



# Heterogeneous strip originated from the separate enriched melt: Innate character and physical mechanism



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## ABSTRACT

The chemical heterogeneity, well-known as macrosegregation, is a major problem in the casting of steel ingots. Most heterogeneity generally originated from the solute partition and solute distribution in the solidification process with interdendritic convection. In this article, a new heterogeneous phenomenon originated from the movement of separate enriched melt is discovered in a steel ingot. A strip characterized as ferrite chains and MnS chains is revealed by macro-etching. The formation mechanism for the heterogeneous strip is proposed. In the mushy zone of the ingot, a large amount of separate S-enriched melts move laterally and upwards. Some S-enriched melts will remain in the moving trace. Such residual S-enriched melts produce a large amount of MnS inclusion chains. In the subsequent solid phase transition process, promoted by the MnS chains, ferrite prefers to be transited from the austenite near the MnS inclusions and shows as a large amount of separate ferrite chains. A large amount of ferrite chains align in a strip-like zone, which results in the heterogeneous strip phenomenon in macro-etching process. The physical model about the driving force for the movement of separate S-enriched melts is further theoretically analyzed. In the mushy zone, the interface tension resultant applied on the separate S-enriched melts can act as the drive force for the lateral movement of separate S-enriched melts. And the buoyance applied on the separate S-enriched melts acts as the drive force for the upwards movement. The interaction between the impurity movement and the solute segregation is also discussed.

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## 1. Introduction

The heterogeneity of steel ingots significantly damages the mechanical properties of forgings produced from ingots [1–3]. For example, some disasters happened in crucial nuclear plant equipment could be induced by the non-uniform properties of the ingots. Generally, such heterogeneity of steel ingots is well known as macrosegregation. Because the macrosegregation can hardly be eliminated by forging and heat treatment process, the macrosegregation [2,4–8] is considered as one of the most severe problems in steel ingots. Many researches have been done about formation mechanism of macrosegregation. Generally, the macrosegregation is considered as the result of microsegregation and solid–liquid relative movement in solidification process. The interdendritic flow is induced by density difference, gravity, deformation and shrinkage [9–13]. Therefore, the interdendritic flow was highly evaluated for the macrosegregation, and many excellent numerical simulations about macrosegregation were performed based on thermosolutal flow calculation, shrinkage and

deformation [2,12,14–18]. Besides, some experiment research with model alloy system were also performed [19,20]. Among these experimental works, the thermosolutal convection and the formation of macrosegregation have been directly observed [20].

Interestingly, before establishing the mechanism of interdendritic thermosolutal flow, in the earlier research period, the researchers simply accepted the understanding that the solidification begins at the outside of the ingot. And the advancing solidification frontier layer will reject the impurities into the interior melt [21]. This early understanding about impurity rejection by solidification frontier is rebutted by the mush zone model proposed in 1960s. According to the mush zone model, only negligible solute buildup can occur in front of the mush zone [8]. It is further realized that the interdendritic flow can carry the enriched interdendritic melt out of the mushy zone.

Although great achievement was gained in the understanding about heterogeneity of steel ingots, some questions should be further clarified. Firstly, the concept of impurities is different from the interdendritic melt. The impurities should include all objects which have distinct compositions difference with the interdendritic melt. Generally, for the plain carbon steel, common impurities includes the liquid complex inclusions and the highly

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enriched melt compared with common interdendritic melt. Besides the thermosolutal convection, the movement of impurities should also be focused in the research about the heterogeneity of steel ingots. Secondly, there may be no contradiction between the impurity rejection and thermosolutal convection. The thermosolutal convection can carry both the impurity and the enriched interdendritic melt out of the mushy zone. But it may also be possible that the rejection of impurities from the mushy zone can be contributed by some driving forces other than thermosolutal convection. The rejection of inclusions in front of the solid–liquid interface has been reported by the in-situ experimental observation [22]. Thus, re-evaluation about the impurity rejection by the mushy zone should be performed. If the impurity can be rejected by the mushy zone, the partitioned solute may further be carried out of the mushy zone together with the movement of inclusions. Some previous work has shown such phenomenon that inclusion floatation will contribute to the solute movement and induce channel segregation [23,24].

In order to clarify above questions and possibility, the reliable basic information about heterogeneity must be acquired firstly by the detailed experimental characterization in steel ingot. And further, theory model about the phenomenon provided by the experimental characterization should be analyzed and established.

## 2. Experimental procedures

In this article, the nominal chemical compositions (weight percent) of the steel is C 0.30, Si 0.09, Mn 0.35, S 0.03, P 0.03, Cr 0.36, Ni 0.22, Cu 0.03, and Fe balanced. The steel was melted at 1873 K (1600 °C) by induction furnace, and was Al deoxidized. The oxygen content of the steel is wt. 0.0045%. In order to investigate the formation of inclusions, some melt was sampled and quenched before pouring. The inclusions in the quenched sample are investigated by the scanning electron microscope and the energy dispersive spectrometer.

The 500 kg sand mold steel ingot was bottom filled at 1823 K (1550 °C) in the atmosphere. In order to carefully characterize the heterogeneity of the steel ingot, the solidified ingot was cut in half along the longitudinal axis. The sectioned slice was further grinded and polished. In order to reveal the heterogeneity, the traditional macro-etching (etched by 20 vol% $\text{HNO}_3$ –5 vol% $\text{H}_2\text{SO}_4$ – $\text{H}_2\text{O}$  solution, 20 vol% $\text{HNO}_3$ – $\text{H}_2\text{O}$  solution and 5 vol% $\text{HNO}_3$ – $\text{H}_2\text{O}$  solution, rinsed by hot water and  $\text{C}_2\text{H}_5\text{OH}$ , and subsequently dried by hot air) was carried out. And in order to reveal further information about the heterogeneity of the steel ingot, an improved macro-etching technology was adopted (etched by 20 vol% $\text{HNO}_3$ –5 vol%  $\text{H}_2\text{SO}_4$ – $\text{H}_2\text{O}$  solution, 20 vol% $\text{HNO}_3$ – $\text{H}_2\text{O}$  solution and 5 vol%  $\text{HNO}_3$ – $\text{H}_2\text{O}$  solution, wiped with  $\text{C}_2\text{H}_5\text{OH}$  and cotton, rinsed by hot water and  $\text{C}_2\text{H}_5\text{OH}$ , and finally dried by hot air). The macrographs for the whole ingot and amplified macrograph for specific heterogeneous positions were observed. In order to reveal the further microstructural information about the heterogeneity, the specific section of the heterogeneous positions was etched by the 4 vol% $\text{HNO}_3$ – $\text{C}_2\text{H}_5\text{OH}$  solution, and was further observed by optical microscope and scanning electron microscope. The chemical compositions in the heterogeneous positions were tested by the spectrochemical analysis.

## 3. Experimental results

### 3.1. the macrograph of the heterogeneity

The macrograph revealed by traditional macro-etching technology is shown in Fig. 1(a), which displays obvious heterogeneous strips. The heterogeneous strips show as continuous strip-like dark

zones with about 50 mm away from the ingot surface. The improved macro-etching technology can further reveal the appearance and additional information about the heterogeneous strips, as shown in Fig. 1(b). Some distinct features are displayed compared with the macrograph revealed by traditional macro-etching technology. Revealed by the improved macro-etching technology, the heterogeneous strips are actually strip-like zones with a large amount of special dark lines intermingled in the common matrix. It should be noted that the special dark lines are actually separate and very slim. The special dark lines can be observed more clearly in the amplified part of macrograph, as shown in Figs. 1 (A), (B) and 2(a). The slim dark lines start from the outer position and stretches to the interior and upward position in the ingot.

### 3.2. The microstructures and compositions difference account for the special dark lines

In order to reveal the innate character of the slim dark lines, we etched the sample in the position of Fig. 2(a) with the 4 vol% $\text{HNO}_3$ – $\text{C}_2\text{H}_5\text{OH}$  solution. The etched sample is shown in Fig. 2(b). And the microstructures of two slim dark lines are further observed with optical microscope. Fig. 3(a) and (b) respectively show the optical microscopic pictures of dark line 1 and dark line 2 located in Fig. 2. In Fig. 3, the white zone is the ferrite, and the black zone is the pearlite. We can see that the normal microstructures of the ingot matrix are consisted by the ferrite on original austenite grain boundary, the pearlite and the acicular intragranular ferrite. The dark line 1 is characterized by some special discrete distributed ferrite, and the dark line 2 is characterized by some special continuous distributed ferrite. Both the discrete distributed ferrite and the continuous distributed ferrite arrange and display as ferrite chains.

In order to further reveal the innate character of the slim dark line, we observed the A, B and C zones in Fig. 3 with SEM. As shown in Fig. 4, we can see that the common matrix surrounding the slim dark lines is the microstructures constituted by the acicular ferrite, the pearlite and the grain boundary ferrite. In the dark line zones, the acicular ferrite reduces dramatically. The ferrites in the dark line zones are mainly constituted by the granular ferrite and network ferrite. The granular ferrite and network ferrite align as chains in the whole. Therefore, the innate character of the slim dark lines is actually the different appearance and distribution of ferrite.

The chemical compositions in the ferrite chains and in the common matrix were tested by the spectrochemical analysis. The chemical compositions (weight percent) in the ferrite chains are C 0.3544, Si 0.1806, Mn 0.4298, S 0.0414, P 0.0445, Cr 0.3122, Ni 0.1277, Cu 0.0393. The chemical compositions (weight percent) in the common matrix are C 0.3519, Si 0.1723, Mn 0.4139, S 0.0183, P 0.0230, Cr 0.3009, Ni 0.1177, Cu 0.0362.

### 3.3. The MnS inclusions inside and outside the heterogeneous strip

We further observed the ferrite chains inside the heterogeneous strip by SEM in several typical positions, as shown in Fig. 4. The positions a, b, c, d and e are in the ferrite chains of heterogeneous strip, and the position f is in the common matrix of heterogeneous strip. As shown in Fig. 5, the important information lies in that much MnS inclusions appear in the inner of the ferrite chains. As shown in Fig. 5(a), (c) and (e), the eutectic MnS appear in the interior of the network ferrite. And as shown in Fig. 5(b), (d) and (f), the rod-shaped and spherical MnS inclusions appear in the inner of granular ferrite and acicular ferrite. And the rod-shaped MnS inclusions are either parallel or perpendicular to each other.

The MnS inclusions outside the heterogeneous strip are also observed by SEM, as shown in Fig. 6. Similarly, the spherical

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