the primary Mg₂Si crystal sizes were distributed in a same range as that of the No. 3 sample, as shown in Fig. 8(c). The average size was further decreased to 202 μm and the standard deviation was approximately 96 μm, which is almost the same as that of the No. 3 sample. For the two samples treated through process Nos. 3 and 4. The ratios of the primary Mg₂Si crystals with

sizes over 400 μm in these two samples were 6% and 4%, respectively. However, the total ratios of the primary Mg₂Si crystals with sizes over 400 μm amounted to 40% and 24% for the sample without AC treatment and the No. 2 sample, respectively.

Figure 9 shows the statistical histograms of the primary Mg₂Si crystals sizes in the hypereutectic Mg-Si alloy treated through process Nos. 5 to 7. The size interval for counting in these histograms was 50 μm. For the sample treated through process No. 5, the primary Mg₂Si crystal sizes were distributed in a same range as that of the No. 3 sample, as shown in Fig. 9(a). The statistical average size and standard deviation were 220 μm and 89 μm, respectively. For the sample treated through process No. 6, the distribution of primary Mg₂Si crystal sizes became narrower, as shown in Fig. 9(b). The statistical average size and standard deviation were decreased to 199 μm and 79 μm, respectively. For the sample treated through process No. 7, the primary Mg₂Si crystal sizes were distributed in the narrowest range in all samples, as shown in Fig. 9(c). The average size was 196 μm, which was almost the same as that of the sample treated through process No. 6, while the standard deviation was further decreased to 68 μm. Some coarse primary Mg₂Si crystals with sizes over than 400 μm were present in the two samples treated through process Nos. 5 and 6, and the total ratios of primary Mg₂Si crystals with sizes over 400 μm were 4.5% and 2.5%, respectively. However, coarse primary Mg₂Si crystals with sizes over than 400 μm were not observed in the sample treated through process No. 7.

Fig. 8 Statistical histograms of the primary Mg_2Si particles sizes in the hypereutectic Mg-Si alloy treated through process No. 2 (a), process No. 3 (b) and process No. 4 (c). The size intervals for counting in these three histograms were 50 μm .

Fig. 9 Statistical histogram of the primary Mg_2Si particles size in the hypereutectic Mg-Si alloy through process No. 5 (a), process No. 6 (b) and process No. 7 (c). The size intervals for counting in these three histograms were 50 μm .

The results show that the primary Mg_2Si crystals could be refined by application of AC into the hypereutectic Mg-Si melt during solidification. The temperature range for application of AC was a significant factor to determine the effect of modification on the primary Mg_2Si crystals. When the AC was applied from a high starting temperature of 770°C , the average sizes of the samples were reduced with the decrease in the ending temperature. Agglomeration of the refined primary Mg_2Si crystals occurred if the AC was turned off at the ending temperatures of 700 and 630°C . If the AC was applied from a starting temperature of 700°C , no obvious agglomeration of the refined primary Mg_2Si crystals was observed. The primary Mg_2Si crystals could be effectively modified, even though the sample was treated in the narrow temperature range between 700 and 680°C . The average sizes of the primary Mg_2Si crystals and their standard deviations could be further reduced with decrease in the ending temperature to 650 and 630°C . The lowest average size and highest uniformity of size were obtained when the hypereutectic Mg-Si melt was treated through process No. 7, i.e., application of AC in the temperature range between 700 and 630°C .

4. Discussion

For the present experimental conditions, the cooling curves for all samples were recorded. The effect of the AC on the cooling rate was negligible and the average cooling rates from 760 to 640°C were almost the same, at 1.9°C/s , for all samples. Details regarding the effect of AC on the cooling rate has been discussed previously.²³⁾

The primary Mg_2Si crystals began to nucleate and grow in the hypereutectic Mg-Si melt after the temperature decreased to less than the liquidus temperature of 761°C .²³⁾ The growth characteristics of the primary Mg_2Si crystals had a close relation with the cooling rate. In the studies performed by Ourfali *et al.*²⁴⁾ and Qin *et al.*,²⁵⁾ it was found that the morphologies of the primary Mg_2Si crystals changed from coarse equiaxed to dendritic over a critical velocity for the cooling rate. The crystal structure of the Mg_2Si is face centered cubic (FCC) and the dendrite arm grows along the preferential $[100]$ crystallographic direction.^{5,25)} For the sample without AC treatment, the morphologies of the primary Mg_2Si crystals are mainly characterized by dendrites (shown in Fig. 4), which implies that the solidification cooling rate was above the critical velocity, as reported by Ourfali *et al.*²⁴⁾ and Qin *et al.*²⁵⁾

To date, many studies have investigated the mechanisms for microstructure refinement caused by EMV, and some mechanisms have been proposed.^{9–14,17–22)} The refinement of solidified structures subjected to EMV is mainly determined by two factors. One is that EMV promotes the nucleation rate of primary crystals. The other is that EMV contributes to the fracture of the primary dendritic crystals. For example, the study performed by Huang *et al.*⁹⁾ showed that an optimum AC contributed to a significant increase in the nucleation rate. On the other hand, the dendritic primary phase could be fractured by EMV and the small fragmental particles of the primary phase could also act as nuclei for the growth of primary crystals.^{10,17)}

When the hypereutectic Mg-Si melt is treated by AC J , with frequency f , an alternating magnetic field B , with the same frequency is induced in the melt.^{16–19)} The alternating magnetic field then interacts with the electric current and induces an electromagnetic vibration (EMV) in the melt.^{16–19)} Mg_2Si crystals with complex dendritic morphologies are disintegrated at weak parts in the crystals by the EMV. For dendritic crystals, the weak parts are the base regions where secondary dendrites grow from, such as the primary trunk of dendrites, or third dendrites grow from the trunk of secondary dendrites.²⁶⁾ These base regions become gradually smaller if the melting point decreases, due to enrichment of the solutes rejected from the surfaces of the dendrites.²⁶⁾ Consequently, the primary Mg_2Si crystals are effectively modified and their sizes are significantly refined. For the three samples treated from the starting temperature of 700°C , the standard deviations and total ratios of Mg_2Si crystals with sizes over $400\mu\text{m}$ to the 200 primary Mg_2Si crystals were gradually decreased with the decrease in the ending temperature, which implies that modification of the primary Mg_2Si crystals caused by application of the AC continued until the eutectic reaction ended.

The thickness of the electromagnetic skin layer δ , was calculated to be approximately 8.7 mm when the frequency of the AC was 1 kHz . This means that the actual passage area of the AC is less than the total longitudinal section of the sample. For the hypereutectic Mg-Si melt containing primary Mg_2Si crystals, the electrical conductivity of the Mg_2Si crystals should be much lower than that of the melt, because the Mg_2Si phase is an n-type semiconductor,⁵⁾ and an Archimedes electromagnetic force is developed that acts on the Mg_2Si crystals to resist the motion caused by the EMV.^{13,27,28)} Therefore, it is possible that some primary Mg_2Si crystals are moved away by the Archimedes electromagnetic force to some regions where the AC does not flow. These primary Mg_2Si crystals are not fully modified by the EMV and they grow into large Mg_2Si crystals. The primary Mg_2Si crystals present in the regions where the AC did not flow were not considered in the present study. Further study is required to investigate the effect of EMV on the distributions of primary Mg_2Si crystals in the entire sample.

In the case of the higher starting temperature of 770°C , agglomeration of the refined primary Mg_2Si crystals was observed in the samples treated through process Nos. 3 and 4, as shown in Figs. 3(c) and 3(d). This might occur only when the AC is continuously imposed upon the hypereutectic Mg-Si melt until temperatures below 740°C . The melt containing Mg_2Si particles vibrates periodically and the direction of movement of the melt and the particles are opposite under the imposition of AC, due to the low electrical conductivity of solid Mg_2Si compared with that of the melt. For a particle, its motion velocity, which is driven by the Archimedes electromagnetic force, is mainly controlled by its diameter.^{27,28)} A larger particle has greater velocity.²⁷⁾ Particles with different diameters should continuously move in the same direction, due to the inertial force after the direction of the EMV changes. The distance that a particle moves to a position where the direction of the particle changes is also a function of its diameter.²⁸⁾ A larger particle will have a longer

Fig. 10 Mechanism of coagulation of particles under electromagnetic vibration.²⁷⁾

amplitude of movement, because a larger particle is subjected to more inertial force. It is therefore possible that particles with different diameters coagulate together, as shown in Fig. 10, which was proposed by Kameyama *et al.*²⁷⁾ Consequently, many primary Mg₂Si crystals that are coagulated together could be kept in certain zones of the melt, resulting in the formation of larger agglomerations, as shown in Figs. 3(c) and 3(d).

In the study of Kameyama *et al.*,²⁷⁾ the Al₂O₃ particles coagulated together in the Al-Cu melt by the effect of EMV and resulted in the increase of the Al₂O₃ particle size.²⁷⁾ In the present study, agglomerations of the primary Mg₂Si crystals occurred with fragmentation of the dendritic arms of the crystals. On the other hand, some large primary Mg₂Si crystals were moved by the Archimedes electromagnetic force to regions where the AC did not flow. Therefore, the effective refinement of primary Mg₂Si crystals was achieved in the present study, even though agglomeration of the refined Mg₂Si crystals occurred.

Agglomeration of the refined primary Mg₂Si crystals resulted in the formation of primary Mg₂Si crystal-free areas, as shown in Figs. 3(c) and 3(d). The reason why the primary Mg₂Si crystal-free areas were formed is not clear. After agglomeration of the Mg₂Si crystals, it can be reasonably inferred that the electrical conductivity in the regions containing agglomerations of Mg₂Si crystals is relatively lower than that of the pure melt. Small primary Mg₂Si crystals that are subsequently precipitated would be moved to regions with lower conductivity by the Archimedes electromagnetic force. Consequently, the conductivity would increase in regions where the primary Mg₂Si crystals were moved away from. The primary Mg₂Si crystals precipitated in these regions would be more easily moved away to the regions with lower conductivity. As a result, primary Mg₂Si crystal-free areas were formed, as shown in Figs. 3(c) and 3(d).

5. Conclusions

An alternating current (60 A, 1 kHz) was imposed on a hypereutectic Mg-4.8 mass% Si melt during solidification over a range of temperatures to examine the modification of the primary Mg₂Si crystals. The following conclusions were made:

(1) Primary Mg₂Si crystals could be effectively refined by imposing an alternating current on the hypereutectic Mg-Si melt during solidification. The temperature range during

imposition of the alternating current was a significant factor to determine the modification effect on the primary Mg₂Si crystals.

(2) When imposition of the alternating current was begun from a higher starting temperature of 770°C, the average sizes of the samples were significantly reduced with the decrease in the ending temperature to 700 and 630°C, and agglomeration of the refined primary Mg₂Si crystals occurred under these conditions.

(3) No obvious agglomeration of the refined primary Mg₂Si crystals was observed if imposition of the alternating current was begun from 700°C. The average sizes of primary Mg₂Si crystals and their standard deviations were reduced with the decrease in the ending temperature to 630°C. The lowest average sizes and the highest uniformity of size were obtained when the hypereutectic Mg-Si melt was treated in the temperature range between 700 and 630°C.

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