The Mechanism of Reducing "A" Segregates in Steel Ingots*

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Synopsis

The mobility of interdendritic liquid metal under the semi-solid state was measured by sucking the liquid to a vacuum chamber. Si and Mo contents in steel investigated were varied by lowering the Si content or by adding Mo in order to study the reducing mechanism of "A" segregates in steel ingots. The experimental results obtained are summarized as follows:

(1) The amount of metal sucked decreases as the fraction of solid increases. An abrupt increase in the fluidity resistance takes place at a lower fraction of solid in the case of low Si steel as compared with that of high Si steel. The effect of Si is notably clear in the case of low-alloy steel containing Ni, Cr, Mo and V.

(2) These phenomena can not be explained only by the difference in the density or in the viscosity between the interdendritic fluid liquid and the remaining bulk liquid. Consequently they are explained in terms of the morphology of dendrite when the solidification front is being formed in the semi-solid region.

(3) The effects of Si and Mo contents on the reducing mechanism of "A" segregates can be explained as follows; The solidification front is formed at a lower fraction of solid when the Si content is lowered or Mo is added. Accordingly the driving force to float up the solute-enriched liquid does not become large enough to form "A" segregates since the solute is not sufficiently enriched in the interdendritic liquid at the solidification front.

To conclude, the reducing mechanism of "A" segregates can be explained in terms of a shift of the solidification front to the side of lower fraction of solid resulting from the change in the morphology of dendrite.

I. Introduction

In an attempt to clarify the formation mechanism of "A" segregates, which are often found in large killed steel ingots, the authors¹⁾ have already made an authentic study using a special device to reproduce "A" segregates in laboratory-scale ingots. The result obtained from the study led to a conclusion that "A" segregates are formed at the interface between solid-like (quasi-solid) and liquid-like (quasi-liquid) portions in the semi-solid region, in other words at the solidification front. It was also pointed out that the study on the solute-enrichment in the liquid phase at the solidification front would be important to make clear the mechanism. The characteristic phenomena of this solute-enrichment at the solidification front was discussed on the basis of the experimental results of direct sampling from the solidification front in conjunction with studying the influence of alloying elements on the appearance of "A" segregates in the experimentally reproduced ingots. It was made clear from the study that as the cooling rate becomes lower with the progress of solidification, the solidification front is formed at a higher fraction of solid, which is

increasing the severeness of solute-enrichment. It was, however, found that the shift of the solidification front to a higher fraction of solid, which prevents the formation of "A" segregates is suppressed by the addition of 1% Mo.

Since all these phenomena are considered to result from the difference in the morphology of dendrite while it is being formed, a series of experiments was achieved in the way that the fluidity of quasi-liquid in the semi-solid region is measured and compared with the morphology of dendrite being formed. In this work, the reducing mechanism of "A" segregates in large killed steel ingots is discussed with explaining why the vacuum carbon deoxidation process is industrially effective in reducing the amount of "A" segregates in low-alloy steel ingots containing Mo.

II. Experimental

The morphology of dendrite at the solidification front is considered to greatly influence the mobility of the interdendritic liquid. The fluidity of the interdendritic liquid was therefore measured on a low-alloy steel by means of vacuum-forced suction described below.

1. Experimental Apparatus and Procedure

Figure 1 shows a schematic view of the experimental apparatus. The sample weighing 140 g was melted

Fig. 1. Experimental apparatus.

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in a MgO crucible of 30 mm I.D. and 50 mm high at 1560°C for 5 min under an atmosphere of argon. About 3 g of the melt was subsequently sucked into an opaque silica tube of 3 mm diameter to determine the initial concentration of the bulk liquid. An alumina pipe of 3.5 mm I.D. was immersed in the center of the melt and held for 5 min. The melt was then cooled to a desired temperature in the solid-liquid co-existing temperature range at the rate of 5°C/min by controlling electric power supplied to a heat resistance furnace. Temperature was controlled by a thermocouple inserted in the base of the crucible which had precalibrated the temperature difference between the bulk liquid and the base of the crucible. A vacuum chamber was held under vacuum of a constant degree by opening cock B connected to a vacuum pump and by closing cock A connected to the alumina pipe. After a certain period of time, cock B was closed and cock A was opened to suck the interdendritic liquid at any fraction of solid corresponding to a holding temperature. The power supplied to the furnace was simultaneously cut to cool the melt. A cooling rate of 20°C/min was obtained by this operation. The sucked metal was weighed and the composition was determined to give the concentration of quasi-liquid phase (C_L). On the other hand, the composition of dendrite remaining in the crucible was determined by EPMA to give the concentration of solid-phase (C_S) at the holding temperature.

2. Samples

The samples used for the experiment were prepared by melting high carbon and low carbon NiCr-MoV and MnNiMo steels without silicon in a 3 kg vacuum induction furnace. Silicon was added to some of the samples prior to the experiment. Chemical compositions of the samples are shown in Table 1.

III. Results

From the data obtained, it is clear that the amount of sucked metal decreases with decreasing holding temperature. In Fig. 2, the carbon content of sucked metal C_L^g , and that of remaining dendrites, C_S^g , are

plotted against the holding temperature. In the figure, the dot and dashed line and the dashed line respectively represent the calculated liquidus line (T_s) calculated from the formula given by Mori *et al.*⁵⁾ C_S^g could not be obtained for low carbon samples because EPMA analysis was not accurate enough in measuring the carbon content of dendrite.

In the semi-solid region, the solute is distributed in solid and in liquid according to the equilibrium distribution except in the case of very rapid solidification. Therefore the temperature of any position in the semi-solid region corresponds to the liquidus temperature of the enriched-liquid existing at that position.³⁾ An empirical equation, proposed by Mori *et al.*⁵⁾ for the liquidus temperature of steel with varying composition, is fairly accurate and it fits in with the temperature arrests on the cooling curves obtained in the present experiment, as shown in Fig. 2. On the

Fig. 2. Relation between quasi Fe-C system and C_L^g and C_S^g .

Table 1. Chemistry of experimental molten steel.

Material			Chemical composition (%)								
			C	Si	Mn	P	S	Ni	Cr	Mo	V
Low carbon series	Mn-Ni-Mo steel	Low Si	0.13/0.18	0.01/0.10	1.05/1.18	0.006/0.008	0.005/0.008	0.74/0.77		0.45/0.53	
		High Si	0.14/0.19	0.31/0.36	1.20/1.31	0.007/0.009	0.007/0.010	0.75/0.78		0.51/0.56	
	Ni-Cr-Mo-V steel	Low Si	0.16/0.21	0.06/0.11	0.26/0.30	0.009/0.013	0.011/0.016	3.63/3.84	1.66/1.94	0.42/0.47	0.12/0.13
		High Si	0.20	0.37/0.42	0.31/0.32	0.009/0.012	0.007/0.011	3.72/3.85	1.28/1.80	0.43/0.49	0.12/0.14
High carbon series	C (1%Mo) steel	Low Si	0.57/0.59	0.03/0.04	0.52/0.55	0.014/0.016	0.008/0.009			1.01/1.03	
		High Si	0.56/0.60	0.24/0.25	0.55	0.017/0.019	0.007			0.97/1.05	
	Mn-Ni-Mo steel	Low Si	0.60/0.66	0.01/0.11	1.14/1.20	0.005/0.010	0.008/0.009	0.71/0.81		0.50/0.54	
		High Si	0.61/0.65	0.24/0.26	1.17/1.18	0.005/0.006	0.008/0.010	0.78/0.81		0.51/0.54	
	Ni-Cr-Mo-V steel	Low Si	0.59/0.65	0.02/0.12	0.28/0.34	0.006/0.009	0.008/0.009	3.36/3.57	1.53/1.66	0.50/0.53	0.13/0.14
		High Si	0.61/0.62	0.29/0.31	0.29/0.30	0.006/0.007	0.009/0.010	3.35/3.52	1.61/1.62	0.49/0.52	0.13/0.14

other hand, thermal analysis for the measurement of solidus temperature is very difficult to achieve. Mori *et al.*⁵⁾ commented that the empirical equation for T_s is less reliable than that for T_L . Also in the present experiment no well-marked temperature arrest for T_s was observed on the cooling curves. The solidus line was, therefore, determined by tying the carbon contents, C_s^g , measured by EPMA for the remaining dendrites after experiments. The solidus lines thus obtained are shown in Fig. 2 by solid lines. Since C_s^g could not be obtained by EPMA analysis for low carbon samples, the solidus line calculated from the equation given by Mori *et al.* was substituted. A modified quasi Fe-C binary phase diagram is shown in Fig. 3. Drawing a horizontal line for the holding temperature on Fig. 3, C_s^g and C_L^{g*} are obtained from the intersections of the line and the solidus and the liquidus lines respectively. (Note that C_L^{g*} is distinguished from C_L^g , the carbon content of sucked metal). The fraction of solid g_s^c is calculated from these values and the carbon content of initial liquid, C_0^g .

$$g_s^c = \frac{C_L^{g*} - C_0^g}{C_L^{g*} - C_s^g} \dots\dots\dots(1)$$

Thus the calculated fractions of solid for each experiment are plotted against the amount of metal sucked in Figs. 4 and 5. In spite of some scattering these figures clearly represent that on abrupt drop in the amount of metal sucked emerges at lower fractions of solid in the case of low Si steel as compared with that of high Si steel. This tendency is notably clear in the case of NiCrMoV steel. As shown in Fig. 2 the carbon content of metal sucked, C_L^g , often shows a lower value than the calculated equivalent content, C_L^{g*} . If only the liquid existing between dendrite arms was sucked, C_L^g should have agreed well with C_L^{g*} . The reason why C_L^g often shows a lower value than C_L^{g*} is considered that not only the liquid but also the free dendrite particles existing in the liquid were sucked together and that the carbon content of the metal sucked was diluted.

Defining W_L and W_s as the weights of liquid and solid in the sucked specimen weighing W , the following equations are obtained.

$$W = W_L + W_s \dots\dots\dots(2)$$
$$WC_L^g = W_L \cdot C_L^{g*} + W_s \cdot C_s^g \dots\dots\dots(3)$$

Fig. 3. Calculation method of solid fraction from a modified quasi Fe-C system.

Combining Eqs. (2) and (3), it becomes

$$\frac{C_L^{g*} - C_L^g}{C_L^{g*} - C_s^g} = \frac{W_s}{W} \equiv \beta^c \dots\dots\dots(4)$$

Equation (4) implies that if C_L^{g*} , C_L^g and C_s^g are known, the weight ratio of solid in the sucked specimen, β^c , can be readily calculated. The contents of other elements than carbon can be measured in dendrite arms and the content of arbitrary element “ i ” is symbolized as C_s^i . Assuming that the weight ratio calculated for carbon, β^c , is also valid for element “ i ” Eq. (4) can be written for “ i ” and is transformed to obtain the content of “ i ” in the liquid as described below.

$$C_L^{i*} = \frac{C_L^i - \beta^c \cdot C_s^i}{1 - \beta^c} \dots\dots\dots(5)$$

Where C_L^i is the content of “ i ” in the sucked specimen. The contents of Cr and Mo in dendrite arms are determined by EPMA analysis. These values are plotted against the temperature in Fig. 6. Solid lines

Fig. 4. Effect of Si on the relation between the solid fraction and the amount of liquid-like phase sucked (Low-C system).

Fig. 5. Effect of Si on the relation between the solid fraction and the amount of liquid-like phase sucked (High-C system).

Fig. 6. Solidus lines of quasi Fe-Mo and Fe-Cr systems.

connecting these points plotted are regarded as the solidus lines for quasi Fe-Cr and Fe-Mo phase diagrams. Drawing a horizontal line for the holding temperature of each sample in the present experiment, C_s^i can be obtained as the intersection of the horizontal line and the solidus line. Using the value C_s^i along with C_L^i and β^c , C_L^{i*} can be readily calculated from Eq. (5). Once C_L^{i*} is obtained, the fraction of solid, g_s^i , which is based on the concentration analysis of element “ i ”, is calculated in a similar manner as the calculation of g_s^c from Eq. (1).

$$g_s^i = \frac{C_L^{i*} - C_o^i}{C_L^{i*} - C_s^i} \dots\dots\dots(1)'$$

Thus g_s^{Cr} and g_s^{Mo} obtained were compared with g_s^c . As shown in Fig. 7 they were in fair agreement. This fact implies that the procedure of obtaining the fraction of solid from the holding temperature on quasi phase diagrams is adequate. If all solid existing in the liquid is sucked from a site of the fraction of solid g_s^c , the following equation should be valid.

$$\beta^c = g_s^c, g_s^{Cr}, g_s^{Mo} \dots\dots\dots(6)$$

When the dendritic network starts to form the fraction of solid at solidification front, the amount of solid sucked is decreased abruptly. When suction is done from a site which has a higher fraction of solid than that of front, the following equation is set up.

$$\beta^c \leq g_s^c, g_s^{Cr}, g_s^{Mo} \dots\dots\dots(7)$$

The relationship between β^c and g_s^c is shown for each sample in Fig. 8. Depending on the kind of steel and the Si content, there exists a critical fraction of solid, g_s^* , for Eq. (7). The value g_s^* is considered to be the fraction of solid at the solidification front. There is a clear tendency in each kind of steel that low Si steel has a lower critical fraction of solid at which the dendritic network firmly forms. This agrees well with the previously mentioned fact that a low Si material shows an abrupt drop in the amount of metal sucked at lower fractions of solid due to the formation of dendritic network. Another tendency can be seen from Fig. 8 is that the more the kinds of alloying element in a low-alloy steel is, the lower the fraction of solid at the solidification front becomes.

Fig. 7. Comparison between the solid fractions calculated from equilibrium partition of different kinds of solute (C, Mo, Cr).

Fig. 8. Relation between g_s^c and β^c .

This tendency agrees well with the fact that “A” type segregates in large low-alloy steel ingots are notably reduced by the low Si vacuum carbon deoxidation process.

IV. Discussion

1. Flow of Liquid Phase between Dendrite Arreys

Piwonka and Flemings⁶⁾ proposed a formula for the flow of liquid phase between dendrite arms in the semi-solid region, adopting Darcy’s Law⁷⁾ which dealt with liquid flow in a porous material.

$$Q = k \cdot \Delta P / \eta \cdot L \dots\dots\dots(8)$$

where Q is the flow rate per unit area, ΔP is the pressure drop across length L , η is the viscosity of liquid, and K is the permeability. The permeability K has the dimension of cm^2 and is given as

$$K = \frac{f_L^2}{8n\pi t} \dots\dots\dots(9)$$

Where f_L is the fraction of liquid, n is the number of flow channels, and t is "tortuosity factor" to account for the fact that the liquid flow channels are not straight and smooth. In the present experiment ΔP is given by ΔP_0 , the pressure difference between the atmosphere and that of vacuum chamber (ΔP_0 is about 1 atm, $1.0133 \times 10^6 \text{ dyn/cm}^2$), the density of liquid ρ , the length l , and the acceleration of gravity g as follows;

$$P = \Delta P_0 + \rho g l \dots\dots\dots(10)$$

Takahashi and Kudo⁸⁾ showed that the alleviation of macrosegregations such as "A" type segregates by decreasing the Si content is due to the effect of Si on the density difference between the solute-enriched liquid and the bulk liquid. That is, as is shown in Fig. 9, the density of solute-enriched liquid is increased by decreasing the Si content. This suppresses the driving force to float the solute-enriched liquid from the solidification front and results in the alleviation of macrosegregation. In the case of forced suction as in the present experiment, however, the density term in Eq. (10), $\rho \cdot g \cdot L$, is no more than $1.6 \times 10^4 \text{ dyne/cm}^2$ and is extremely small as compared with ΔP_0 . (The following values are used to calculate $\rho g L$; $\rho = 6 \sim 7 \text{ g/cm}^3$, $g = 980 \text{ cm/sec}^2$, $L = 2 \sim 3 \text{ cm}$). In the present experiment, therefore, a change in the density of solute-enriched liquid, caused by different Si contents, has a very small effect on ΔP . Secondly, the effects of Si content on the viscosity η in Eq. (8) should be considered. As can be seen from the experimental data⁹⁾ shown in Fig. 10, η decreases with

Fig. 9. Change in the density difference between solute enriched liquid and bulk liquid during solidification.

increasing Si content. However the difference in viscosity η is very small in the range of $0 < \text{Si} < 0.3\%$. The effect of Si content on the viscosity η is too small to cause a large change in the amount of metal sucked, as much as several times, depending on the Si content.

From the consideration mentioned above, the difference in the amount of metal sucked or the ratio of solid phase in the specimen sucked can be attributed to the difference in permeability K . It is often observed in commercial products from steel ingots or in etched cross-sections of large test ingots that the dendrite array is finer in the case of low Si steel. In the experimental study of reproducing "A" segregates in laboratory-scale ingots, reported in a previous paper,²⁾ it was observed that the formation of "A" segregates were suppressed and the dendrite array was found in an ingot containing 1% of Mo. Above mentioned facts are assured by the morphology of dendrite observed in the remaining solid samples after suction, as shown in Photo. 1. The characteristic morphology of dendrite in a low Si steel is described as follows;

- 1) Curvature at the tip of dendrite is small.
 - 2) Dendrites of higher degree tend to form easily.
- Character 1) qualitatively means that dendrites are easily connected each other because of the high growth rate of dendrite tips and the solidification front is easily formed. Character 2) means that the space for flowing liquid, *i.e.* flow channel, is narrow. The mechanism of how the Si content affect the morphology of dendrite as in 1) and 2) is left for further study, however, it was made clear that the morphology of dendrite in the semi-solid region is affected by lowering the Si content, and that the alleviation of "A" segregates is attributed to decrease the fraction of solid at the solidification front.

2. The Reducing Mechanism of "A" Segregates Achieved by the Shift of Solidification Front

From the results of the present experiment, it was made clear that the fraction of solid at which the solidification front forms is shifted to the lower side by lowering the Si content. As is easily noticed, there are two major differences in the process of solidification between one in the present experiment and the other in actual commercial ingots. In the present experiment, the boundary between quasi-

Fig. 10. Viscosity of liquid Fe-Si alloy.

Photo. 1.
Dendrite structure appeared after suction.

liquid and quasi-solid phases was determined by forced suction which can not occur in the natural solidification of commercial ingot. In the present experiment the samples were held at constant temperatures in the semi-solid region, whereas commercial ingots are cooled continuously in the manufacturing process even though the cooling rate is slow. That is why the fraction of solid at the solidification front obtained from the present experiment shows higher value than that obtained from commercial ingots or experimental ingots used for the reproduction of "A" segregates. However the phenomenon that the solid fraction at the solidification front is shifted to the lower side by lowering the Si content is still valid for commercial ingots. The reducing mechanism of "A" segregates in commercial ingots can be explained by the above mentioned fact.

In Fig. 11, the way of reducing "A" segregates is schematically shown in connection with the formation of solidification front at a lower fraction of solid. Let us consider the semi-solid region in the process of horizontal solidification from the surface to the center of an ingot. As the temperature decreases from the liquidus temperature T_L to the solidus temperature T_S , the fraction of solid increases from 0 to 1. Liquid concentration C_L between dendrite arms increases with increasing fraction of solid from the initial concentration C_0 . Figure 11 (d) shows that when the position of solidification is shifted depending on the Si content, the fraction of solid at the solidification front of low Si steel $g_{s, \text{Low Si}}^*$ becomes smaller than that of high Si steel, $g_{s, \text{High Si}}^*$. Corresponding to the small g_s^* , an enrichment of solute at the solidification front of low Si steel is reduced. "A" segregates are considered to be formed from the solute-rich liquid trapped by the solidification front or quasi-solid phase while it is floating up. To give the driving force to float up the solute-rich liquid, a sufficient density difference is required to exist between the

Fig. 11. Schematic explanation for the reduction of solid fraction at solidification front by low Si.

solute-enriched liquid and bulk liquid. In the solidification of low Si steel, a sufficient driving force for the floatation of solute-rich liquid can not be obtained because of insufficient enrichment of the liquid and insufficient decrease in the density of the liquid.

From the results of sampling at the solidification front in the horizontal solidification experiments, it is known that the fraction of solid at the solidification front increases as the solidification proceeds toward the center of the ingot and the cooling rate decreases accordingly. The observed relationship

between the solid fraction at the solidification front, therefore, it can be reasonably assumed that even in the region where the fraction of solid increases because of low cooling rate, low Si steel still has a lower fraction of solid at the solidification front than that of high Si steel, as shown in Fig. 12 (a). In this figure g_s^{*A} designates the critical fraction of solid for producing a sufficient solute enrichment to bring about "A" segregates. Drawing a horizontal line from g_s^{*A} in Fig. 12 (a), the critical cooling rates to give g_s^{*A} are obtained from the intersections in the figure for low Si steel and high Si steel. The critical cooling rate for low Si steel, ϵ_L^A , is smaller than that for high Si steel, ϵ_H^A . ($\epsilon_L^A < \epsilon_H^A$). Figure 12 (b) shows the profile of cooling rate along the radial direction of an ingot, from the surface to the center. Figure 12 (c) is obtained by combining Figs. 12 (a) and (b), and shows the relationship between the radial distance and the fraction of solid at the solidification front. It is known from the figure that in the course of the solidification of high Si steel, a sufficient enrichment of solute occurs and the solidification front is formed at the position relatively close to the surface of the ingot. If a low Si steel is poured into a mold in which ϵ_L^A is too low to be achieved even in the center, the formation of "A" segregates is suppressed. Although the explanation described above is not the one determined quantitatively, the experimental results thus obtained ensures a favorable effect of lowering the Si content on the alleviation of "A" segregates in Mo steel containing Ni, Cr and V.

The reducing mechanism of "A" segregates in steel ingots by lowering the Si content and by alloying Mo is explained as follows: the growth rate of higher-order dendrite branches become higher and the solidification front is formed at a lower fraction of solid so that there does not exist the densely-solute-enriched liquid necessary to form "A" segregates. According to the calculation made by Takahashi *et al.*,⁸⁾ the density difference between the interdendritic liquid and the bulk liquid is reduced by lowering the Si content. Therefore the driving force to float the solute-enriched liquid, if ever exists, is lessen. This might contribute to the alleviation of "A" segregates.

V. Conclusions

In the formation of "A" segregates the solute-enrichment of liquid at the solidification front plays an important role. Therefore the mobility of the liquid at the solidification front was measured by sucking the liquid to a vacuum chamber. Si and Mo contents in the steels investigated were varied by lowering the Si content or by adding Mo in order to study the reducing mechanism of "A" segregates in steel ingots. The experimental results obtained are summarized as follows;

(1) The amount of metal sucked decreases with increasing fraction of solid because of the fluidity resistance against liquid flow. An abrupt increase in the resistance occurs at a lower fraction of solid in the case of low Si steel as compared with that of high Si steel. This tendency is notably clear in the case

Fig. 12. Schematic explanation for the reduction of Λ segregation by low Si.

of Ni-Cr-Mo-V low-alloy steel.

(2) The lower the Si content is, the ratio of solid in a sucked specimen is reduced to be zero at a lower fraction of solid. This theoretically supports the phenomenon that in a low Si steel the solidification front is formed at the side of lower fraction of solid.

(3) Effects of Si on the amount of metal sucked, in the case of Mo containing steel, can not be explained only by the difference in the density or in the viscosity between the interdendritic liquid and the bulk liquid. These should be considered from the difference in the morphology of dendrite formed during the formation of solidification front in the semi-solid region.

(4) In the solidification of low Si steel, higher-order dendrite branches tend to form easily. The growth rate at the tip of these dendrites is supposed to be high.

From the above mentioned phenomena and from the experimental results obtained in the series of previous study to clarify the formation mechanism of "A" segregates, the reducing mechanism of "A" segregates in large steel ingots is inclusively explained as follows;

(1) As the solidification proceeds toward the center of an ingot, the cooling rate at the solidification front decreases and the fraction of solid increases. The solute is accordingly enriched in the interdendritic liquid and the difference in the density between the interdendritic and the remaining bulk liquids becomes larger. This gives a driving force to float the solute-enriched liquid, resulting in the formation of "A" segregates.

(2) When the Si content is lowered, the solidification front tends to be formed at the side of lower fraction of solid. In this case a shift of the fraction of solid at the solidification front toward the side of higher fraction of solid is suppressed even when the cooling rate at the solidification front is decreased.

(3) The reasons why "A" segregates are not formed in a low Si steel under the conditions permitting the formation of "A" segregates in a high Si steel are as follows;

(a) The fraction of solid at the solidification front is lower in the case of low Si steel.

(b) Solute-enrichment in the interdendritic liquid is not severe in the case of low Si steel, and the density difference between the interdendritic and bulk liquids is small.

(4) It is possible to produce "A" segregate-free large ingots if the fraction of solid at the solidification front is controlled under the critical fraction of solid by reducing the Si content or by some other means.

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