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Effects of alternating current imposition and alkaline earth elements on modification of primary Mg_2Si crystals in hypereutectic Mg-Si alloy

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Abstract: The effects of alternating current imposition and/or alkaline earth elements on modification of the primary Mg_2Si crystals in the hypereutectic Mg-Si alloy were investigated. An alternating current of 60 A with frequency of 1 kHz was applied into the hypereutectic Mg-Si melt which was alloyed with alkaline earth elements or not in the fixed temperature range from 700 to 630 ℃. The results show that the primary Mg_2Si crystals could be refined by imposing alternating current or adding alkaline elements. Compared with the samples treated by adding 0.4% Ca or 0.4% Sr, higher modification efficiency could be obtained for the samples treated by imposing alternating current. No further modification efficiency could be obtained for the samples treated by imposing alternating current combined with 0.4% Ca or 0.4% Sr addition.

Key words: Mg-Si alloys; primary Mg_2Si crystal; alternating current imposition; alkaline earth elements; refinement

1 Introduction

Magnesium alloys, the lightest metal structural materials, have been extensively paid attention to be applied in the automobile industry during the past two decades[1]. Creep resistance is a major requirement for use of magnesium alloys in automobile powertrain components[2]. Several kinds of magnesium alloys have been developed with higher creep resistance[2−6]. Among them, the magnesium alloys containing Mg_2Si have excellent creep resistance[2,6]. 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 ordinary ingot metallurgy process have very low ductility and strength due to the large primary Mg_2Si particle size and the brittle eutectic phase. It is well known that the refinement of microstructures is one of the effective routes to improve mechanical properties for metal products.

Some advanced processing techniques such as hot extrusion[6], rapid solidification[7], and mechanical alloying[8] have been applied to produce alloys with fine Mg_2Si particles uniformly dispersed in Mg-matrix. Compared with these techniques, the ingot metallurgy process is a more practical method, because it is commercially available at low production cost and can be accepted by the engineering community for general applications.

For the hypereutectic Mg-Si alloys prepared by ingot metallurgy process, their microstructures were traditionally refined by the addition of refiners[9−11]. Among them, alkaline earth elements of calcium and strontium are effective elements to modify the morphology of Mg_2Si crystals[2, 11−13]. In the recent studies performed by the present authors, the results showed that the primary Mg_2Si crystals could be effectively refined by imposing the alternating current with high frequency on the hypereutectic Mg-Si melt during solidification[14−15].

In the present study, the alternating current with high frequency was imposed on the hypereutectic Mg-Si melt which was alloyed with alkaline elements of calcium or strontium, in order to clarify whether higher refining efficiency of primary Mg_2Si crystals could be obtained for the hypereutectic Mg-Si alloy modified by the combination of alternating current imposition and

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alkaline earth elements.

2 Experimental

In the present study, the Mg-Si alloy with approximately 4.8% Si (mass fraction) was prepared in advance. To alloy the Mg-Si melt, the Mg-10%Ca (mass fraction) and Mg-10%Sr were used. The samples were treated by imposing the alternating current as follows. The hypereutectic Mg-Si alloy of about 25 g was melted at 800 ℃ and then the melt was poured into an Al_2O_3 tube which was preheated to 700 ℃ using a self-made small power electric resistance furnace. This Al_2O_3 tube was held with a clamp fixed on a bracket in advance, as shown in Fig.1. The size of the Al_2O_3 tube is 21 mm in inner diameter, 25 mm in outer diameter and 70 mm in height.

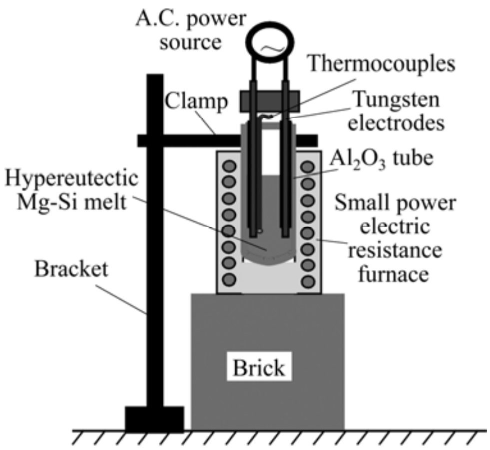

Fig.1 Schematic view of experimental apparatus

After the melt was poured into the Al_2O_3 tube, a couple of tungsten electrodes (3 mm in diameter) were inserted into the melt quickly. The distance between the two tungsten electrodes was 12 mm. The tungsten electrodes were covered using Al_2O_3 pipe with the size of 3 mm in inner diameter, 5 mm in outer diameter and 60 mm in height. The end part of the tungsten electrode with length of 5 mm was not covered to apply alternating current into the melt. The distance between the end of tungsten electrodes and the bottom of the Al_2O_3 tube was 10 mm. A couple of K-type thermocouples were fixed on one of the two tungsten electrodes, and the temperature of the melt was recorded using digital recorder. The temperature was begun to record from about 770 ℃. And then, the self-made electric resistance furnace was turned off and pulled out. The Al_2O_3 pipe filled with the hypereutectic Mg-Si melt was air-cooled. The alternating current of 60 A with frequency of 1 kHz was applied into the melt in a fixed temperature range from 700 to 630 ℃.

Six samples were prepared in the present study. They were the samples without any treatment, treated only by imposing the alternating current, treated only by adding 0.4% Ca or 0.4% Sr and the samples treated by imposing the alternating current combined with 0.4% Ca or 0.4% Sr.

The cylindrical ingots were cut longitudinally along the middle line parallel to the electrodes. Then, metallographic samples were cut at the position that was 20 mm from the bottom of the ingots. The samples for microstructure observation were prepared using a standard procedure with a final polishing with 0.05 μm alumina suspension. After that, the samples were etched with 3% HF (volume fraction) solution for 1 min. The etched samples were observed by a scanning electron microscope(SEM) (Keyence, VE−7800). The middle area between the two tungsten electrodes was selected as SEM observation area. The size of the observed area was 15 mm×15 mm.

More than five pictures were taken for every sample from the observed area. In the present study, the length of primary trunk of the dendritic Mg_2Si crystal was measured as the size of Mg_2Si particle. All Mg_2Si particles existing in one picture taken from the observed area were measured. After that, the Mg_2Si particles in another picture were continuously measured till 200 primary Mg_2Si particles for every sample were measured. Then the 200 values of sizes were analyzed by statistical method. The average value and standard deviation for the 200 primary Mg_2Si particles were used to evaluate the effects of the alternating current imposition and/or alkaline elements on the modification of primary Mg_2Si crystal in the hypereutectic Mg-Si alloy.

3 Results

3.1 SEM observation of primary Mg_2Si crystals

Fig.2 shows the SEM images of the hypereutectic Mg-Si alloy treated by different routes. For the sample without any treatment, the primary Mg_2Si crystals present mainly complex dendritic morphologies, like the crystals denoted by A and B in Fig.2(a). In addition, the primary Mg_2Si crystals with polygonal morphologies could also be observed, like the crystals denoted by C.

However, the coarse dendritic primary Mg_2Si crystals could hardly be found when the sample was treated by imposing the alternating current with a constant current of 60 A and frequency of 1 kHz, as shown in Fig.2(b). For the samples treated by adding 0.4% Ca or 0.4% Sr, there were many primary Mg_2Si crystals with dendritic morphologies existing in these two samples. However, the primary trunks of the primary Mg_2Si crystals with dendritic morphologies were obviously shorter than those of the coarse primary Mg_2Si crystals in the sample without any treatment, as shown in

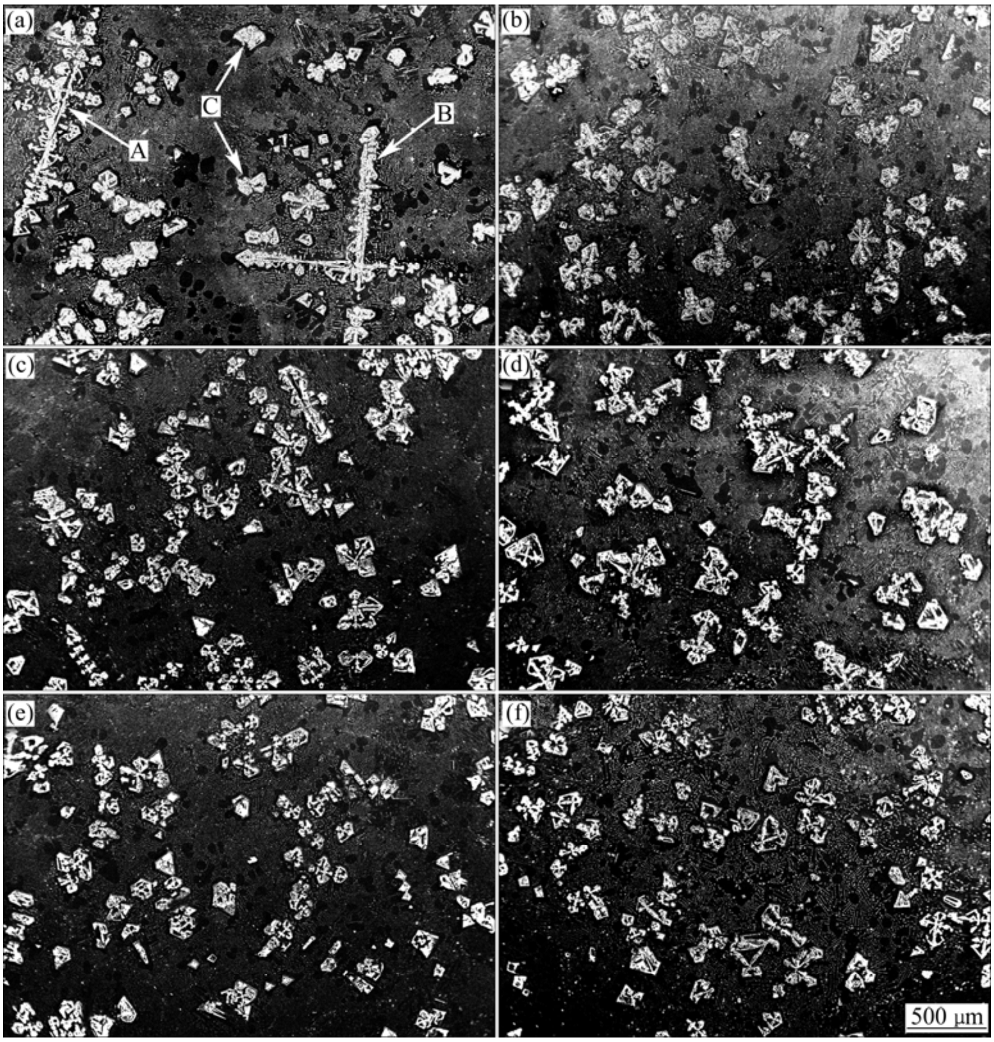

Fig.2 SEM images of hypereutectic Mg-Si alloy without treatment (a), treated by imposing alternating current (b), treated by adding 0.4% Ca (c) and 0.4% Sr (d), treated by imposing alternating current combined with 0.4% Ca addition (e) and 0.4% Sr addition (f)

Figs.2(c) and (d). Compared with the samples treated by adding 0.4% Ca or 0.4% Sr, the primary Mg_2Si crystals could be further refined for the samples treated by imposing the alternating current combined with 0.4% Ca or 0.4% Sr addition, as shown in Figs.2(e) and (f).

3.2 Statistical analysis results of sizes of primary Mg_2Si crystals

For every sample, the sizes of 200 primary Mg_2Si particles were measured. The statistical histograms of the primary Mg_2Si sizes are shown in Fig.3 for the six samples prepared in the present study. In these figures, the size intervals for counting for the sample without any treatment and for the other five samples are 100 μm and 50 μm, respectively. The results of statistical average size and standard deviation are listed in Table 1.

For the sample without any treatment, the sizes of primary Mg_2Si crystals distributed from 50 μm to 1 600 μm. Its statistical average size and standard deviation are 403 μm and 241 μm, respectively, as shown in Fig.3(a). For the sample treated by imposing the alternating current, the large primary Mg_2Si particles with sizes over 400 μm could hardly be found in the sample. Its distribution range of the Mg_2Si crystals sizes became narrow and the sizes located in the range from 50 μm to about 400 μm, as shown in Fig.3(b). Their statistical average sizes and standard deviations were decreased to 196 μm and 68 μm, respectively.

For the samples treated by adding 0.4% Ca or 0.4% Sr, the distribution ranges of the Mg_2Si crystals sizes were almost the same and the sizes located in the range from 50 μm to about 600 μm. The statistical average sizes were 233 μm and 245 μm for these two samples, respectively. However, their standard deviations were both 106 μm. Compared with the samples treated by adding 0.4% Ca or 0.4% Sr, the statistical average sizes and standard deviations were further decreased to about 188 μm and 67 μm after the samples were treated by imposing the alternating current combined with the 0.4% Ca or 0.4% Sr. However, it should be noted that the

Table 1 Statistical results of primary Mg_2Si sizes in hypereutectic Mg-4.8%Si alloy treated by different routes

Sample No.	Treated route	Statistical average size of primary Mg_2Si/μm	Standard deviation/μm
1	Without any treatment	403	241
2	Alternating current imposition	196	68
3	0.4% Ca addition	233	106
4	0.4% Sr addition	245	106
5	Alternating current imposition combined with 0.4% Ca addition	189	66
6	Alternating current imposition combined with 0.4% Sr addition	186	68

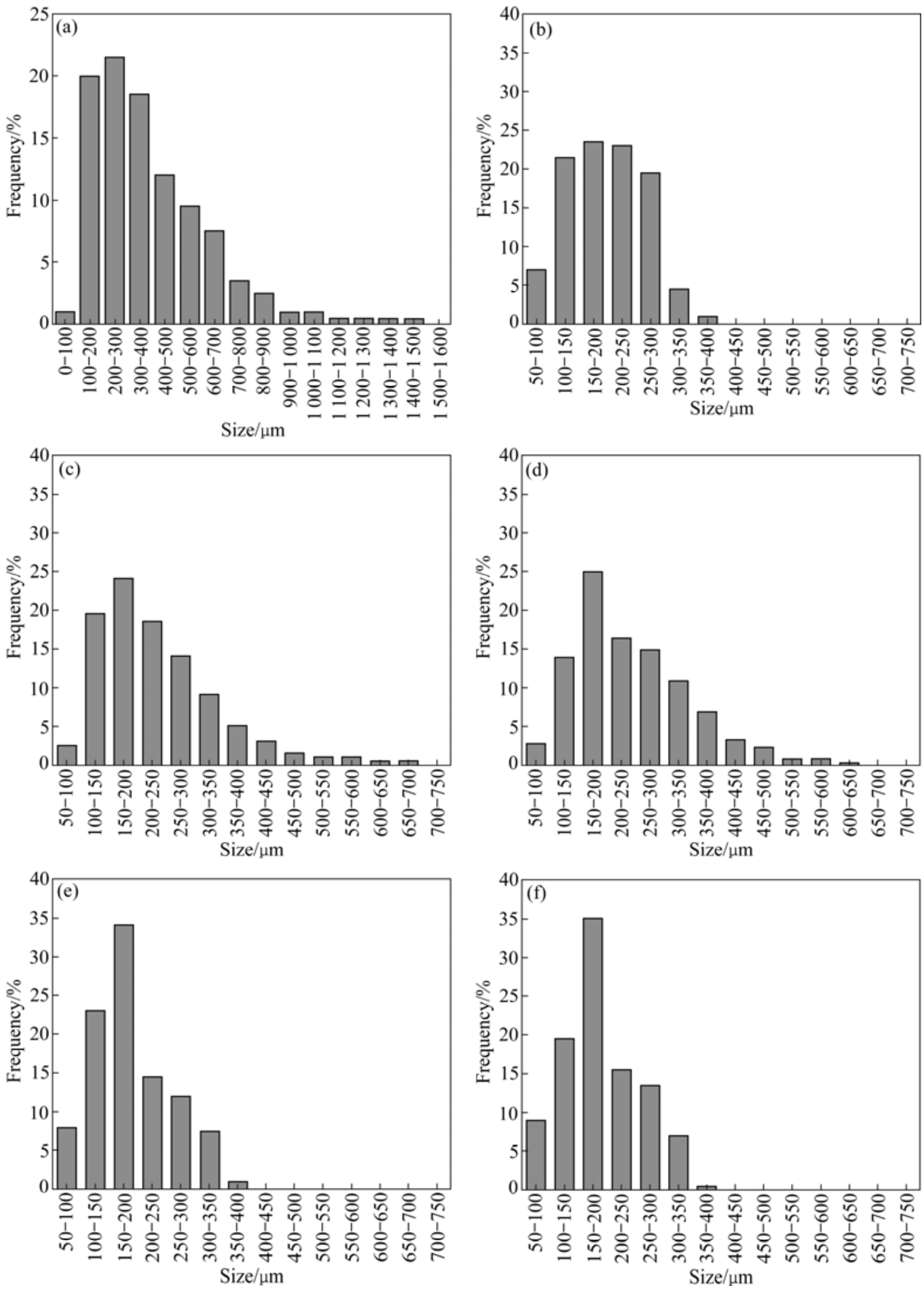

Fig.3 Statistical histograms of primary Mg_2Si particles size in hypereutectic Mg-Si alloy without treatment (a), treated by imposing alternating current (b), treated by adding 0.4% Ca (c) and 0.4% Sr (d), treated by imposing alternating current combined with 0.4% Ca addition (e) and 0.4% Sr addition (f)

statistical average sizes and standard deviations for these two samples had no obvious change for these two samples compared with the sample treated by imposing the alternating current.

Based on the above results, a conclusion could be drawn that primary Mg_2Si crystals in the hypereutectic Mg-Si alloys could be refined effectively by whether imposing the alternating current or adding alkaline elements. Compared with the samples treated by adding 0.4% Ca or 0.4% Sr, higher modification efficiency could be obtained for the sample treated by imposing the alternating current. However, no further modification efficiency could be obtained for the samples treated by imposing the alternating current combined with 0.4% Ca or 0.4% Sr addition.

4 Discussion

Under the present experimental conditions, the effect of alternating current imposition on the cooling rate could be negligible and the average cooling rates from 760 ℃ to 640 ℃ were almost the same with about 1.9 ℃/s for all samples[14]. After the temperature decreased to less than the liquidus temperature of 761 ℃, the primary Mg_2Si crystals began to nucleate and grow in the hypereutectic Mg-4.8%Si melt[14]. For the Mg_2Si crystal, its structure belongs to face centered cube(FCC) and its dendrite arm should grow along the preferential [100] crystallographic directions[16]. As a result, the morphologies of the primary Mg_2Si crystals in the sample without any treatment are mainly characterized by dendrites with complex morphologies, as shown in Fig.2(a).

When the hypereutectic Mg-Si melt was imposed by an alternating current with frequency f=1 kHz, an alternating magnetic field B with the same frequency could be induced in the melt[17−18]. Then, this alternating magnetic field interacted with the electric current itself and induced an electromagnetic vibration (EMV) in the melt[17−18]. Under this condition, the electromagnetic force(EMF) drove the conductor to vibrate periodically. Under the present experimental conditions, the electromagnetic force was evaluated as 126 N/m^3 when the current intensity was 60 A with frequency of 1 kHz.

When EMV is imposed upon the melt with a mushy zone, it was widely accepted that EMV contributes to the fracture of the primary dendritic crystals, resulting in the refinement of the solidified structure[18−19]. In the present study, the fragmentation of the primary Mg_2Si crystals caused by EMV was possibly responsible for the refinement caused by the alternating current imposition, which has been discussed in the previous studies [14−15]. The coarse Mg_2Si crystals with complex dendritic morphologies should be broken at some weak parts by EMV. For the dendritic crystals, the weak parts should be the root regions where secondary dendrites grow from primary trunk of dendrites or tertiary dendrites grow from the trunk of secondary dendrites[20]. These weak root regions were named as “shrinkage neck”[20].

After the alternating current was imposed on the hypereutectic melts containing primary Mg_2Si crystals, some coarse Mg_2Si crystals with complex dendritic morphology could be broken from the parts of “shrinkage necks” by inducing EMV into small crystals with polygonal morphology. As a result, the average size of the primary Mg_2Si crystals could be decreased after the hypereutectic Mg-Si melt was treated by imposing the alternating current, as shown in Fig.2(b).

For the effects of alkaline elements on the modification of Mg_2Si crystals, many investigations have been performed to disclose their modification mechanisms. It was usually accepted that the alkaline elements of Ca and Sr are interface active elements which can be adsorbed on the surfaces of Mg_2Si crystals during solidification[11−13]. As a result, the dendritic growth of the Mg_2Si crystals was effectively restricted. Therefore, compared with the sample without any treatment, the average sizes of the samples treated by adding 0.4% Ca or 0.4% Sr were also obviously decreased and the sizes located in a narrow range, as shown in Figs.3(c) and (d). The primary dendritic Mg_2Si crystals with short trunks were also broken after the alternating current was applied into the melt with 0.4% Ca or 0.4% Sr addition. Therefore, the average sizes of the samples treated by imposing alternating current combined with 0.4% Ca or 0.4% Sr addition were further decreased.

Compared with the coarse primary Mg_2Si crystals with complex dendritic morphologies in the sample without any treatment, the number of the weak parts, i.e. “shrinkage neck”, existed in the small dendritic primary Mg_2Si crystals in the samples treated by adding 0.4% Ca or 0.4% Sr should be small. Moreover, after the dendritic primary Mg_2Si crystals were broken into small crystals with polygonal morphology, these small primary Mg_2Si crystals could not be further broken by the weak EMV because no obvious weak parts existed in these crystals. Therefore, compared with the sample treated by imposing alternating current, no further modification efficiency could be obtained for the samples treated by imposing the alternating current combined with the alkaline elements additions.

5 Conclusions

1) The primary Mg_2Si crystals in the hypereutectic Mg-Si alloys could be refined effectively by whether

imposing alternating current or adding alkaline earth elements.

2) Compared with the samples treated by adding 0.4% Ca or 0.4% Sr, higher modification efficiency could be obtained for the sample treated by imposing alternating current. The average sizes of primary Mg_2Si particles were decreased by almost a half after the hypereutectic Mg-Si melt was treated by imposing alternating current.

3) Compared with the samples treated by imposing alternating current, no further modification efficiency could be obtained for the samples treated by imposing the alternating current combined with 0.4% Ca or 0.4% Sr addition.

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