Effect of Magnesium Treatment on the Hot Ductility of Ti-Bearing Peritectic Steel

Tianpeng Qu, Deyong Wang, Huihua Wang *, Dong Hou and Jun Tian

Shagang School of Iron and Steel, Soochow University, Suzhou 215021, China; qutianpeng@suda.edu.cn (T.Q.); dywang@suda.edu.cn (D.W.); houdong0702@suda.edu.cn (D.H.); jtian@suda.edu.cn (J.T.)
* Correspondence: hhwang@suda.edu.cn; Tel.: +86-0512-6716-5621

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Abstract: Surface cracking is a major defect in the production of continuous casting slabs of peritectic steel. The difference in crystal structure between δ phase (before peritectic transformation of steel) and γ phase (after peritectic transformation) results in volume contraction, which leads to uneven cooling of mold and thus forming slab shells with different thicknesses. Then, coupled with the concentration of local stress, surface cracking occurs on slabs. In this paper, the effect of magnesium treatment on the hot ductility of Ti-bearing peritectic steel was studied, and the characteristics of solidification structure and TiN particles were analyzed. Magnesium treatment for Ti-bearing peritectic steel could significantly improve the hot ductility of continuous casting slabs by refining the original austenite structure. After the magnesium treatment, the average grain size of the original austenite of peritectic steel decreased by about 18.7%, and the size of Mg-rich TiN particles decreased by about 41%. In addition, the minimum reduction of area at the third brittle zone after the magnesium treatment was higher than 60%, and the fracture appearance changed from intergranular fracture to ductile fracture after the treatment. The contents of Mg, Ti, O, and N in peritectic steel and the cooling conditions were adjusted reasonably to promote the formation of highly dispersed Mg-rich TiN particles with a sufficient number density and a proper size in the initial solidification stage of peritectic steel, so as to induce the high-temperature δ-ferrite nucleation. Based on the fine δ structure formed by peritectic transformation, through the use of structure heredity and the pinning effect of secondary-precipitated nano TiN particles on the austenite grain boundary, a fine and dense original austenite structure could be obtained to improve the hot ductility of peritectic steel. Industrial tests showed that through the magnesium treatment, the surface cracks of Ti-bearing peritectic steel were effectively restrained, and the corner cracks of slabs were basically eliminated.

Keywords: magnesium treatment; hot ductility; Ti-bearing peritectic steel; continuous casting

1. Introduction

Peritectic reaction of steel is an important transformation mode of Fe-C alloy with a carbon content ranging from 0.10% to 0.53% in the initial stage of solidification [1–4]. When the primary high-temperature δ-ferrite reacts with the residual molten steel L to produce the γ-austenite, the difference in crystal structure between the δ phase (BCC structure) and the γ phase (FCC structure) results in a large volume contraction in the process of solidification, which causes uneven cooling of the primary shell in the mold, thus forming shells with different thicknesses and high cracking susceptibility [5–10]. This cause is considered as the main reason for the surface cracking of this type of steel. So far, cracking of peritectic steel slabs is still a major production problem troubling steel mills. The surface cracking involved includes longitudinal surface cracking [11,12], transverse cracking [13–17], and star-shaped cracking [18]. In addition, the hypo-peritectic steel with a carbon content of 0.10–0.15% has the most severe contraction, leading to coarse original austenite grains and
the highest probability of occurrence of longitudinal surface cracking [19] (as shown in Figure 1). The cracks on slabs require surface cleaning, or in severe cases, they may cause slab scrapping and even breakout accidents [20, 21].

Currently, the common control measures against the cracking of peritectic steel include: (1) Improving the heat transfer of the mold and reducing the solidification contraction, such as slow cooling of mold [22, 23], optimization of the mold flux [24, 25], and optimization of the taper of mold; (2) Composition control. In the design of steel type and composition control, the peritectic composition range is avoided as far as possible. However, for some steel types, especially when the content of Mn, Al, or Si is high, the coexisting zone of $L + \delta + \gamma$ phases is enlarged, as shown in Figure 2, and the current control measures cannot fundamentally eliminate the surface cracking defect of peritectic steel. Therefore, it is necessary to systematically study the heterogeneous nucleation mechanism of liquid phase in the initial solidification stage of peritectic steel and the peritectic transformation behaviors under non-equilibrium conditions, thus providing theoretical guidance for improving the quality of continuous casting slabs of peritectic steel. In addition, coarse original austenite grains are a key factor leading to the formation and expansion of cracks along grain boundaries. If the original austenite grains can be refined in the continuous casting process, the hot ductility of the continuous casting slabs can be significantly improved, thereby restraining the occurrence of surface cracking on the slabs.

![Figure 1. Influence of carbon content on shell characteristics.](image1.png)

![Figure 2. Pseudo-binary phase diagram of complex Fe-C alloy system.](image2.png)
2. Materials and Methods

The steel used in this study was Ti-bearing hypo-peritectic steel with a carbon content of 0.13%. Its composition is shown in Table 1 and the contents of C, Si, Mn, Ti, etc. in the steel were detected using a Spectro-Lab analyzer (NCS Inc., Beijing, China). The element N was detected using an ONH-3000 analyzer (NCS Inc., Beijing, China), and the element Mg was detected using a 6300 ICP analyzer (Varian Medical Systems, Palo Alto, CA, USA). The production process was BOF-LF-CC, and the cross section of the continuous casting slab was 220 mm × 1860 mm. For studying the effect of magnesium treatment on the hot ductility of peritectic steel, Mg-bearing cored wire (20% Mg-5% Al-75% Fe) was fed into the ladle through a wire feeder after LF refining. The parameters of the continuous casting process were kept the same. High-temperature tensile specimens were obtained from the 1/4 area below the slab surface. The sampling position and the specimen size are shown in Figures 3 and 4, respectively. High-temperature tensile tests were then carried out using a thermal-mechanical physical simulation system Gleeble 3800 (Data Sciences International (DSI), St. Paul, MN, USA). During each test, high-purity argon was used to prevent oxidation, and the temperature was raised to 1300 °C at a speed of 10 °C/s and then held for 2 min to obtain full austenite phase. After that, the specimen was cooled down to the tensile temperature at a speed of 5 °C/s. After holding the temperature for 3 min, the specimen was tensiled at a deformation rate of 10^{-3} \text{s}^{-1}. The temperature setting is shown in Figure 5. The cooling rate 5 °C/s was close to the cooling rate of continuous casting slab surface. The tensile test was conducted at a deformation rate of 10^{-3} \text{s}^{-1}, which was close to the straightening deformation rate 0.5 \times 10^{-3} \text{s}^{-1} - 0.7 \times 10^{-3} \text{s}^{-1}.

Table 1. Composition of the peritectic steel (wt%).

| Element       | C  | Si  | Mn  | P  | S   | Al  | Ti  | Mg  | O   | N   |
|---------------|----|-----|-----|----|-----|-----|-----|-----|-----|-----|
| Before Mg treatment | 0.13 | 0.02 | 0.34 | 0.01 | 0.009 | 0.0226 | 0.020 | 0 | 0.0032 | 0.0041 |
| After Mg treatment  | 0.12 | 0.03 | 0.36 | 0.01 | 0.007 | 0.0235 | 0.018 | 0.0015 | 0.0035 | 0.0039 |

Figure 3. Sampling position for high-temperature tensile test.

Figure 4. Specimen for hot ductility testing.
The effect of magnesium was used to analyze the microscopic characteristics of the inclusions. As can be seen, the minimum reduction of area at 780 °C was significantly higher than that of specimens with no magnesium added, and the reduction of area of specimens with magnesium added and that of specimens with no magnesium added both reached a minimum at 780 °C. After the magnesium treatment, the minimum reduction of area in the third brittle zone was higher than 60%, and the valley area of the curve of magnesium-added specimens was greatly reduced, indicating the hot ductility of the magnesium-added specimen was greatly improved, concluding that magnesium treatment could greatly improve the hot ductility of slabs.

3. Analysis and Discussion

3.1. Analysis of High-Temperature Test Results

The fracture appearance and the macro appearance of grains were analyzed using a ZEISS Axio Vert metallographic microscope (Carl Zeiss Inc., Jena, Germany). The grain size was quantitatively analyzed using IPP image analysis software (Image Pro Plus 6.0, Media Cybernetics Inc., Rockville, MD, USA). The specimens before and after the magnesium treatment were all dissolved through electrolysis in non-aqueous solution. In order to extract TiN particles from the solution, the inclusion electrolyte was separated out using a HC-2518 centrifuge (Zonkia Inc., Hefei, China) at 10,000 r/min. The appearance characteristics of the extracted non-metallic inclusions were analyzed using an SU5000 electron scanning microscope (Hitachi Inc., Tokyo, Japan). Specimens were then prepared using an ion polishing system (FIB, HELIOS NanoLab 600i, FEI Inc., Hillsboro, OR, USA) to be analyzed on a transmission electron microscope. After that, a high-resolution TEM (HRTEM, FEI TalosF-200x, accelerating voltage 200 kV, point resolution 0.25 nm, STEM resolution 0.16 nm, FEI Inc., OR, USA) was used to analyze the microscopic characteristics of the inclusions.

Figure 5. Temperature setting for the tensile test.
The fracture appearance of specimens after tensile failure at 780 °C was analyzed using the SEM, as shown in Figure 8. As can be seen from the figure, the fracture before the magnesium treatment was a typical intergranular fracture, while after the magnesium treatment, obvious dimples were found at the fracture position, and there were obvious plastic deformation marks around the dimples. Therefore, it can be inferred that the fracture was a ductile fracture. The formation of dimples was mainly because the dislocation loops accumulated around particles moved to the interface under the action of shear stress to form micropores, and the micropores further expanded and grew to form dimples. The appearance of dimples was related to the toughness of material and the test temperature. When the toughness of the material was poor, its plastic deformation ability was poor, and both the size and the depth of dimples were small. As the temperature rose, it was more conducive to the nucleation and expansion of dimples, thus increasing the width and depth of dimples. Therefore, according to the appearance of dimples, it could be concluded that the magnesium-added specimens had a better ductility, which was consistent with the analysis results of the hot ductility curve.

Figure 6. Variation curve of the reduction of area before and after magnesium treatment.

Figure 7. Macro fracture appearance of tensile specimens. (a) Before magnesium treatment. (b) After magnesium treatment.
3.2. Solidification Structure Analysis

Picric acid corrosion was performed on specimens from the central area of the slab thickness before and after the magnesium treatment. The appearances of their original austenite grains were compared, and the results are shown in Figure 9. The original austenite grain boundary was outlined using the proeutectoid ferrite structure precipitating along the original austenite grain boundary [26]. It can be seen that the austenite grain size after the magnesium treatment was smaller than that before the magnesium treatment. The average austenite grain size before the magnesium treatment was 155 µm, which was reduced by 18.7% to 126 µm after the magnesium treatment.

The room-temperature appearances of the steel specimens before and after the magnesium treatment are shown in Figure 10. Photos of grains were taken at the same magnification times, and ten photo groups were selected to calculate their average grain sizes and plot histograms, as shown in Figure 11. As can be seen, the number of grains in 1 mm² of room-temperature structure before the magnesium treatment was 116, while after the magnesium treatment, changed to 156, increasing by 34.5%; and the grain size of the room-temperature structure before the magnesium treatment was equivalent to a diameter of 115 µm, while after the magnesium treatment, was equivalent to a diameter of 89 µm, reduced by 22.6%. The grains were obviously refined, indicating that the magnesium treatment improved the as-cast structure of peritectic steel.
was less. In addition, TiN tended to nucleate on the MgO or MgAl spinel phase. MgAl and the nucleation rate was higher. Therefore, a large number of composite TiN particles with MgO or MgAl as the heterogeneous nucleation sites at the beginning of solidification, the required nucleation energy decreased by about 41% to 5.76 µm, which decreased by about 41% to 5.76 µm after the magnesium treatment. If the TiN in molten steel took MgO or MgAl₂O₄ as the heterogeneous nucleation sites at the beginning of solidification, the required nucleation energy was less. In addition, TiN tended to nucleate on the MgO or MgAl₂O₄ surface with a good wet interface, and the nucleation rate was higher. Therefore, a large number of composite TiN particles with MgO or MgAl₂O₄ as the core were formed in the Ti-bearing peritectic steel after the magnesium treatment.

3.3. Appearance Characteristics of Inclusions in Steel

Figure 12 shows the appearance of TiN particles in the specimens and the distribution of elements before the magnesium treatment, and Figure 13 shows the appearance and the distribution after the magnesium treatment. As can be seen, the two-dimensional appearance of TiN was a square, and the TiN in specimens with no Mg added mostly existed in pure TiN phase or in a composite phase with Al₂O₃. After Mg was added into the steel, the TiN particles in specimens showed an obvious core. In terms of the core composition, there were generally two types. According to the element distribution diagram, if the Mg and O were evenly distributed in their respective circles, then MgO was considered as the core; similarly, if Mg, O and Al were evenly distributed in their respective circles, the core of TiN was the magnesium-aluminum spinel phase MgAl₂O₄. It can be seen that the average size of TiN particles in the specimen before the magnesium treatment was about 9.54 µm, which decreased by about 41% to 5.76 µm after the magnesium treatment. If the TiN in molten steel took MgO or MgAl₂O₄ as the heterogeneous nucleation sites at the beginning of solidification, the required nucleation energy was less. In addition, TiN tended to nucleate on the MgO or MgAl₂O₄ surface with a good wet interface, and the nucleation rate was higher. Therefore, a large number of composite TiN particles with MgO or MgAl₂O₄ as the core were formed in the Ti-bearing peritectic steel after the magnesium treatment.
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Figure 12. Appearance of TiN particles in specimens before magnesium treatment.

Figure 13. Appearance of TiN particles in specimens after magnesium treatment.

3.4. Precipitation Characteristics of Nano TiN Particles

The precipitates in the slab specimens before and after the magnesium treatment were sampled using the carbon extraction replica technique. The appearance of nano TiN particles and its variation were analyzed using a TEM. Figure 14 shows the distribution of nano TiN particles in each slab specimen before and after the magnesium treatment. As can be seen, the nano TiN particles in the steel before the magnesium treatment were generally in chain distribution, and were inferred to be distributed along the grain boundaries. After the magnesium treatment, the distribution of TiN particles in the specimens was more dispersed, and no obvious chain distribution was found. It can be inferred that the TiN particles were basically in a state of dispersed distribution in the matrix. The size and the number density of TiN particles were calculated. The average size of nano TiN particles before the magnesium treatment was about 30 nm, which is the same as after the magnesium treatment, indicating the magnesium treatment did not insignificantly influence the size of nano TiN particles. This was mainly because the nano TiN particles were generally precipitated under the action...
of desolventizing during cooling, and the magnesium treatment mainly promoted the nucleation of micron TiN particles formed at the initial stage of solidification [27,28].

**Figure 14.** Appearance of nano TiN particles in Ti-bearing peritectic steel specimens: (a–c) before magnesium treatment; (d–f) after magnesium treatment.

From the above analysis, it can be seen that the hot ductility of Ti-bearing peritectic steel after the magnesium treatment has been significantly improved. The reason is not only related to the refined original austenite structure, but also to the dispersed distribution of nano TiN particles precipitating from the solid slab after full solidification.

**Figure 15** shows how the magnesium treatment refines the grains of Ti-bearing peritectic steel during solidification. After the Mg was added into the Ti-bearing peritectic steel, it rapidly formed superfine high-melting inclusions (MgO or MgAl₂O₄ particles) with dissolved O and Al in the steel. With the decrease of molten steel temperature, the concentration product of [Ti][N] in the molten steel before solidification exceeded the equilibrium concentration product in the steel, and then TiN began to precipitate. Due to the good interfacial wettability between TiN and MgO/MgAl₂O₄, TiN nucleated on the surface of MgO/MgAl₂O₄ particles. When the temperature of molten steel dropped below the liquidus, δ phase began to form from the liquid phase. Similarly, based on the good lattice matching between TiN and δ phase, δ phase was more liable to nucleate on the surface of TiN particles, which promoted the grain refinement of high-temperature δ phase. When the temperature dropped to the peritectic transformation temperature, the peritectic transformation δ + L→γ occurred. Based on the heredity in phase transformation, the refined original austenite structure was obtained, and accordingly, the hot ductility of the steel was significantly improved. The refined ferrite structure could also be obtained from the refined original austenite structure during the γ→α phase transformation. Combined with the experimental study, the refinement of the surface and internal grain structure of the slab could effectively improve its hot ductility, and thus significantly preventing the surface cracking defect in the production of peritectic steel.
4. Conclusions

For the surface cracking defect of Ti-bearing peritectic steel slab, a method for suppressing this defect through magnesium treatment was proposed in this study. The feasibility of this method was validated via laboratory and industrial tests, and its inherent mechanism was also systematically analyzed.

The original austenite structure and the room-temperature structure of Ti-bearing peritectic steel after the magnesium treatment were refined significantly. After the magnesium treatment, the average grain size of the original austenite of peritectic steel decreased by about 18.7%, and the grain size of room-temperature structure decreased by about 22.6%. After the magnesium treatment, MgO or MgAl2O4 cores were generally found in TiN particles, and the particle size decreased.

The hot ductility of Ti-bearing peritectic steel was significantly improved after the magnesium treatment. The tested steel with no Mg added commonly showed hot ductility deterioration at 780 °C. However, the minimum reductions of area in the third brittle zone after the magnesium treatment were all higher than 60%, and the fracture appearance changed from intergranular fracture to ductile fracture after the treatment. Industrial tests showed that through magnesium treatment, the surface cracks of Ti-bearing peritectic steel were effectively restrained, and the corner cracks of slabs were basically eliminated.

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