Structures and macrosegregation of a 2024 aluminum alloy fabricated by direct chill casting with double cooling field

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Abstract: Central region coarse grains and centerline segregation are common defects in aluminum ingots fabricated by direct chill (DC) casting. A double cooling field was introduced into the DC casting process to reduce these defects, whereby the external cooling was supplied by the mold and water jets, and intercooling was achieved by inserting a rod of the same alloy into the molten pool along the central axis of the ingot. Rather than forming a good metallurgical interface during solid-liquid compound casting, in the present work, the purpose of inserting the rod is to enforce internal cooling and consequently decrease the sump depth. Moreover, the insertion provides more nucleation sites with the unmolten α -Al particles. The structure and the macrosegregation of 2024 Al alloy ingots prepared by DC casting with and without the inserts were investigated. Results show that when the inserting position is 50 mm above the upper edge of the graphite ring, significant grain refinement in the central region of the ingot and a reduced centerline segregation are achieved.

Keywords: aluminum alloy; direct chill casting; double cooling field; grain refinement; macrosegregation

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1 Introduction

Large-size aluminum alloy ingots produced by traditional direct chill (DC) casting, especially the 2xxx and 7xxx series, often have defects such as inhomogeneous structure, macrosegregation, and hot cracks^[1-4]. Although adding grain refiners improves the quality of large-size ingots, it could lead to agglomeration of nucleating particles and fading effect^[5-6]. In addition, physical methods, including mechanical stirring^[7-8], ultrasonic vibration^[9], and electromagnetic stirring^[10], are not easy to apply in the DC casting process because of the expensive and specific equipment requirements.

Recently, several new methods have been developed by adding intercooling in the DC casting process. For instance, an in-mold cooler with a cooling end made of highly pure graphite can be inserted into the melt to absorb and transfer the heat during the liquid or gas flow in the cooler^[11-12]. Another way to enhance

intercooling is by inserting a smaller cold material of the same compositions into the melt during copper alloy DC casting^[13]. This method was also studied in the squeeze casting of aluminum alloys^[14-15] and die casting of magnesium alloys^[16]. However, the purpose of that method is to obtain an excellent metallurgical bonding between the insert and the melt. Our previous study found that inserting a cold 2024 Al alloy rod into the 2024 Al melt during mold casting can refine the grains^[17]. Eskin et al.^[18] confirmed the grain refinement effect of introduction the same-alloy rod into the melt in a standard TP-1 mold casting and achieved significant grain refinement. Therefore, it is of great use to refine grains in the DC casting process by inserting cold material into the melt.

In this study, a 2024 Al alloy rod was inserted into the 2024 Al alloy melt during hot-top DC casting, whose chemical compositions are Al-4.0wt.% Cu-1.6wt.% Mg-0.6wt.% Mn-0.12wt.% Fe-0.03wt.% Si. Compared with traditional DC casting with a single cooling field (SCF), the cold 2024 Al alloy rod provides another cooling field with its latent heat consumption while melting. The intercooling from the insert and the cooling from the mold and water jet are called a double cooling field (DCF), which are expected to decrease the

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temperature gradient from the surface to the center of the ingot and provide more nucleation sites. The solidification structures and macrosegregation of the ingots with SCF or DCF were examined, and the effects of the 2024 Al alloy insert on the grain refinement and segregation reduction were investigated.

2 Experimental procedure

Commercial 2024 Al alloy (without grain refiner) and 2024 Al alloy extrusion rods, as shown in Fig. 1, were used as ingot materials and inserts, respectively. The rods are 20 mm in diameter and 1,500 mm in length. Their surfaces were polished by 400 grit silicon carbide papers, treated by alkaline cleaning and acid pickling. The 2024 Al rods consist of typical fibrous

Fig. 1: Images of 2024 Al rod (a), ingot (b), and OM images of 2024 Al rod (c)

Fig. 2: Schematic view of DC casting process with DCF (a), and sampling positions for structure observation and composition testing (b, c)

Three transverse slices [Fig. 2(b)] were cut from the ingot processed with SCF or DCF, then, the slices were cut along the center plane. Samples for macro-corrosion [red area in Fig. 2(c)] were etched in 12wt.% NaOH water solution. A Sony A6000 digital camera was used to capture an image of the macrostructure. Samples for microstructure observation were cut along the radial direction of slices. These samples were polished using diamond suspensions and anodized under 20 V DC in 2.5wt.% HBF₄ water solution. An OLYMPUS-DX23 optical microscope (OM) was used to observe the microstructure. The average grain size was measured according to the standard of

structure, as shown in Fig. 1(c).

A schematic view of the DC casting process of a $\Phi 300$ mm 2024 Al ingot is shown in Fig. 2(a), and the OM image of the ingot is shown in Fig. 1(b). First, the 2024 aluminum alloy was melted in the resistance furnace followed by degassing, stirring, and slag removing. When the temperature of the melt stabilized at 730 °C, the melt was poured into the crystallizer. Then, the melt was cast at a speed of 65 mm·min⁻¹ under a cooling water flow rate of 150 L·min⁻¹. The casting process was performed in the same way as traditional DC casting. Then, the 2024 Al rod, with the protection of a thermal-insulation device, was lowered to Position 1 or 2 [Fig. 2(a)], the thermal-insulation device was fixed, and the rod was gradually inserted into the molten pool along the central axis of the hot-top. The cylindrical thermal-insulation device was used to prevent the rod from contacting the melt in the upper part of the hot-top. The feeding speed of the rod was 150 mm·min⁻¹. As shown in Fig. 2(a), Position 1 is on the central axis of the ingot and 50 mm above the upper edge of the graphite ring, and Position 2 is on the same level as the upper edge of the graphite ring. With the upper surface of the hot-top serving as the reference plane, two marking lines were cut on the outer surface of the thermal-insulation device according to the vertical distances between Position 1 or 2 and the upper surface of the hot-top, thus ensuring the thermal-insulation device can be actually lowered to Position 1 or 2.

ASTM E112-96, using the linear intercept method. A ZEISS Ultra Plus scanning electron microscope (SEM) equipped with an energy dispersive spectroscope (EDS) was used to analyze the solute content inside different grains. In addition, a Foundry-Master Pro spark optical emission spectroscopy (OES) was used to analyze the macrosegregation of Cu and Mg elements along the radial direction of the ingots. Sixteen samples from each casting condition were cut, as indicated by the blue lines in Fig. 2(c). Then, composition testing was performed at the flank of the samples [blue area in Fig. 2(c)]. Macrosegregation of Cu and Mg is characterized using an index ΔC (relative deviation),

$\Delta C = (C_i - C_0)/C_0$ [3], where C_i represents the average Cu or Mg content at one test position and C_0 represents the average Cu or Mg content of the ingot.

3 Results

3.1 Structures

Figure 3 exhibits the macrostructures of the ingots prepared by DC casting with SCF or DCF. For the ingot cast with SCF, the macrostructure consists of solidified shell near ingot surface, an area with feathery grains next to the shell, and coarse equiaxed grains in the region of the ingot center. For the ingots cast

with DCF (with the insertion of 2024 Al rod at Position 1), an area of about 70 mm in width with fine equiaxed grains can be observed in the center [dashed red rectangle in Fig. 3(b)]. With lowering the inserting position to Position 2, marked by the dashed red rectangle in Fig. 3(c), the rod is agglomerated in the center region of the ingot, and a 10–20 mm wide transition zone [marked by the dashed red ellipse in Fig. 3(c)] can be observed between the insertion and the ingot. The results show that the insertion of the 2024 Al rod does not significantly affect the solidified shell and the feathery grains zone but significantly refined the coarse equiaxed grains zone when the rod is inserted from Position 1.

Fig. 3: Macrostructures of ingots with SCF (a) and DCF (b, c)

Figure 4 shows the microstructures of different ingots within a 1/3 radius (50 mm) to the center. As shown in Fig. 4(a), the microstructure of the ingot cast with SCF mainly consists of coarse equiaxed grains. In addition, typical floating grains [marked by black dashed irregular graphic in Fig. 4(d)] with coarse-cell dendrites in the interior [marked by yellow dashed irregular graphic] and fine-cell dendrites in the periphery are observed in the center region of the ingot. For the ingot cast

with DCF (with the insertion of 2024 Al rod at Position 1), interestingly, grains [marked by the black dashed ellipse in Fig. 4(e)], which have large non-dendritic areas in the interior, are found in the fine equiaxed grains zone. With the insertion of 2024 Al rod at Position 2, a good metallurgical interface is formed between the insertion and the ingot, as shown in Fig. 4(c). Moreover, the spherical grains in the 2024 Al alloy insertion show a typical semi-solid structure [19–20]. In addition,

Fig. 4: OM images at center area of ingots with SCF (a, d) or DCF (b, c, e–g)

a large number of pores are found in the insertion [marked by the black arrows in Fig. 4(f)]. Finally, a mixed structure consisted of fine non-dendritic grains [marked by the dashed black rectangle in Fig. 4(g)] and coarse dendritic grains can be observed in the transition zone.

Figure 5 shows the microstructures at the positions of 75 mm, 95 mm, and 125 mm from the center of ingots. There is no

difference of microstructure between the ingots cast with SCF and DCF in the range of 75–125 mm from the center of ingots. Moreover, feathery grains can be found in the outer region of the ingots. Thus, the microstructures in Fig. 4 and Fig. 5 indicate that the insertion of the 2024 Al rods mainly affects the center region of ingots.

Fig. 5: OM images at different positions of ingots with SCF or DCF

Figure 6 shows the average grain size distribution of equiaxed grains from the center to the surface of the different ingots. With the insertion of 2024 Al rod at Position 1, the average grain size in the center of the ingot cast with DCF is 369–488 μm , at the distance of 0–25 mm from the center of ingot, which is about 50% smaller than that of the ingot cast with SCF (891–896 μm). With the insertion of 2024 Al rod at Position 2, the average grain size is the smallest of the three ingots due to the fine spherical grains in the center. Moreover, the fine non-dendritic grains result in a decrease in the average grain size of the transition zone. Hence, at 25 mm from the center of the ingot, the average grain size with the 2024 Al rod inserted at Position 2 is also smaller than that of the ingot without the insertion. However, at 50 mm, 75 mm, and 95 mm from the center of the ingot, the average grain size does not change much with the application of DCF.

Figure 7 shows the SEM images and area scan spectrum maps of the samples taken from different points of the ingots cast with SCF or DCF. Several typical grains can be observed in

Fig. 6: Average grain size at different positions of ingots cast with SCF or DCF

each of the SEM images as being the same as those in the OM images (Fig. 4). The typical coarse-cell structures of floating grains are marked by the dashed black rectangle in Fig. 7(a). The grains with large non-dendritic areas in the interior are

Fig. 7: SEM images and area scan spectrum maps of different samples taken from ingots cast with SCF (a) or DCF (b, c, d)

marked by the dashed black ellipse in Fig. 7(b). The nearly spherical grains of the 2024 Al insertion are shown in Fig. 7(c), and the equiaxed grains with coarse-cell structures are shown in Fig. 7(d). For all cases, Cu and Mg elements concentrate in the eutectic structure, inter dendritic arms or grain boundaries. However, the Mn element uniformly distributes within the grains. The points [marked by the black cross in Fig. 7(a-d)] at each typical grain were analyzed by using EDS through point scan spectrum (Table 1) to identify the differences in contents of solute elements between the different typical grains. As shown in Table 1, the content of Mn shows the most uniform distribution among the three elements. In addition, the Cu and Mg element contents are nearly the same between Points 1 and 4, and a similar result is also observed between Points 2 and 3. However, the Cu and Mg element contents at Points 1 and 4 are much lower than those at Points 2 and 3. The results mean that the coarse-cell structures of floating grains in the sample cast with SCF and the grains in the transition region of the ingot cast with DCF (Position 2) are confirmed to be solute-lean.

Table 1: Point scan spectrum results taken from different points as denoted by crosses in Fig. 7

Points	Cu (wt.%)	Mg (wt.%)	Mn (wt.%)
Point 1 in Fig. 7(a)	0.78	0.76	0.47
Point 2 in Fig. 7(b)	1.51	1.01	0.46
Point 3 in Fig. 7(c)	1.85	1.24	0.51
Point 4 in Fig. 7(d)	0.82	0.70	0.49

However, the Cu and Mg contents in the spherical grains of the 2024 Al insert and the special grains in the center of the ingot with DCF (Position 1) are closer to the nominal composition.

3.2 Macrosegregation

Figure 8 shows the macrosegregation of Cu and Mg elements along the radius of ingots. As shown in Fig. 8(a), within 20 mm from the center of the ingot, the relative deviation of the Cu element in the ingot with SCF is significantly greater than that in the ingots cast with DCF [marked by the dashed black ellipse in Fig. 8(a)]. At the center of the ingot cast with SCF, the relative deviation of Cu is -0.073 . For the ingot cast with DCF, the relative deviation of the Cu element is about 0.003 (with the insertion of 2024 Al rod at Position 1) and 0.034 (with the insertion of 2024 Al rod at Position 2). The results show a general negative centerline segregation region in the ingot prepared by traditional DC casting, and the negative centerline segregation of the Cu element can be decreased with the insertion of 2024 Al rod. Within 20–40 mm from the center of the ingots, the relative deviation of Cu element is about -0.065 [marked by the dashed black rectangle in Fig. 8(a)] with the insertion of 2024 Al rod at Position 2. This range corresponds to the transition region between unmolten insert and the ingot [Fig. 3(c)]. Hence, there is apparent negative segregation in the transition region. For all the cases, the relative deviation of the Cu element is relatively uniform in the range of 40–140 mm, and is particularly high in the subsurface and surface region. As the relative deviation of Cu element in the subsurface region is less than zero and that in the surface region is greater than zero, there is strong negative segregation in the subsurface region

Fig. 8: Determined macrosegregation of Cu (a) and Mg (b) at different points of ingots cast with SCF and DCF

and positive segregation in the surface region of all the cases.

According to Fig. 8(b), the distribution trend of the Mg element is similar to that of the Cu element in each ingot. Strong positive segregation is formed in the surface region, and strong negative segregation is formed in the subsurface region of all ingots. For the ingot cast with SCF, the negative segregation of the Mg element develops in the center region [marked by the dashed black ellipse in Fig. 8(b)]. For Mg element, similar segregation also forms in the transition region of the ingot cast with DCF (with the insertion of 2024 alloy rod at Position 2) [marked by the dashed black rectangle in Fig. 8(b)]. Moreover, the negative segregation of the Mg element is reduced in the ingot cast with DCF (with the insertion of 2024 alloy at Position 1).

4 Discussion

4.1 Effect of double cooling field on solidification structures

With traditional DC casting, the heat is removed only through the outer mold and the water jet. The external melt solidifies at a high cooling rate, while the internal melt solidifies at a low cooling rate [21]. Hence, coarse grains form in the center region of the ingots due to the low cooling rate, and feathery grains form in the outer radius of the ingots because of the high cooling rate and the high temperature gradient [22-23]. In DC casting with DCF, the heat is removed not only through the outer mold and the water jet but also through latent heat absorption due to the melting of 2024 Al insert. The insertion of 2024 alloy rod results in apparent grain refinement in the center region of the ingot, while it does not show significant effect on the structures in the outer radius of the ingots.

Solidification structures in the center of the ingot mainly depend on the melting degree of the insert and the chill-affected zone. With the insertion of the 2024 Al rod, a chill zone formed around the surface of the insert in the upper part, and the heterogeneous nucleation substrate for α -Al phase formed by the surface of the insert [14]. As a result, grains nucleated on the surface of the insert and separated from the surface before settling to the solidification front. The length

of contact between the melt and insert differs, and so does the melt temperature around the insertion. As a result, some of the grains remelt in the moving process, and the unmolten grains become the source of equiaxed grains. These factors depend on the insertion positions of the 2024 Al rods. Due to the heat transfer from the melt to the insert and from the insert to the cooled ingot, the insert melts from solid state to semi-solid or liquid state and resolidifies from semi-solid or liquid state into part of the ingot. A low melting degree of the insert will result in an obvious interface between the insert and the ingot. A high melting degree of the insert means the liquid phase of the insert mixes with the melt, and the unmolten α -Al particles significantly influence the solidification structures. The melting degree of the insert is also affected by the inserting position.

Since the 2024 Al rod was inserted from Position 1, it went through a higher temperature region and was heated for a longer time. Therefore, most grains originating from the surface of the insert remelted in the sump. Moreover, a higher melting degree of the insert was achieved, resulting in a high-volume fraction of the liquid phase and a large number of non-dendritic and nearly spherical α -Al particles. The solid α -Al particles act as the nucleus of heterogeneous nucleation, which make contributions to the increase in the number of nuclei and the refining of grains. In addition, these α -Al particles are the source of the special grains in Fig. 4(e). Insertion at Position 2 is 50 mm below Position 1 and is closer to the bottom of the sump, which means that the melt temperature is lower, the distance to the solidified ingot is shorter, and the heating time is shorter. Hence, when the 2024 Al rod was inserted from Position 2, lots of fine non-dendritic grains nucleated and separated from the surface region, part of which settled to the solidification front without much growth, and the others acted as the source of equiaxed grains in the transition region. In addition, the melting degree of the insert was lower. The structure of the 2024 Al rod was changed from primary fibrous structure to non-dendritic and spheroidal structure after the inserting process, which was the typical structure of semi-solid 2024 Al alloys. In this state, as a small-volume fraction of liquid phase located at the boundary of α -Al phase, the semi-solid state of the insert had good liquidity and agglomerated in the center of the ingot.

4.2 Effect of double cooling field on macrosegregation

Macrosegregation is a common defect in ingots prepared with DC casting. Generally, the surface layer of the ingot is removed, which means that the strong positive segregation in the surface and negative segregation in the subsurface have less effect on the application of ingots. However, the negative centerline segregation of the ingots cannot be eliminated, which seriously influences the composition uniformity of the ingots.

With conventional DC casting, the centerline segregation is mainly affected by thermal convection, floating grains, and the horizontal component of shrinkage-induced flow. The first causes positive (normal) centerline segregation, and the latter two contribute to negative centerline segregation^[24-27]. Generally, the effects of floating grains and shrinkage-induced flow play the dominant role in the segregation, which cause the negative centerline segregation of ingots. As for DC casting with DCF, when the 2024 Al rod is inserted from Position 1, the solute content of the Cu and Mg elements in the unmolten particles from the 2024 Al rods is higher than that in the floating grains and closer to the nominal composition, which is beneficial for reducing negative segregation. Due to the latent heat from the fusion of the insert, the isotherms in the center of the mushy zone could resemble a hillock-shape or become flatter. If the isotherm resembles a hillock-shape, the horizontal component of shrinkage flow is towards the center of the ingot, which is contrary to the horizontal component of shrinkage flow in traditional DC casting. If the isotherm becomes flatter, the horizontal component of shrinkage flow will be smaller. Therefore, with the application of DCF, the intensity of the horizontal component of shrinkage flow in the center region is decreased no matter whether the isotherm becomes hillock-shaped or flatter. Overall, the negative centerline segregation is reduced when the 2024 Al rod is inserted from Position 1. As the insertion position drops from Position 1 to Position 2, the grains in the transition region form earlier than the grains in the equiaxed grains region. Since the distribution coefficients of Cu and Mg elements are less than unity^[21, 25], the concentrations of these elements in the solid phase are lower than that in liquid during the solidification process. Thus, the grains in the transition region are solute-poor, which causes the negative segregation in the transition region.

5 Conclusions

The effects of DCF on the structures and macrosegregation of a 2024 Al ingot were studied, and the main conclusions are as follows:

(1) Introducing a 2024 Al alloy rod into a melt of the same alloy during the DC casting process significantly refines the grains and reduces the negative centerline segregation, which is related to the inserting position of the rod.

(2) When inserted from Position 1 (50 mm above the upper edge of the graphite ring), the 2024 Al rod is fully melted, which

consists of a high-volume fraction of liquid phase and a large number of unmolten α -Al particles. As the α -Al particles act as the nucleus of heterogeneous nucleation and increase the number of nuclei, the average grain size in the center region of the 2024 Al ingot cast with DCF is decreased by about 50 %, compared with that of the ingot cast with SCF. Meanwhile, the α -Al particles originating from the insert are relatively solute-rich, which can reduce the negative centerline segregation of Cu and Mg.

(3) When inserted from Position 2 (on the same level as the upper edge of the graphite ring), most of the 2024 Al rod cannot be melted and the unmolten particles agglomerate in the center of the ingot, the microstructure of the unmolten rod is transformed from primary fibrous structure into non-dendritic and spheroidal structure. However, the insert in the center region of the ingot, and the transition zone between the insert and the ingot lead to inhomogeneity in composition and structure.

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