Control of the Non-Metallic Inclusions near Solidification Front by Pulsed Magnetic Field

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Abstract: Aiming to investigate the behavior of non-metallic inclusions near an advancing solid-liquid interface and the effect of a pulsed magnetic field on the distribution of inclusions, motions of inclusions without and within a pulsed magnetic field were observed in real-time during the solidification of 45 steel. The distribution of inclusions and the evolution of the microstructure were investigated. It was found that a pulsed magnetic field favors the engulfment of inclusions. A uniform distribution of inclusions was obtained. The microstructure was mainly composed of acicular ferrite within the pulsed magnetic field. A mathematical model was proposed to describe the reaction between inclusions and the advancing solid-liquid interface and the phenomenon of inclusion engulfment/pushing by the solidification interface. Furthermore, the critical velocity of inclusions to be engulfed was calculated. It can be concluded that the numerical value of the critical velocity of inclusions to be engulfed differs from the experimental value. After the revision of the mathematical model, the numerical value of the critical velocity was about 4.47 µm/s, validating the experimental value.

Keywords: solidification; microstructure; magnetic field; acicular ferrite

1. Introduction

When a liquid metal contains dispersed second-phase inclusions, the solidification interface and the inclusions interact during the solidification process. The inclusions are engulfed or pushed by the solidification interface. Engulfment causes inclusions to be evenly distributed in the solid phase. Pushing causes inclusions to segregate in the final solidified area, such as the grain boundary or the area between dendrites [1–4]. The phenomenon of inclusion engulfment is particularly important in the fabrication of metal-matrix composites [5–7]. Chen et al. discussed the influence of the inclusions present near the solidification interface on interface shape during the solidification of Al7075–Al2O3np-based metal matrix composites and suggested that smaller inclusions are easily engulfed by the interface rather than pushed. Fadavi Boostani et al. made aluminum matrix composites with a uniform distribution of the inclusions, and the tensile ductility improved by 84%, which was due primarily to the variation of the solidification mechanism from inclusion pushing to inclusion engulfment during solidification. As mentioned above, inclusion engulfment has been studied with different metal-matrix composites, but the reports about applications to the iron-based alloy are few because of the high melting point. Moreover, 45 steel is an important structural material. Therefore, this paper investigated the inclusion engulfment/pushing in 45 steel from 1600 °C to 1150 °C. Inclusion behavior near the solidification interface was studied by quantitative measurement and calculation.
In the past decade, intra-granular ferrite (IGF) nucleated on non-metallic inclusions in iron-based alloys has attracted much attention in order to promote the toughness of the heat-affected zones (HAZs) [8–10]. Li et al. investigated the effect of Mg addition on the formation of intra-granular acicular ferrite structures in low-carbon steel. Fattahi et al. studied the influence of inclusions on the acicular ferrite microstructure and mechanical properties; the results showed that the microstructure was changed to acicular ferrite with the intra-granular nucleation of ferrite on inclusions, and the mechanical properties were improved. This improvement is attributable to the increased percentage of acicular ferrite due to the uniform distribution of inclusions. In general, a microstructure consisting mainly of acicular ferrite provides optimum weld metal and HAZ mechanical properties, both from a strength and toughness point of view, by virtue of its fine effective grain size and high-angle grain boundaries. However, previous works were only confined to the effect of inclusion nature and size on the microstructure [11–16], and due to the limitation of inclusion control technology, few works have referred to the effect of inclusion distribution.

Pulsed magnetic field processing (PMF) in the solidification of alloy has been considered an effective method to reduce the segregation of solute elements, refine grains and improve the mechanical properties of the alloys, and more attention has been paid to it in recent years [17–20]. Shao et al. reported that the uniformity of the alloy steel 20Cr2Ni4A is improved by using pulsed magnetic treatment. Zhang et al. found that the solidification microstructure of Mg-7Zn alloy was further refined with a pulsed magnetic field. Chen et al. applied a pulsed magnetic field to pure aluminum; the grain size of the sample was smaller, and the size distribution was more uniform. Therefore, this paper investigated the effect of a pulsed magnetic field on inclusion distribution and microstructure. In this study, a new technique that combines a high-temperature confocal scan laser microscope (HTCSLM) is first proposed. The behavior of inclusions near the advancing solidification interface was observed in situ. Based on the results, inclusion velocities were calculated. A mathematical model was proposed to describe inclusion motion, forces acted on the inclusions in the melt, and engulfment/pushing of inclusions occurred by the solidification interface. The effect of the pulsed magnetic field on the inclusion behavior near the advancing solidification interface was discussed.

In the present work, the experimental and theoretical analysis of the inclusion velocity near the advancing solidification interface was compared. The theoretical considerations will be applied to the experimental findings for the engulfment of inclusions.

2. Materials and Methods

2.1. Materials and Heat Treatment

A specimen with a longitudinal height of 60 mm was cut from the bottom of a 45 steel-casting blank. Samples of $\Phi$ 7 mm × 3 mm were obtained at the position of 1/2 from the section center to the edge, whose chemical composition (mass fraction, %) is shown in Table 1.

Table 1. Chemical compositions of the experimental steel (%).

| C  | Si   | Mn   | P    | S    | V    | Cr   | Al   | Ti   | N    |
|----|------|------|------|------|------|------|------|------|------|
| 0.470 | 0.270 | 0.750 | ≤0.015 | ≤0.020 | 0.080 | 0.200 | 0.020 | 0.012 | 0.010 |

In this work, a high-temperature confocal scanning laser microscope (HTCSLM, VL2000DX-SVF17SP, T/C: R type, YONEKURA, Tokyo, Japan) was chosen to observe inclusion behavior under the protection of argon gas. It can control the cooling rate precisely and dynamically observe the inclusion motion during solidification at high temperatures. The PMF generator is self-designed equipment [21] (North China University of Science and Technology, Tangshan, China). It is sited between the microscope and the sample, and the sample is on the central axis of the magnetic field. Based on the results of HTCSLM, the inclusion motion is measured, and the inclusion velocity is calculated. The experimental apparatus for solidification is shown as a sketch in Figure 1.
Experiments have been conducted to study the influence of cooling rate and holding time on the nature of inclusions and microstructure of 45 steel [22]. The results show that: (1) inclusions inducing the nucleation of intra-granular ferrite were mainly MnS, TiN-VN-MnS, TiN- VN; (2) the cooling rate of 100 °C/min from 1600 °C to 1150 °C is favorable for the nucleation of intra-granular ferrite; (3) when the melt was held for 3 min at 1150 °C, the proportion of inclusions less than 10 μm increased to 96%; (4) when the melt held for 10 min at 750 °C, the area ratio of ferrites increased to 41%. In order to study the effect of PMF on the inclusion motion in molten steel, samples were melted at 1600 °C and austenitized at 1150 °C. The samples were heated from room temperature to 1600 °C and held for 10 min; after that, they were cooled down to 1150 °C at a cooling rate of 100 °C/min and held for 3 min, then cooled down to 750 °C at a rate of 100 °C/min, and held for 10 min. Finally, they were cooled down to room temperature. The melts were solidified with and without PMF. PMF was applied in the early stage of solidification from 1600 °C to 1150 °C; the temperature range is marked with red in Figure 2. The voltage of the PMF was 100 V with a pulse frequency of 0.5 Hz. Figure 2 shows the heat treatments employed in the present study.

**Figure 1.** Sketch of the experimental apparatus.

**Figure 2.** Schematic sketch of phase transformation stages treated with a PMF from 1600 °C to 1150 °C.
2.2. Measurement and Calculation

A rectangular coordinate system is established on the microscopic image. The inclusion coordinates are recorded every 0.3 s, and the inclusion trajectory is obtained by connecting these coordinates. Note that 0.3 s is relatively short enough for the entire movement process, so it can be assumed that the inclusions move in a straight line at a uniform speed within 0.3 s. The average velocity of inclusion in every 0.3 s can therefore be estimated as

$$v_p = \frac{\sqrt{(x_2 - x_1)^2 + (y_2 - y_1)^2}}{0.3}.$$  (1)

where $v_p$ is the inclusion velocity; $x_1$ and $y_1$ are the initial horizontal and vertical coordinates of inclusion in every 0.3 s, respectively; and $x_2$ and $y_2$ are the terminal horizontal and vertical coordinates of inclusion in every 0.3 s, respectively.

3. Results

3.1. The Effect of PMF on Microstructure

Before solidification, inclusions in the liquid steel migrate at high speed. When there is PMF within molten steel (shown in Figure 3a), the PMF promotes the floating up of inclusions. The number of inclusions in the field of view is greater (shown in Figure 3c).

The size of inclusions in molten steel was analyzed by image processing software. The extracted inclusions are marked in red. Figure 3a shows inclusions in melt without PMF. Those red areas in Figure 3b are inclusions (Figure 3a) identified by the software. Figure 3b shows inclusions in the melt with PMF. Those red areas in Figure 3d are inclusions (Figure 3c) identified by the software. Hence, the size of inclusions was calculated based on the pixels of the marked area. The analysis results of extracted inclusions are shown in Table 2.

| Application of PMF | Average Circumference (µm) | Radius (µm) |
|--------------------|---------------------------|-------------|
| No PMF             | 20.34                     | 3.24        |
| PMF                | 34.57                     | 5.50        |

Figure 3. Extracted inclusions by image processing software (a,b) without PMF and (c,d) with PMF.
The average size of inclusions within PMF is larger. This is because the PMF promotes inclusion floating up; the probability of inclusion collision and segregation increases during the ascent because the inclusions become bigger.

In the solidification process, the motion of inclusions near the solidification front is recorded without and within PMF. The dynamic process in nine consecutive time cells (one time cell = 0.3 s) is shown in Figure 4.

![Figure 4](image_url)

**Figure 4.** Dynamic behavior of particle motion in nine consecutive time cells, and the tracked inclusions with five colored circles (a) without PMF and (b) with PMF.

The movement of the tracer inclusions (marked by circles of different colors) is clearly contrasted. Circles of the same colour indicate the same inclusion at different times. Without PMF, the inclusions detour when they approach the solidification front and are pushed back to the molten steel, as shown in Figure 4a. Within PMF, more inclusions arrive at the solidification front instead of detouring, and the velocity of inclusions decreases more quickly to zero when they arrive at the front, as shown in Figure 4b. The results show that the application of PMF in the solidification process can change the motion of inclusions. The solidification process is also affected by PMF. The application of PMF will inhibit solidification. The corresponding solidification temperatures decrease, as shown in Figure 5.

![Figure 5](image_url)

**Figure 5.** Motion traces of inclusions in molten steel, and five tracked inclusions marked as five numbers (a) without PMF and (d) with PMF. The distribution of particles in the last regions to solidify, and all visible inclusions marked by red circles (b) without PMF and (e) with PMF. The solidified microstructure of samples (c) without PMF and (f) with PMF.
When the melt is held without PMF, at the beginning of solidification (shown in Figure 5a), inclusion 1, inclusion 3 and inclusion 5 detour when they are close to the solidification front. Inclusion 2 and inclusion 4 arrive at the solidification front, and their velocities decrease to zero; that is, they are engulfed. The motion traces of inclusions are long and straight, which indicates that inclusions take a longer time to be engulfed. In the middle of solidification, inclusions (marked by a circle, as shown in Figure 5b) segregate to the molten steel between the grains and move with high speed. As shown in Figure 5c, the final solidified microstructure is a developed dendrite.

In contrast, in the case of PMF, five tracer inclusions are all engulfed when they approach the solidification front at the beginning of solidification, as shown in Figure 5d. The motion traces of inclusions are short and curved, which indicates that inclusions take a shorter time to be engulfed and withstand more complex forces. In the middle of solidification, inclusions (marked by a circle, as shown in Figure 5e) are rare in the molten steel between the grains. Inclusions are mostly distributed inside the solid phase zone or solidification front. It is indicated that PMF can promote inclusion engulfment and suppress segregation, which is shown in Figure 5f. In other words, applying PMF during solidification is in favor of uniform inclusion distribution.

The percentage of inclusions engulfed in the observable inclusions in the view is defined as the engulfment rate. The influence of PMF on the engulfment rate is shown in Figure 6.

![Figure 6. Histogram of engulfment rate from 1600 °C to 1499 °C without and with PMF.](image)

Inclusions in the field of view from 1600 °C to 1499 °C were counted. The statistical result is shown in Figure 6. The inclusion engulfment rate in PMF is about three times the case without PMF. In summary, the application of PMF in the solidification process improves the probability of inclusion engulfment.

With the aim of revealing the effect of PMF on solidification microstructure, a further microscopy study using a DMI5000M optical microscope was conducted. After grinding and polishing, the specimens were etched with a solution of 96 mL ethanol and 4 mL nitric acid. Phases can be easily distinguished from the matrix because they present different contrasts under optical microscopy. The white phase is ferrite islands, and the gray phase is pearlite. Ferrite laths were mainly precipitated at the grain boundary, a few acicular ferrites and massive ferrites were precipitated inside the grain, and the grain boundaries were coarse (as shown in Figure 7a). After the PMF was applied, more ferrites were precipitated inside the grain, and most of them were acicular, fine and cross-interlocked; the grain boundaries were clear and sharp, as shown in Figure 7b. The result indicates that the application of PMF largely promotes intra-granular ferrite nucleation [23].
3.2. Theoretical Consideration for the Critical Velocity

The control of inclusion, that is, whether the inclusion is engulfed or pushed by the solidification front, is determined by the competition of forces acting on the inclusion. The balance of forces can produce a steady-state pushing speed [24–29], namely critical velocity; otherwise, the disruption of balance can cause inclusions to be engulfed or pushed. The different forces experienced by inclusions near the solidification front are shown in Figure 8.

![Figure 7. Optical microscope images of samples (a) without PMF and (b) with PMF.](image)

![Figure 8. Schematic diagram of the forces acting on a particle in the vicinity of the solid–liquid interface.](image)

The forces acting on the inclusion during the solidification process vary in time. At the initial stage, the distance between the inclusion and solidification front is large, and the repulsive and attractive forces are very small. When the solidification front approaches inclusion to the nanometer level, the repulsive forces become larger. In time, the forces rise steeply, and the balance between the repulsive forces and the opposing forces becomes very delicate. Based on the balance of forces acting on the inclusions, the expression of the critical velocity is generated. It can quantify the inclusion push/engulf transition. When the inclusion velocity is lower than the critical velocity, the inclusions are pushed, and when the inclusion velocity is higher than the critical speed, they are engulfed [30,31].

The interfacial force \( F_I \) is derived either from the interfacial energy or van der Waals forces, which repulse the inclusion away from the solidification front. The expression takes the form:

\[
F_I = 2\pi R_P \Delta \gamma_0 \left( \frac{a_0}{a_0 + d_{\text{min}}} \right)^2 \left( \frac{k_p}{k_l} \right).
\]

(2)
where $R_p$ is the inclusion radius, $\Delta \gamma_0$ is the interfacial energy change, $a_0$ is the interatomic distance, $k_p$ and $k_l$ are the thermal conductivities of the inclusion and metal melt, $d_{\text{min}}$ is the distance between the inclusion and the solidification front, and the values for $d_{\text{min}}$ vary over four orders of magnitude from 0.1 nm up to 1000 nm. Kaptay proposed that $d_{\text{min}}$ should be around $250 \times a_0$; this is also confirmed by more sophisticated FEM results published recently [32].

The electromagnetic force ($F_p$) is subject to electromagnetic extrusion pressure due to the electric conductivity difference between the inclusion and the conductive liquid. The direction of the electromagnetic extrusion pressure may be changed with the magnetic field or the current of the conductive melt. The electromagnetic force per unit of volume produced by a conductive melt in an electromagnetic field is

$$f_l = \frac{B_0^2}{\mu_m \delta} e^{-\frac{2x}{\delta}} \cos\left(\frac{x}{\delta} - \omega t\right) \times \left[\cos\left(\frac{x}{\delta} - \omega t\right) + \sin\left(\frac{x}{\delta} - \omega t\right)\right].$$

where $\mu_m$ is the permeability of conductive melt; $\delta$ is the skin layer thickness, $\delta = \sqrt{2/\mu_m \sigma \omega}$, $\sigma$ is the electric conductivity; $\omega$ is the angular frequency of the magnetic field, $x$ is the position of the sample; $B_0$ is the magnetic field strength; and $t$ is the action time of the magnetic field. The electromagnetic force is only generated in the melt with good conductivity and does not occur in inclusions with poor conductivity. Therefore, the inclusions are affected by the opposite electromagnetic force (electromagnetic repulsion). For many non-metallic inclusions,

$$F_p = -\frac{3}{4} \frac{\pi R_p^3}{\delta} f_l$$

(4)

The drag force ($F_D$), which resists the movement of inclusions away from the solidification front, results from liquid flow around the inclusion. For a spherical inclusion near the interface, it is given by the modified Stokes’ law.

$$F_D = 6 \pi \eta V_p \frac{R_p}{d_{\text{min}}} \left(\frac{k_p}{k_l}\right)^2.$$

(5)

where $\eta$ is the dynamic viscosity of the melt and $V_p$ is the inclusion velocity.

The gravity force ($F_G$) is important only for inclusions larger than 10 $\mu$m in diameter and the case in vertical casting conditions. The inclusion size in this study is less than 6 $\mu$m; therefore, the effect of gravity can be eliminated.

The force $F_\sigma$, generated by the concentration gradient of the solute in the melt, is a function of the temperature and concentration at the interface. As usual, if $F_\sigma \ll F_p$, the effect of $F_\sigma$ on the inclusion behavior can be ignored.

Under a steady state, the critical velocity for the engulfment/pushing transition of the inclusion is obtained from the force balance by setting $V_p = V_c$.

$$F_l + F_p - F_D = 0.$$

(6)

$$\frac{3}{4} \frac{\pi R_p^3}{\delta} \frac{B_0^2}{\mu_m \delta} e^{-\frac{2x}{\delta}} \cos\left(\frac{x}{\delta} - \omega t\right) \times \left[\cos\left(\frac{x}{\delta} - \omega t\right) + \sin\left(\frac{x}{\delta} - \omega t\right)\right]$$

$$= 2 \pi R_p \Delta \gamma_0 \left(\frac{a_0}{\pi d_{\text{min}}} + \frac{d_{\text{min}}}{k_p}\right)^2 \left(\frac{k_p}{k_l}\right)^2 - 6 \pi \eta V_p \frac{R_p}{d_{\text{min}}} \left(\frac{k_p}{k_l}\right)^2.$$

(7)

The expression for the critical growth velocity $V_c$ can be obtained from Equation (7), which reads then as

$$V_c = \frac{\Delta \gamma_0 a_{\text{min}} k_p}{3 \pi R_p^{\delta} \frac{B_0^2}{\mu_m \delta} \left(\frac{k_p}{k_l}\right)^2} e^{-\frac{2x}{\delta}} \cos\left(\frac{x}{\delta} - \omega t\right) \times \left[\cos\left(\frac{x}{\delta} - \omega t\right) + \sin\left(\frac{x}{\delta} - \omega t\right)\right]$$

$$= 390.23 - 0.36 \times \cos(0.119 - 3.14t) \times \left[\cos((0.119 - 3.14t)) + \sin((0.119 - 3.14t))\right] \mu m/s.$$
where \( R_p = 5.5 \, \mu m; a_0 = 0.36662 \, nm; d_{\text{min}} = 91.66 \, nm; k_p = 1.0 \, W \cdot m^{-1} \cdot K^{-1}; k_l = 31.2 \, W \cdot m^{-1} \cdot K^{-1}; \eta = 0.0062 \, kg \cdot m^{-1} \cdot s^{-1}; \Delta T_0 = 0.879 \, \text{J} \cdot m^{-2}; B_0 = 0.009 \, T; \mu_m = 4\pi \times 10^{-7} \, \text{H} \cdot \text{m}^{-1}; \sigma = 7.14 \times 10^3 \, \text{S} \cdot \text{m}^{-1}; \omega = 2\pi \alpha = 3.14 \, \text{rad} \cdot s^{-1}; \delta = \sqrt{2/\mu_m \sigma \omega} = 0.84 \, m; \) and \( x = 0.1 \, m. \) The effective value of the critical velocity (\( V_c^{\text{eff}} = V_c^{\text{max}} / \sqrt{2} \)) is about 389.93 \( \mu m/s \) within PMF.

### 3.3. Experimental Value of Critical Velocity

To further investigate the behavior of inclusions near the solidification front, the velocity of inclusions was measured and calculated according to Equation (1). The evolution curve of inclusion velocity over time is plotted in Figure 9.

![Figure 9. Velocity evolution of particles near the advancing solidification front (a) without PMF and (b) with PMF.](image)

In the absence of PMF, velocities of inclusions fluctuate with time, as shown in Figure 9a. The fluctuation is caused by the flow of molten steel; in addition to this, the influence of the passing solidification front must also be considered. After applying PMF, fluctuations can also be seen in Figure 9b. The fluctuation is mainly caused by the flow of molten steel and the electromagnetic force. Each maximum value in fluctuation represents a force balance. The maximum value in the curve (\( V_{\text{max}} \)) is about 389.93 \( \mu m/s \).

3.4. Comparison of Theoretical and Experimental Values of Critical Velocity

As mentioned above, the experimental result differs greatly from the theoretical calculation results of Equation (8). Accordingly, a correction is made for the expression of the critical velocity. Assuming that inclusion velocity varies evenly before engulfment, \( d_{\text{min}} \) can be obtained according to the following Equation.

\[
d_{\text{min}} = \frac{1}{2} \Delta t^2 = \frac{1}{2} \Delta t^2.
\]  

(9)

Based on the experimental result of Figure 9, the value of \( d_{\text{min}} \) is about 2.68 \( \mu m \) within PMF. Then, Equation (8) changes to

\[
V_c = 13.40 - 10.49 \times \cos(0.119 - 3.14t) \times [\cos((0.119 - 3.14t)) + \sin((0.119 - 3.14t))] 
\]  

(10)

The numerical value of the critical velocity is about 4.47 \( \mu m/s \), which is close to the experimental value. The value of \( d_{\text{min}} \) is an important factor affecting the critical velocity.
4. Conclusions

In this work, experimental and theoretical analyses are used to study the effect of PMF on the behavior of inclusions near the solidification interface. The most important conclusion of this work is as follows:

1. Applying PMF during solidification can change the motion of inclusions and promote inclusion to be engulfed by the solidification front.
2. Due to the improvement of the engulfment probability within PMF, a uniform inclusion distribution was obtained.
3. A large amount of fine, acicular and cross-interlocked intra-granular ferrites was observed with the application of PMF.
4. When $d_{\text{min}} \approx 2.68 \, \mu\text{m}$, the theoretical is consistent with the experimental value. The experimental result of the critical velocity was validated by the prediction of the model.

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