Reducing iron content in molten aluminum by super-gravity segregation

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Abstract: Effects of super-gravity on the removal of iron from molten aluminum were investigated under condition of centrifugal furnace heating with a super-gravity coefficient of 400. Hypereutectic, eutectic and hypoeutectic Al-Fe alloys were used in the experiments with initial iron mass fraction of 2.88 %, 1.73 % and 0.99 % respectively. The alloy sample with covering protective slag was heated for 30 min at 800 °C, then the keeping the molten sample rotating at specified angular velocity until the sample was cooled to room temperature with a rate of 10 °C/min. The sample was cut into halves along the direction of super-gravity. One-half of the samples used for chemical analysis by ICP-OES at different locations, four 2mm-wide slices that evenly distributed along the direction of super-gravity were chipped out for chemical analysis. The other half was prepared for optical microscope.

The following conclusions were obtained from the experimental results for the samples under the super-gravity field. (1) The primary Al_3Fe particles were propelled to the bottom of crucible, the iron concentration of the hypereutectic， eutectic and hypoeutectic alloy increased along the direction of super-gravity. (2) The concentration ratios between the bottom and surface part reached 7.44, 2.53 and 3.21 respectively. (3) The iron content of eutectic was the limitation of hypoeutectic Al-Fe alloy super-gravity segregation under high cooling rate. (3) Super-gravity segregation can make the aluminum alloy with a higher purity by directional gathering primary α-Al.

Keywords: Super-gravity, Al-Fe alloy, Al_3Fe, segregation

1 Introduction

Iron is the most common and detrimental impurity in aluminum. Due to its low solid solubility (0.05*wt%*), most of iron form Fe-rich phases with other elements. These platelet-like or massive iron-rich intermetallic compounds have negative effects on the castability, the mechanical properties (particularly ductility), the corrosion resistance, and the machinability [1]. The techniques to reduce the detrimental effects of iron focus on two strategies, reducing iron content or neutralizing its negative effects [2]. It is known that the addition of other elements such as Mn, Cr and Ni can reduce the negative effect of iron by modifying the platelet-like morphology to a less harmful form. However, some additives are harmful to aluminum alloy[3]. The better method is to find an economically way to remove iron from the molten aluminum. Many researchers have proposed techniques for removing iron from aluminum, such as gravity sedimentation [4], composite purification [5] and electromagnetic separation [6,7]. Gravity sedimentation is an effective method to remove iron from aluminum, but it is difficult to adapt to the industrial production because of its low efficiency and non-continuous processing. Electromagnetic separation is a new method to remove primary iron-rich phases from aluminum melt, this method have not be applied to the practical production.

Super-gravity segregation is a new way that can remove iron from the aluminum rapidly and economically. Hiromi Matsubara et al.[8] had studied removal of the Fe from aluminum alloys by applying super-gravity during the solidification. The iron content decreased from 2.07 *wt%* to 0.27 *wt%* and the reduction ratio reached 87%. The results were attributed to the macro-segregation caused by super-gravity during the solidification. Lixin Zhao et al.[9] had used super-gravity to remove low-content impurities from aluminum. The macrosegregations of Fe and Si were remarkable along the direction of super gravity, and the concentration ratios between the bottom and surface part under super gravity of 1000 *g* (g is the normal gravitational acceleration with the value of 9.81 m/s^2) reached 4.05 and 2.80, respectively. Unfortunately the super-gravity segregation as a purification means of metals has not been systematically investigated.

As mentioned above, it is essential to investigate the super-gravity segregation in refining the metallic melts. In this work, the effect of super-gravity on the removal of iron from molten aluminum alloys were investigated the iron distribution and the purification effects were discussed.

2 Experimental

2.1 Materials preparation

Three typical alloys，namely hypereutectic, eutectic and hypoeutectic Al-Fe alloy were chosen as the experimental materials， and denoted by S-1， S-2 and S-3 respectively. The chemical composition of the alloys prepared for the investigation is shown in Table 1. Al-Fe alloys were prepared by encapsulating appropriate compositions of Al (purity: 99.9%) and Fe (purity: 99.99%) in alumina crucible followed by melting at 800 ^{o}C for several hours. The resulting ingots were cut into small pieces for the followed experiment. The concentration of Fe was tested by inductively coupled plasma optical emission spectrometer (ICP-OES).

Table 1. Chemical composition of Al-Fe Alloys (%, mass fraction)

Alloy	Fe	Al
S-1	2.88	bal.
S-2	1.73	bal.
S-3	0.99	bal.

2.2 Experimental apparatus

The super-gravity field was generated by a centrifugal furnace in this experiment. Figure 1 shows the schematic of the experiment apparatus, the furnace was fixed into the centrifugal rotor. The gravity coefficient G is calculated as the ratio of super-gravitational acceleration to normal-gravitational acceleration *via* Eq. (1).

$$G=\frac{\sqrt{g^2+(\omega^2 r)^2}}{g}=\frac{\sqrt{g^2+(\frac{N^2\pi^2 r}{900})^2}}{g} \qquad (1)$$

Where ω is the angular velocity, rad·s^{-1}; N is the rotating speed of the centrifugal, r·min^{-1}; r is the distance from the centrifugal axis to the sample, 0.25m; g is normal-gravitational acceleration 9.8m/s^2. At N=0, G is equal to 1.

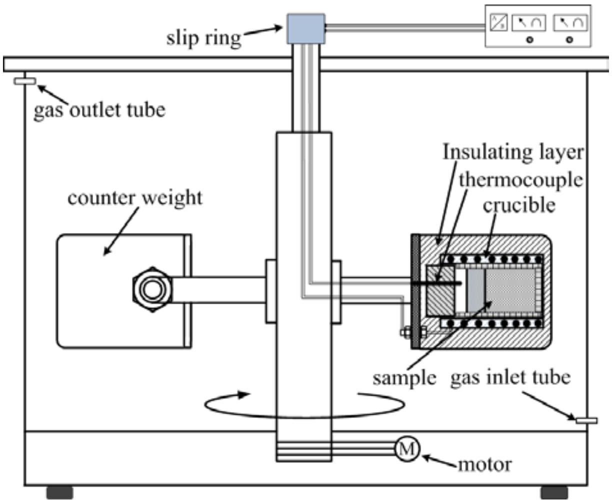

Figure 1 Schematic sketch of the experimental centrifugal apparatus

2.3 Super-gravity segregation

30g alloy was placed in alumina crucible and covered with 10g slag. The slag was the mixture of 45*wt%* sodium chloride and 55*wt%* potassium chloride, and both were analytical reagents. The alloy was heated for 30min at 800°C, then the centrifugal apparatus was started up and adjusted to the specified angular velocity. The apparatus was kept rotating until the sample was cooled to room temperature with a rate of 10 °C/min. This experiment was operated under the protection of nitrogen.

The sample was cut into halves along the direction of super-gravity. One-half of the samples used for chemical analysis by ICP-OES at different locations, four 2mm-wide slices that evenly distributed along the direction of super-gravity were chipped out for chemical analysis. Figure 2 shows the positions of the slices for Fe concentration analysis inside the sample. The other half was burnished, polished and then investigated by optical microscope.

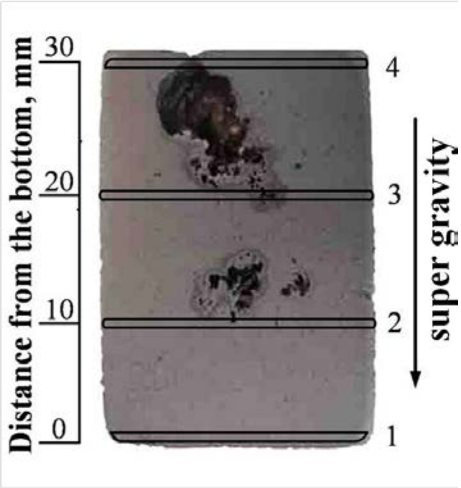

Figure 2 Positions for chemical analysis inside samples

3 Results and discussion

3.1 Super-gravity segregation of hypereutectic Al-Fe alloy

Figure 3 shows the structures of the S-1 samples solidified under different gravity field, a schematic of the macrostructure is provided on the right of each macrograph. The needle-like Al_3Fe particles evenly distribute in the sample that solidified under normal gravity (G=1). Unlike being solidified under normal gravity, an Al_3Fe-rich zone is locates near the sample bottom under super-gravity. Moreover, the size of the Al_3Fe particles increases with the increases of gravity.

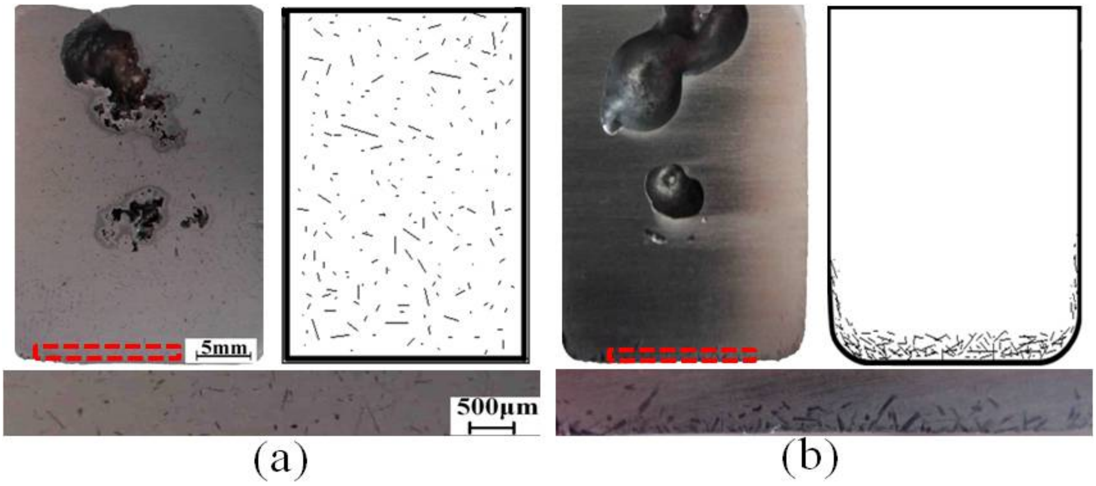

Figure 3 Macro structures and micro sketches of the S-1 samples solidified under different gravity field, (a) G=1, (b) G=400

The effect of super-gravity on the distribution of Fe from bottom to top inside samples is shown in figure 4. The distribution of Fe inside samples along the direction of super-gravity also indicates the enrichment of Al_3Fe particles. As the distance from the top of sample increases, the Fe content increases due to the super-gravity segregation of the

Al_3Fe particles. In contrast, there is no visible concentration difference under normal gravity. When G=400, the concentration of Fe in the sample top reduces to 1.34%, the concentration ratio of Fe between the bottom and the top reaches 7.44.

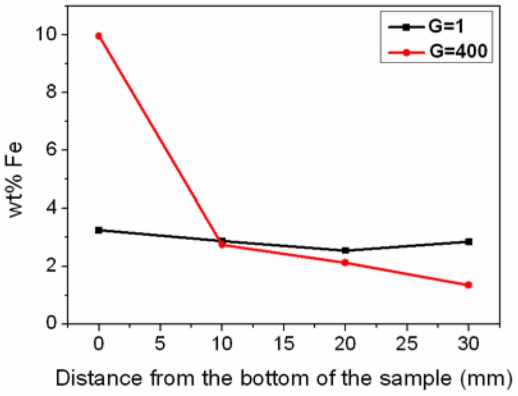

Figure 4 The distribution of Fe inside S-1 samples solidified under different gravity field

For Al-2.88%Fe hypereutectic alloy, the initial precipitated phase is Al_3Fe during solidification process. Due to its higher density (3896Kg/m^3[10]), the Al_3Fe particles were propelled to the bottom of crucible along the direction of gravity. Under normal gravity, sedimentation of primary phase need a relatively long time[4]. As the high cooling rate (10^{o}C/min) in this experiment, Al_3Fe particles did not have enough time for sufficient sedimentation. In contrast, super-gravity field largely intensify the gravity sedimentation, the super-gravity segregation got an ideal effect in shorter time.

3.2 Super-gravity segregation of eutectic Al-Fe alloy

Figure 5 shows the distribution of Fe inside eutectic S-2 samples solidified under different gravity field. There is no visible concentration difference under normal gravity observed. When G=400, the concentration increased from up to bottom along the direction of super-gravity. The concentration of Fe in the sample top reduced to 1.53%, the concentration ratio of Fe between the bottom and the top reaches 1.24. Compared with the effect of S-1 alloy super-gravity segregation (concentration ratio 7.44), the variation trend of concentration was not obvious.

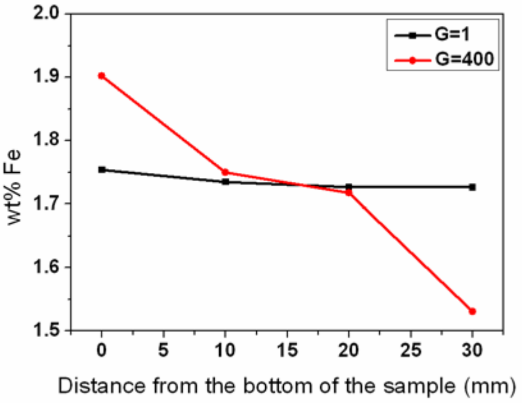

Figure 5 The distribution of Fe inside S-2 samples solidified under different gravity field

For eutectic alloy, when the experiment temperature reached the eutectic temperature during super-gravity solidification process, α-Al and Al_3Fe precipitated out. Then Al_3Fe particles (the denser solid) are propelled to the crucible bottom and α-Al particles float to the top. Because of high cooling rate, and the viscosity increased with the precipitate of solid particles. It seems difficult to segregation the latish precipitated α-Al and Al_3Fe particles. As can be seen from Figure 5, Fe concentration difference at the middle position of super-gravity sample is not visible. Thus the concentration ratio cannot obviously compared with the segregation of hypereutectic alloy.

Figure 6 shows the distribution of Fe inside S-1 and S-2 samples solidified under super-gravity field (G=400). No significant difference in the upper part concentration of Fe between two samples can be found there. As mentioned above, the primary phase can be effectively separated by super-gravity, but latish precipitated α-Al and Al_3Fe particles cannot be separated in a short time. The variation trend of concentration decreased in the upper part of S-1 sample. The concentration ratio between the position 2 and 4 was 2.04. So the eutectic Fe content could not be the limitation of hypoeutectic Al-Fe alloy super-gravity segregation under high cooling rate.

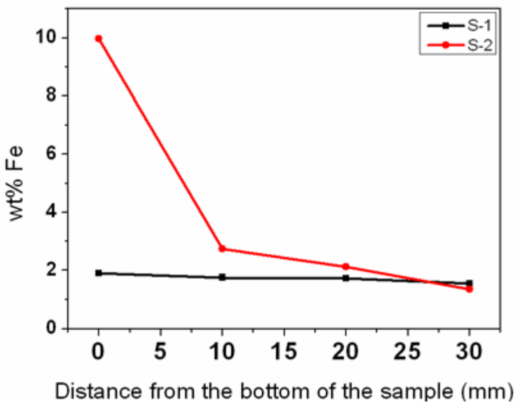

Figure 6 The distribution of Fe inside S-1&S-2 samples solidified under super-gravity field G=400

3.3 Super-gravity segregation of hypoeutectic Al-Fe alloy

The distribution of Fe inside hypoeutectic S-3 samples solidified under different gravity field is shown in Figure 7. Under the super-gravity field, the concentration of Fe in the sample top reduced to 0.49%, the concentration ratio of Fe between the bottom and the top was 2.53.

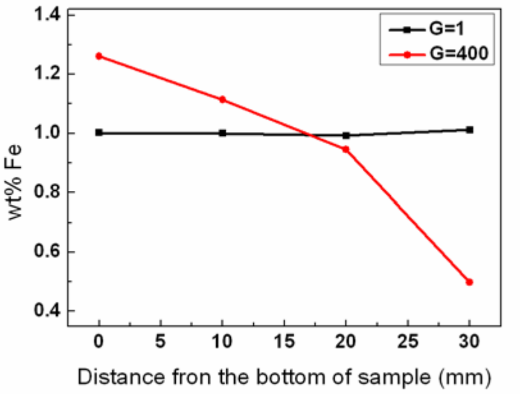

Figure 7 The distribution of Fe inside S-3 samples solidified under different gravity field

In Lixin Zhao's investigation [9], the results were attributed to the redistribution of impurities during solidification, super-gravity was a kind of directivity agitation. However, the concentration of Fe was high in the present experiment. The primary phase was α-Al during solidification, the α-Al particles were propelled to the top of crucible by super-gravity. The principle of this segregation mostly like the one used in fractional crystallization separation, super-gravity can greatly strengthen the separation effect of α-Al particles. As can be seen from the results of eutectic Al-Fe alloy super-gravity segregation, it is difficult to segregation the latish precipitated α-Al and Al_3Fe particles in this experiment conditions. So the gradient distribution of Fe should attribute to the migration of α-Al particles under super-gravity field. For hypoeutectic Al-Fe alloy, super-gravity segregation can reach a higher purity of aluminum by directional gathering primary α-Al.

4 Conclusions

The effect of super-gravity on the removal of iron from molten aluminum was investigated under condition of a centrifugal furnace heating with a super-gravity coefficient of 400. Three types of alloys, namely hypereutectic, eutectic and hypoeutectic Al-Fe alloy were used in the experiments. The iron contents in the alloys were as high as 2.88 mass%, 1.73 mass% and 0.99 mass% respectively.

1) Under the super-gravity field, the primary Al_3Fe particles were propelled to the bottom of crucible, the concentration of Fe in Al-2.88%Fe hypereutectic alloy increased along the direction of super-gravity, the concentration of Fe in the sample top reduced to 1.34%, and the concentration ratio between the bottom and surface part reached 7.44.

2) The concentration of Fe in the eutectic Al-Fe sample top reduced to 0.49%, the concentration ratio of Fe between the bottom and the top was 2.53. The iron content of eutectic was the limitation of hypoeutectic Al-Fe alloy super-gravity segregation under high cooling rate.

3) For hypoeutectic Al-0.99%Fe alloy, the concentration of Fe in the sample top reduced to 0.26%, and the concentration ratio between the bottom and surface part reached 3.21. Super-gravity segregation can get aluminum with a higher purity by directional gathering primary α-Al.

Acknowledgement

This work has been supported by the comprehensive strategic cooperation project between Guangdong Province and Chinese Academy of Sciences (2009B091300047), which are greatly appreciated.

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