Research on Grain Refinement in Hypoeutectic Al-Si Alloy during Solidification under an Alternating Electric Current Pulse

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Abstract: Experiments on transient directional solidification were carried out to study the columnar to equiaxed transition (CET) in Al-7Si alloy without and with an alternating electric current pulse (AECP). Without AECP, the macrostructure consists of typical columnar and equiaxed zones, separated by a near horizontal plane. As the AECP is applied during solidification, an additional fine equiaxed zone (FEZ) occurs in the as-cast macrostructure. From measured temperature profiles, cooling rate and temperature gradient are determined. It is found that CET occurs for a critical value of the cooling rate, which is observed to be about 0.14 K·s⁻¹ in the present investigation. Furthermore, the macrostructural observation with mold for embedding the mesh plate demonstrates that the major factor responsible for the formation of fine equiaxed grains is the detachment of crystal nuclei from the upper contact surface and the lateral wall. The detachment is in turn ascribed to electric current-associated free energy change (ΔG_e)-induced the driving force F.

Keywords: columnar to equiaxed transition; directional solidification; casting; grain refinement; aluminum alloys

1. Introduction

The mechanical properties of cast alloys can be enhanced by grain refinement or microstructure modification. In the past decades, various physical fields such as electric current pulse (ECP) [1–6], ultrasonic vibration [7–10] and electromagnetic stirring [11–15] have been applied during the solidification of alloys in order to refine the solidification structure. Recently, there has been much progress in ECP treatment, and much attention has been attracted to revealing the mechanism of ECP on grain refinement of alloys. Nakada et al. [16] found a transition from columnar to globular grains in Sn-15Pb alloys when a high ECP by discharging a capacitor bank was applied. They concluded that the strong local shear force could break off the dendrites. Barnak et al. [17] reported a reduction in the eutectic colony size of near-eutectic Pb-Sn alloys and pointed out that either a reduction in the energy difference between the liquid and solid states or an increase in the liquid-solid interfacial energy increased the nucleation rate. Liao et al. [18] considered the multiplication of crystal nuclei to be the main reason for grain refinement under ECP with up-down electrodes and also proposed that the skin effect enhanced the refining effect. Similar experiments with parallel electrodes were conducted by Li et al. [19] and they suggested that the shock wave on the free surface led to the formation of crystal rain and subsequent grain refinement. Obviously, ECP can induce melt flow, which
gives rise to temperature and concentration fluctuations in the mushy zone during the solidification process. Räbiger et al. [20] investigated the directional solidification of Al-7Si alloy under rectangular ECP with parallel electrodes and proposed that the formation of fine equiaxed grains was ascribed solely to the electro-vortex flow driven by Lorentz force. Based on the improved experiments, Zhang et al. [21] additionally indicated that the forced melt flow promoted dendrite fragmentation rates. From experimental investigation of the relevance of forced melt flow to the solidification structure of pure Al in the mold for embedding the wire mesh under an alternating electric current pulse (AECP) with up-down electrodes, our prior work [22] concluded that melt flow could lead to the columnar fragmentation and make the crystal nuclei on the mold wall fall off and drift in the liquid. Furthermore, Zhang et al. [23] pointed out that the columnar fragmentation resulted from a solutal remelting of dendrite side arms in pure Al with a low purity grade due to the remarkable melt flow. In summary, previous studies have proposed various explanations for ECP treatment, and the potential mechanism appears to be complex with respect to the variety of ECP parameters: Wave shape, current density and electrode configuration etc. Since current density is closely related to most of the current effects such as Joule heating, liquid–solid interfacial energy and electric current-associated free energy change ($\Delta G_e$) etc., it should be the dominant ECP parameter for grain refinement during solidification. However, the role of current effects depending on current density to grain refinement is not sufficiently studied. In the present work, the experimental investigation of the effect of a low AECP on the directionally solidified macrostructure of Al-7 wt.% Si alloy was carried out. As indicated above, electromagnetically driven convection plays an important role in grain refinement of solidified alloys [20,22,23]. In order to clarify the role of other current effects on grain refinement, a low enough AECP was selected to lead to a weak forced melt flow which hardly affected the solidification structure of alloys. Under the condition of the negligible convection, we tried to reveal the role of $\Delta G_e$ on grain refinement. It will provide a better understanding of the complicated refinement mechanism under AECP.

2. Experimental Procedures

The Al-7 wt.% Si (hereafter referred to as Al-7Si) alloy prepared by pure Al (99.9%) and Al-20 wt.% Si master alloy was solidified directionally from the bottom in a cylindrical ceramic mold using the experimental setup shown in Figure 1. The ceramic mold has an internal diameter of 34 mm, a height of 110 mm and a wall thickness of 3 mm. Its lateral wall is covered by the Cr20Ni80 resistance wire. The mold was put on a water-cooled copper chill, which was also used as the bottom electrode. Here, the lateral wall was heated to 889 K ($616 \degree C$) and held all the time to prevent a radial heat transfer in the mold. During casting experiments, the Al-7Si alloy was melted and overheated to 1023 K ($750 \degree C$) in an electric resistance furnace (Shanghai Shiyan Electric Furnace Company, Shanghai, China). After holding at 1023 K ($750 \degree C$) for 30 min, the molten metal was poured into the designed mold in which the filling height for the liquid in the mold was 110 mm, and then the top electrode (a rectangle copper block with a size of $50 \times 50 \times 45 \ mm^3$, which was welded to one end of the copper rod with a diameter of 8 mm) was dipped through the free surface into the melt up to an immersion depth of 1 mm, before which the top electrode was preheated to the same temperature as the melt to avoid the immediate formation of the solidification shell. Water was circulated through the cooling jacket keeping the copper chill during solidification process at a constant temperature of about 293 K ($20 \degree C$) and thus achieving transient directional bottom-up solidification. The AECP was generated by controlled transformer and silicon controlled rectifier (Zhuhai Fly-Eagle Electrical Appliance Company, Zhuhai, China). This device provided the frequency from 2-1600 Hz and the current amplitude from 0–10,000 A. Since our previous studies [22] demonstrated that effect of current effects such as Joule heating, melt flow arising from Lorentz force on grain refinement could be insignificant when the AECP with 990 A was applied, the lower effective value of current amplitude is required to clarify the role of $\Delta G_e$. In our study, the selected effective values of current amplitude were 0, 300, 400 and 600 A, respectively. Moreover, to obtain the uniform distribution of electric current in the as-cast
specimens [22,24], which will be helpful in discussing the contribution of the complex current effects, the low pulse frequency of 50 Hz was selected.

Figure 1. Schematic sketch of the experimental apparatus for directional solidification of Al-7Si alloy.

In the present experiments, the cooling of the sample and the AECP treatment were initiated at the same time. Temperature measurements were made within the mold cavity during solidification using an array of type-K thermocouples. Cooling curves were measured using five thermocouples arranged along the axis of the mold at fixed vertical positions of 17, 34, 51, 68 and 85 mm above the bottom of the mold. Each thermocouple was scanned with a frequency of 2.5 Hz.

The specimens were cut longitudinally along the central plane, and then ground on SiC paper for metallographic examination. One section was etched in a CuCl₂ solution, and then taken on a digital camera (Canon EOS D60, Tokyo, Japan) for macrostructure analysis. Here, the spliced macrostructure at the conjunction line is not real macrostructure in this position of original specimen, and an abrupt interrupt can be observed at the conjunction line for each specimen. However, this defect does not affect the macrostructural observations of columnar to equiaxed transition. Longitudinal sections are also cut in the equiaxed regions. Then, these sections were prepared for microscopy using standard techniques with the final polishing stage being produced by 0.5 μm diamond suspension (ZZSM, Zhengzhou, China). The microstructure was etched using 0.5%HF solution reagent, and then examined by optical microscopy (Carl ZEISS Axio Imager A1m, Göttingen, Germany).

3. Results and Discussion

Since the effect of AECP on temperature data was found to be qualitatively identical for the present applied current amplitude, it was decided to give the results obtained during solidification under AECP of 400 A priority in the following discussion. The comparison of cooling curves obtained from temperature measurements at five thermocouples arranged vertically along the axis of the sample without AECP and with AECP of 400 A are displayed in Figure 2. The horizontal dash lines are superimposed on the graphs to indicate the liquidus temperature $T_L$ and the eutectic temperature $T_E$. Without AECP treatment, the release of latent heat is just visible in the signal of the thermocouple at 85 mm as a plateau, both at primary phase growth at the liquidus temperature with some undercooling, as well as at the eutectic reaction also with some undercooling (Figure 2a). However, besides the thermocouple position 85 mm, the application of AECP leads to an obvious temperature plateau at the thermocouple position $z = 68$ mm (Figure 2b).
Figure 2. Cooling curves measured during solidification of Al-7Si: (a) Without alternating electric current pulse (AECP); (b) under AECP with 400 A and 50 Hz.

The data acquisition system, in which temperature is recorded with a frequency of 2.5 Hz, permits accurate determination of the slope of the experimental cooling curves. The cooling rate $dT/dt$ is calculated as the slope of the temperature curve immediately after the passing of the liquidus front at each thermocouple position. The equation given by Gandin [25] is used to evaluate the average temperature gradient $G_L$ ahead of the liquidus isotherm. The calculated results representing $dT/dt$ and $G_L$ without and with AECP are displayed in Figure 3a,b, respectively. This comparison reveals that the experimental values concerning $dT/dt$ and $G_L$ at the thermocouple position ($z = 17$ mm, 34 mm and 51 mm) are almost the same. However, $dT/dt$ and $G_L$ obviously decrease at the thermocouple position $z = 68$ mm when the AECP of 400 and 600 A were applied. As indicated by Eckert [26], a distinct reduction of $G_L$ was experimentally confirmed at all thermocouple positions as a feature for the forced convection, but was not consistent with the present experimental results. This indicates that the obvious melt flow driven by Lorentz force does not occur in our case and its influence on directional solidification process is negligible.
Figure 3. Cooling rate (a) and temperature gradient (b) as a function of position from metal/mold interface.

The macrostructure of directionally solidified Al-7Si alloy without and with AECP in the longitudinal section is shown in Figure 4. Without AECP, the lower part of the ingot presents the upward columnar grains and the upper fourth of the ingot, the coarse equiaxed one. The columnar to equiaxed transition (CET) is sharp and essentially on a near horizontal plane. The application of an AECP with 300 A generates a fine equiaxed zone (FEZ), the size of which is in the order of up to 10 mm between the minimum position of the coarse equiaxed grains and the maximum position of columnar grains (Figure 4b). The FEZ enlarges and the lower dotted line to the ingot bottom has a downward shift with increasing current amplitude. It indicates that the AECP significantly results in an occurrence of grain multiplication and a promotion of CET. Figure 5 presents the corresponding microstructure in the equiaxed zone. It can be observed that grains with dendrite morphology in FEZ are refined compared with those in the coarse equiaxed zone without AECP.
Figure 4. The as-cast macrostructure on the longitudinal section of Al-7Si alloy: (a) Without AECP; (b) under AECP with 300 A; (c) under AECP with 400 A; (d) under AECP with 600 A.

Figure 5. Microstructure of Al-7Si alloy in the equiaxed zone under the different AECP: (a) Without AECP; (b) under AECP with 300 A; (c) under AECP with 400 A; (d) under AECP with 600 A.
CET is affected by several parameters such as convection, $G_L$, $dT/dt$ and the growth velocity of the solidification front $V_L$. Here, $V_L$ can be obtained from the relationship between $G_L$ and $dT/dt$, i.e., $dT/dt = G_L V_L$. The above thermal parameters vary with time and position during directional solidification. As indicated above, the applied AECP has no visible effect on the thermal parameters in the columnar zone and the pre-existing equiaxed zone due to the negligible forced convection. Consequently, CET is not related to the present convection. It is noted that $dT/dt$ is essentially the same in the equiaxed zone without and with AECP treatment. It appears that a more realistic CET criterion should be based on the critical cooling rate of about 0.14 K·s$^{-1}$. This result is almost in agreement with previous critical cooling rate for Al-Si alloy, which are about 0.17 K·s$^{-1}$ [27].

The large number of crystal nuclei induced by AECP is the most important reason for the formation of FEZ. In order to clarify the mechanism for grain multiplication by AECP, it is essential to explain the origin of fine equiaxed grains.

The generation of fine equiaxed grains in the specimen as a result of the application of AECP is supposed to be complex in terms of the intrinsic effects of AECP. And thus, the following discussion focuses on the contribution of these effects. In the present study, the AECP was applied to the superheated melt until the end of solidification as soon as it was poured into the mold. The effect of AECP treatment with respect to the different solidification stages should be considered in detail. During the liquid phase stage above the liquidus temperature, there may be an inoculation effect which can improve the nucleation rate and refine the solidification structure [28]. However, the inoculation effect had not been observed when an AECP with 2070 A was applied to the solidification of Al-7Si alloy [24]. No doubt, the applied AECP, which is no more than 600 A, has no inoculation effect in this alloy. In general, the effect of ECP treatment during the nucleation stage includes the change in the nucleation activity energy, Joule heating and $\Delta G_e$. As was indicated in previous studies [29], the role of the change in the nucleation activity energy to the nucleation rate was obviously insignificant in the Al-7Si alloy under AECP with 990 A, and thus the corresponding contribution is not expected to be significant under the lower AECP. In addition, the contribution of Joule heating is also not expected to be prominent for the present current density being no more than 67 A·cm$^{-2}$ since Joule heating is almost negligible for the current density of ~120 A·cm$^{-2}$ in the Al-7Si alloy [20]. During the crystal growth stage, the forced melt flow induced by ECP can induce local fluctuations of both temperature and concentration in the mushy zone and then promote dendrite fragmentation rates, leading to grain multiplication [20,21]. However, the forced convection is insufficient to promote a solutal remelting of dendrite in our case. Thus, grain multiplication is likely to be the consequence of $\Delta G_e$. It is assumed that heterogeneous crystal nuclei appearing at the contact surface of the top electrode and the molten alloy will be detached by $\Delta G_e$ before the solidification shell formed, leading to the formation of FEZ.

In order to confirm this assumption, a series of casting experiments using sand mold for embedding the plate of stainless-steel mesh were performed. Since crystal nuclei can be prevented to transfer through the mesh plate [18,22,30], the comparison of solidification macrostructure resulting from different regions is considered to clarify the mechanism for grain multiplication by AECP.

As shown in Figure 6, three special casting experiments were designed to confirm where fine equiaxed grains originated from under the different AECP. For each experiment, a mesh plate was embedded in the center of the sand mold along the cross section to prevent crystal nuclei from the upper part of the specimen moving to the lower part of the specimen. The molten alloy with a superheat of 134 K (134 °C) was poured into the sand mold and no ECP was employed during the whole solidification process for Figure 6a. The AECP with 400 and 600 A was applied during the solidification process under the same pouring condition for Figure 6b,c, respectively. Figure 6 represents the corresponding longitudinal as-cast macrostructure of Al-7Si alloy. Without AECP, the solidification structures are coarse grains above and below the mesh plate. When the AECP with 400 A was applied, the number of equiaxed grains increases accompanied by a decrease of equiaxed grain size above the mesh plate, and the solidification structures are still coarse grains below the mesh plate compared with those without AECP (Figure 6b). As increasing AECP to 600 A, the equiaxed grains above the mesh plate and below
become much smaller, but the size of the equiaxed grains above the mesh is still obviously smaller than those below the mesh. Since the selected frequency is 50 Hz, the electric current uniformly passes through the specimen [24]. It implies that pulse current effects are significantly symmetric about the mesh plate in the present case. However, the difference of the solidification conditions above the mesh and below occurs at both surfaces of the upper and lower parts of the ingot. From macrostructural comparison, the most probable origin for refined grains is that a large number of crystal nuclei detach from the contact surface of the top electrode and the molten alloy and move freely in the liquid under AECP with 400 A. As increasing AECP to 600 A, the detachment of crystal nuclei occurs at the lateral wall besides the upper contact surface.

\[
G_e = -\frac{\mu}{8\pi} \int \frac{|j(r) \cdot j(r')|}{|r - r'|} drdr'
\]

(1)

where \( r \) and \( r' \) are two different positions in space, \( j(r) \) and \( j(r') \) are the current densities at position \( r \) and \( r' \), respectively, and \( \mu \) is the magnetic permeability. During nucleation stage, crystal nuclei which have different electrical conductivities from that of melt first appear at the lateral wall and the contact surface of the electrodes and the molten metal. The configuration of crystal nuclei affects the electric current distribution. Hence, various configurations of crystal nuclei correspond to the different free energy \( G_e \). When the crystal nucleus moves from the central axes of the melt towards the lateral wall of mold, the chemical free energy and interface free energy do not change. The total change of the

**Figure 6.** The as-cast macrostructure on the longitudinal section of Al-7Si alloy using sand mold embedded in a mesh plate placed along the cross section: (a) Without AECP; (b) under AECP with 400 A; (c) under AECP with 600 A.
system free energy is equal to $\Delta G_e$, i.e., the current density changes from $j_1(r)$ to $j_2(r')$. The associated free energy change $\Delta G_e$ can be expressed as [31–33]:

$$\Delta G_e = \frac{\mu}{8\pi} \int \int \frac{j_1(r)\cdot j_1(r') - j_2(r)\cdot j_2(r')}{|r-r'|} d^3r d^3r' = -\frac{\sigma_1 - \sigma_2}{2\sigma_1 + \sigma_2} k^2 V$$

(2)

where $\sigma_1$ and $\sigma_2$ are electric conductivities of the melt and crystal nuclei at about 889K (616 °C), respectively, $V$ is the volume of crystal nuclei and $k$ is a positive geometry factor. $\Delta G_e$ will be positive due to the electrical resistivity of crystal nuclei being less than that of the melt matrix and vice versa. Equation (2) has been used to explain the migration of electrically neutral particles in liquid toward the lateral surface from the center of suspension [34,35]. When the AECP passes through the sample during the nucleation stage, the system free energy is not minimum with respect to the initial configuration of crystal nuclei. The electrical conductivity of the molten alloy is approximately $4 \times 10^4$ S·cm$^{-1}$ [36] and that of primary $\alpha$-Al phase is approximately $10^3$ S·cm$^{-1}$ [37] at about 889 K. The value of crystal nuclei of $\alpha$-Al phase is 10 times more than that of the molten alloy. The current density inside crystal nuclei is more than that of the outside. In order to minimize the free energy associated with AECP, crystal nuclei are driven from original site to the center of ingot by a force $F$ from AECP. Here, the direction of the force is from the center of the mold to the lateral surface, perpendicular to that of the electric current and axial symmetrical for $\sigma_1 > \sigma_2$ [34,35]. The direction reverses for the present condition of $\sigma_1 < \sigma_2$. An approximated expression for the value of the driving force from the electric current to crystal nuclei is given as [34,35]:

$$F = -\frac{\sigma_1 - \sigma_2}{2\sigma_1 + \sigma_2} \frac{d}{f(d)} \mu f^2 V = \frac{\mu}{f(d)} \frac{d \Delta G_e}{k}$$

(3)

where $d$ is the distance from the center axis of the melt matrix along the radius and $f(d)$ increases monotonically but nonlinearly as $d$ increases. According to Equation (3), $F$ is proportional to $\Delta G_e$. It suggests that $F$ makes crystal nuclei detaching from the top contact surface and the lateral wall into the melt due to $\Delta G_e$.

According to the above experimental results and analysis, the refinement mechanism by the present AECP with up-down electrodes is schematically illustrated in Figure 7. Without ECP, the casting specimen is covered by coarse equiaxed grains (see Figure 6a), which is one of the typical casting structures. Firstly, crystal nuclei appear at, or close to, the sand mold wall. They rapidly grow into equiaxed grains. Then, new crystal nuclei form ahead of the solidification front due to the constitutional supercooling, which prevent the formation of a columnar zone. Finally, the solidification structures are coarse equiaxed grains in the specimen. As shown in Figure 7b, when the liquid alloy starts to solidify, many small grains having random orientations, are nucleated at the mold inner surface and the contact surface of the top electrode and molten alloy. The direction of the force $F$ imposed to crystal nuclei on the substrate of the contact surface of the top electrode and the molten alloy is perpendicular to the normal direction of contact surface and pointing towards the center. For crystal nuclei on the substrate of lateral wall, the difference is that the direction of imposed $F$ is parallel to the normal direction of the mold inner surface. Since the experiment result (see Figure 6b) suggests that grain refinement is ascribed to $F$ making crystal nuclei fall off from the upper surface, the detachment of crystal nuclei driven by $F$ parallel to the contact surface should be easier than that perpendicular to the contact surface. The detached crystal nuclei on the upper contact surface move down and drift in the undercooled melt under the action of $F$ and gravity, leading to the formation of fine equiaxed grains under AECP with 400 A. As the AECP increases to 600 A, the detachment of crystal nuclei on the lateral wall occurs.
4. Conclusions

The columnar to equiaxed transition was investigated in directional solidification of Al-7Si alloy without and with AECP treatment. The following conclusions can be drawn.

1. The cooling rate and the temperature gradient at the isotherm decrease from the chill face to the upper part of the casting without and with AECP. The present application of AECP has no visible effect on the above thermal parameters in the columnar zone and the pre-existing equiaxed zone due to the negligible forced convection. It was found that a realistic criterion should be based on a critical cooling rate encompassing tip growth rates and temperature gradients of about 0.14 K·s\(^{-1}\) in the present study. The columnar growth is expected to prevail throughout the casting for the cooling rate higher than the critical value.

2. Without AECP, the macrostructure consists of a columnar zone and a coarse equiaxed zone at the top in directionally solidified Al-7Si alloy. The CET occurs in a sharp plane rather than in a zone. The application of AECP with up-down electrodes induces the formation of the fine equiaxed zone.

3. The origin of fine equiaxed grains in Al-7Si alloy is analyzed in detail. From macrostructural observation with mold embedded in the mesh plate along the cross section, it confirms that the detachment of crystal nuclei from the contact surface of the top electrode and the molten metal and the lateral wall, leads to the formation of fine equiaxed grains. Here, Joule heating and the negligible melt flow have no obvious influence on grain refinement of alloy. The force \( F \) from electric current to crystal nuclei, which is proportional to electric current-associated free energy change \( \Delta G_e \), is the driving force for the detachment of heterogeneous crystal nuclei.

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