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Study on as-cast microstructure of Fe-15Mn-10Al-5Ni-0.8C low density duplex steel

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Abstract

The microstructure of Fe-15Mn-10Al-5Ni-0.8C low density duplex steel ingots was studied by laser confocal scanning calorimetry, metallographic microscopy and scanning electron microscopy. The results indicated that: the microstructure of Fe-15Mn-10Al-5Ni-0.8C low density steel was mainly composed of coarse austenite dendrites and $\delta$-ferrite phase in dendrite. The $\delta$-ferrite could be completely eliminated by solution treatment. A dynamic model of austenite grain growth was established. Through analysis, it was concluded that the critical temperature for rapid austenite growth was 1250 $^\circ$C. The activation energy for austenite grain growth was 365 KJ/mol as calculated by Sellars’ model.

1. Introduction

In recent years, researchers have paid great attention to Fe–Mn–Al–C series low density steel for its high strength and light weight to better achieve the development goal of energy conservation and emission reduction. This tendency has taken place in a number of industrial production sectors, such as transportation vehicles, maritime, oceanic submarines and aerospace [1–5]. As Frommeyer noted in the study [6], the density of steel could be reduced by 1.3% per 1% Al. In addition, low-density steel is easily affected by various mechanisms, including transformation-induced plasticity (TRIP), twinning-induced plasticity (TWIP), shear band-induced plasticity (SIP), and microband-induced plasticity (MBIP) when subjected to external forces during use [6–9], which provides high strength and ductility at the same time. Low-density steel usually has high strong plastic product, the strong plastic product of Fe-30.5Mn-2.1Al-1.2C low-density steel developed by the Max Planck Metal Institute of Germany can reach 88GPa % [10]. The microstructure of low-density steel is affected by alloying elements. On one hand, the Mn element in low-density steel plays a role of solid solution strengthening [11], and on the other hand, Mn can also play a role in improving the stability of austenite. Similarly, Ni is also an austenite stabilizing element, which can improve toughness. Higher C content can improve the stability of austenite and enable low-density steel to obtain more retained austenite at room temperature [12], and Al shrinks the $\gamma$ region and promotes the formation of ferrite [13]. Although Mn and C elements will expand the $\gamma$-phase region, lesser amounts of Al elements also lead to the appearance of ferrite in the microstructure [14]. It is important to note that ferrite contains brittle phase $\delta$-ferrite. For some other steels, such as P91 [15], $\delta$-phase forms after austenitization, which will lead to a decrease in toughness.

Large amounts of Mn, Al, and other elements are added to low-density steel, which invites obvious segregation during the process of metal smelting. Therefore, it is difficult to guarantee the structure of low-density steel as a single austenite in actual production. As the density of Al differs greatly from that of Fe, it is easy to precipitate from the liquid phase first during smelting and solidification. As a result, the unsolvable Al may generate $\kappa$-carbides((Fe,Mn)$_3$AlC) with Mn and C without solid solution [16]. Unlike the common M$_2$X$_n$-type
and QX-type carbides that tend to precipitate near grain boundaries [17]. κ-carbide precipitates by spinodal decomposition and may precipitate both at grain boundaries and inside grains, so that the morphology and distribution of κ-carbide have a great influence on the mechanical properties of low-density steels. The unsolvable Al may also be precipitated as other compounds, such as the Fe–Al phase whose crystal structure is usually of B2 type and DO3 types [18]. According to Piston et al [19], B2(FeAl) and DO3(Fe3Al) phases formed on austenite grain boundaries during aging or through an ordering transformation within the ferrite phase of duplex steels. Controlled precipitation of B2 has the potential to significantly strengthen austenitic Fe–Mn–Al–C steels through grain refinement during controlled hot rolling, as well as precipitation of nano-sized B2 on austenite shear bands during subsequent annealing of cold-rolled sheet. Therefore, the size, morphology and distribution of these precipitates have a great influence on the properties of low-density steel.

The effect of precipitation on the mechanical properties of low-density steel is mainly reflected in hot working. Fe–Mn–Al–C low-density steels must contain a large amount of Al for weight reduction requirements. It can be seen from the Fe–C phase diagram that δ-ferrite generally only exists in the high temperature region above 1400 °C, but the addition of Al enlarges the δ phase region, so that the δ-ferrite structure can be stored to room temperature. δ-ferrite is difficult to be completely eliminated. The remaining δ-ferrite will destroy the continuity of the austenite matrix, easily induce cracking when the material is deformed, and make it difficult for austenite to fully exert the properties of TRIP, TWIP, etc to improve plasticity [20]. Therefore, research on how to eliminate the negative effect of δ-ferrite on low-density steel and improve rolling performance is conducive to further expanding the application range of low-density steel. The existing literature on Fe–Mn–Al–C series low-density steel mainly revolves around the process of deformation, and there are few studies on ingot casting and preparation before deformation; the current research on low-density steel containing Ni mainly comes from the work of Kim [21] and Piston [19] et al and lacks systematic research. On the one hand, Ni can reduce the proportion of ferrite in the structure by expanding the γ phase region of low-density steel; on the other hand, Ni can also promote the formation of hard B2 phase (NiAl) or (Fe, Ni)Al. The B2 phase maintains stability at temperatures above 600 °C–900 °C in the austenite recrystallization temperature range. Through rolling and annealing processes, this type of B2 phase can be transformed into a nanometer size, which can improve the strength of the material. The study of Kim et al [21] found that the addition of Ni element to the cold-rolled and annealed sheet of Fe–Mn–Al–C low-density steel can promote the formation of nano-sized B2 phase particles on the face-centered cubic matrix, so that this kind of sheet material can obtain 31.8% fracture elongation while maintaining the high strength of GPa level; Chumak et al [22] studied the Fe–Al–Ni ternary phase diagram, which provided a theoretical basis for studying the effect of adding Ni to low-density steel. The evolution of solidification and heat treatment phase structure of Fe–Mn–Al–C low-density steel containing Ni as a whole needs to be studied in depth.

Based on the above discussion, in the present study, Fe–15Mn–10Al–5Ni–0.8C low density steel ingots were designed and produced. The ingots as-cast were treated with solid solution, and the microstructure evolution after solution treatment was studied. In this study, the heat treatment conditions to completely eliminate δ-ferrites from Fe-15Mn-10Al-5Ni-0.8C steel ingots, thus obtaining single-phase austenite, were investigated. According to Chen et al [11]’s research on Fe-15Mn-10Al-xC composition series of low-density steels, the corresponding temperature range of the δ phase region is 1000 °C–1300 °C. Therefore, in order to eliminate the δ phase, the solution treatment temperature should be higher than 1000 °C but not more than 1300 °C. In addition, Chen et al pointed out that under current laboratory conditions, Fe–Mn–Al–C low-density steel ingots should be homogenized in the temperature range of 1100 °C–1250 °C before rolling. Therefore, in order to match the rolling process of low-density steel, the experiment set the solution treatment temperature range at 1100 °C–1250 °C. This experiment also analyzed the growth pattern of austenite during the solution treatment process by establishing a dynamic model of austenite grain growth, which provided a theoretical reference for the subsequent deformation processing technology, which has certain novelty and research value.

2. Experimental materials and methods

The materials used in this experiment were Fe–Mn–Al–C low-density steel ingots provided by the Institute of Metal Research, Chinese Academy of Sciences. The ingots were drum-shaped with a central height of 17 mm and a maximum bottom diameter of 40 mm. The nominal chemical composition of the ingots was as follows: 15 wt. % Mn, 10 wt. % Al, 0.8 wt. % C, and 5 wt. % Ni. The chemical components analyzed by ICP method are shown in table 1.

The sample used for metallographic observation and solution treatment was a 8.0 × 3 mm cylinder. Firstly, a 8.0 × 12 mm cylinder was machined from the ingot, which was then divided into 8.0 × 3 mm smaller cylinder. The original ingot is shown in figure 1(a) and the sampling position is shown in figure 1(b).
The machined cylindrical specimens were placed in a resistance furnace for solid solution treatment with holding temperatures of 1100, 1150, 1200, and 1250 °C, and holding times of 2, 4, 6 and 8 h, respectively, followed by water quenching. Model NWT X13B heat treatment equipment was used, with rated power of 8.0 kW, rated voltage of 220 V, and furnace chamber dimensions of 30 cm × 20 cm × 20 cm.

Specimens before and after solid solution treatment were taken, the bottom surfaces of which were ground, polished, etched, and then observed with an Axioscope 2 MAT optical microscope (Zeiss, Germany). The etching agent used was 8% nitric acid alcohol.

The microstructures of the Fe-Mn–Al–C low-density steel specimens were observed more clearly with a Zeiss-ultra-55 field emission scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS).

The low-density steel in this study contained a γ-phase (FCC), an α-phase, and a δ-phase (BCC). X-ray diffraction (XRD) was used for qualitative detection of the phases in the experimental alloy, with a Cu target, a voltage of 40 kV, a current of 150 mA, and a scan range of 5°–90°. After the detection, the obtained XRD data were then processed and analyzed with high score plus software to identify the phases by matching them with standard PDF cards.

To further determine the decomposition temperature of δ-ferrites, the specimens were examined using a Netzsch STA 449 F3 differential scanning calorimetry (DSC) analyzer. The detection was a heating process with a maximum temperature of 1500 °C and a heating rate of 10 °C min⁻¹.

Table 1. Elemental composition of sample analyzed by ICP method.

| Mn  | Al  | C   | Ni  | S   | P   | Fe  |
|-----|-----|-----|-----|-----|-----|-----|
| 15.6| 9.2 | 0.84| 4.7 | 0.02| 0.01| Bal.|

Figure 1. (a) Low-density steel ingot used for experiment. (b) Schematic diagram of the sampling position of the sample in the ingot.

Figure 2. Schematic diagram of phase size measurement.
According to ASTM standard, the intersection method was used to measure the grain size, as shown in figure 2. Multiple groups of metallographic photos were taken for each heat treated sample. Each sample was randomly selected from five fields of view with magnification of 100, and the intersection was measured based on the two diagonals of the rectangular image and its parallel harness. When the measuring line intersected and was tangent to the grain boundary, it was recorded as 1 intersection. If the measuring line obviously coincided with the meeting point of three grains, it was recorded as 1.5 intersection. If the end point of the measuring line intersected with the grain boundary, it should be recorded as 0.5 intersection. The measurement results of the five fields of view were then averaged for the same sample. If there were examples with large variance, a new field of view was added, and the measurements were re-taken. The length of the diagonal was divided by the number of austenite or ferrites intercepted to obtain $D_0$. The process was then repeated through the diagonal on the other side to obtain $D_1$. $D_0$ and $D_1$ were averaged to obtain the average grain size of the austenite in the specific field of view. For microstructures with more dendrites, the metallographic microstructure perpendicular to the cooling direction (dendrite growth direction) was selected for observation, and the maximum size of the dendrites (or grains) was used to calculate the average maximum size.

3. Experimental results and discussion

3.1. Microstructure

The metallographic structure of Fe-15Mn-10Al-5Ni-0.8C steel ingot is shown in figure 3(a). The original structure of the as-cast state was coarse dendrites. Dendrites were composed of two parts: the light white structure of the core and the gray structure of the outer layer. This structure was a typical peritectic structure, that is, the liquid phase reacted with the $\delta$ phase firstly precipitated from the liquid phase to form the $\gamma$ phase. It can be inferred that the light white structure in figure 3(a) is $\delta$-ferrite, and the gray structure is austenite. The SEM picture of the as-cast structure is shown in figure 3(b). After magnifying the peritectic structure, it could be found that there was still part of the matrix phase in the area where the core was wrapped. This is because the first precipitated $\delta$ phase grew in the form of dendrites. During the growth process, the dendrite arms interlaced each other, and the incompletely reacted liquid phase was enclosed in the solidified $\delta$-ferrite, thus forming local $\delta$-ferrite in turn wrapped the structure of austenite.

From figure 3(b), it could also be observed that the color depth of the core area of the peritectic structure was inconsistent. To ensure that this was the same phase, an EDS analysis was performed on it, as shown in figure 4 and table 2, where spots 1 and 3 were the central area and spots 2 and 4 were the matrix. From the element content of each point, it can be found that the C, Mn, and Ni content in the as-cast structure was basically close to the design composition, but the content of Al was significantly lower than the design composition, indicating that the segregation of Al element in this scanning area was more serious. The compositions of spots 1 and 3 were close and the contents of C and Mn was higher, even exceeding the design composition, indicating that the solid solubility of these two elements in the matrix was higher. The compositions of spots 2 and 4 were close and the contents of Al and Ni was higher, indicating that the core structure was ferrite and that the solid solubility of Al and Ni in ferrite was higher than in the matrix.

Figure 5 shows the XRD diffraction pattern of the low-density steel, identifying the $\gamma$ and $\delta$-ferritic phases contained in the steel. It should be emphasized that ferrites tended to precipitate other Fe–Al phases during the cooling process under the influence of Al. According to Piston et al.[19], the $\delta$-ferritic distributes at the center of...
Figure 4. Fe-15Mn-10Al-5Ni-0.8C steel as-cast structure: (a) SEM image; (b) EDS pattern of point 1; (c) EDS pattern of point 2; (d) EDS patterns of point 3; (e) EDS pattern of point 4.

Table 2. EDS analysis result of as-cast structure.

| Spot | C   | Mn  | Al  | Ni  |
|------|-----|-----|-----|-----|
| 1    | 0.75| 10.75| 5.50 | 5.61|
| 2    | 1.08| 17.36| 5.98 | 5.08|
| 3    | 0.8 | 10.47| 6.01 | 5.83|
| 4    | 1.07| 15.04| 4.41 | 4.51|
the austenite matrix in as-cast microstructure, and is prone to transform into B2 or DO3 phases during hot rolling or annealing. The δ-ferrite described in this experiment was a general term for these phases, and the diffraction pattern was matched with the Fe3Al diffraction card. In addition, the intensity of the diffraction peaks in figure 5 showed that the content of the γ-phase was greater than that of the δ-ferrite phase.

The relationship between heat flow and temperature changes is shown in figure 6 and table 3. During the entire endothermic process, the heat flow of the base material fluctuates at the beginning of the endothermic process, which was the process for the experimental instrument to adapt to the endothermic and exothermic reactions of various impurities in the sample, and then enter a steady heating process. When the temperature reaches 1304 °C, the first absorption peak appeared, which was the decomposition reaction of δ-ferrite; when the temperature reached 1385 °C, the second absorption peak appeared, which was the process of material melting.

| Peak area (J g⁻¹) | Start point (°C) | Finish point (°C) | Peak value (°C) |
|-------------------|------------------|-------------------|-----------------|
| Peak 1            | 2.768            | 1304.4            | 1319.0          | 1314.6          |
| Peak 2            | 28.44            | 1385.3            | 1428.6          | 1418.2          |

Figure 5. X-ray diffraction patterns of the Fe-15Mn-10Al-5Ni-0.8C steel.

Figure 6. Curve of relationship between heat flux and temperature of the Fe-15Mn-10Al-5Ni-0.8C steel.

Table 3. DSC results for Fe-15Mn-10Al-5Ni-0.8C ingot.
It could be seen that the decomposition temperature of \( \delta \)-ferrite exceeded 1300 °C, which was higher than the heat treatment temperature of general steel. Therefore, the heat treatment process designed in this experiment limits the temperature range to 1100 °C–1250 °C based on this observation, and an attempt was made to eliminate delta-ferrite by extending the holding time. For ordinary steel materials, the \( \delta \) phase was generally transformed into the \( \gamma \) phase during the solidification process and was not retained at room temperature. However, the composition of the low-density steel used in this experiment contains a large amount of Al, which narrowed the \( \gamma \) phase region, so that the \( \delta \) ferrite structure could be obtained at room temperature.

Figure 7. Laser confocal images of the Fe-15Mn-10Al-5Ni-0.8C steel: (a) 552 °C (b) 1351 °C (c) 1385 °C.

Figure 8. SEM image of microstructure after solid solution treatment at 1100 °C for 2 h.

Figure 9. Backscattered electron image of structure holding at 1100 °C for 2 h (perpendicular to the cooling direction of casting).
Figure 10. Line scan image of the specimen structure after holding at 1100 °C for 2 h.

Figure 11. Heat treatment structure of 1100 °C for: (a) 4 h, (b) 6 h and (c) 8 h.

Table 4. EDS spectrum of each point in figure 9.

| Location | C     | Mn   | Al   | Ni   |
|----------|-------|------|------|------|
| Spot 1   | 0.85  | 15.4 | 4.53 | 4.51 |
| Spot 2   | 0.7   | 12.47| 5.75 | 5.71 |
| Spot 3   | 0.82  | 13.07| 6.01 | 5.76 |
Figure 7 shows the laser confocal results for the low-density steel. Comparing the two heat absorption peaks of the DSC results, the outlines of ferrite and austenite could be vaguely observed at 552 °C. The ferrite phase started to melt when the temperature reached 1351 °C, which was slightly higher than the DSC results. When the temperature reached 1385 °C, the matrix started to melt and about half of the observed surface collapsed into the liquid phase, which is consistent with the starting temperature of matrix melting as indicated by DSC.

Figure 8 presents the microstructure of the specimen after holding at 1100 °C for 2 h. After heat treatment at 1100 °C, the overall ferrite microstructure showed a dendritic morphology, whereas the microstructure of specimens treated at higher temperatures did not contain dendrites. Therefore, the microstructure of the specimen treated at 1100 °C is discussed in particular. Figure 8(a) shows the structure along an observation angle parallel to the cooling direction. The corresponding energy spectrum and line scan results are shown in figure 9 and table 4. There were obvious differences in the element distribution between the two phases observed. Because Mn had higher solubility in austenite and Al had higher content in ferrite, the austenite and ferrite phases in the microstructure could be determined. Figure 10 shows the line scan results of the specimen microstructure after holding at 1100 °C for 2 h. The green, yellow, and blue curves represent Mn, Al, and Ni, respectively, to reflect the difference in element distribution between the austenite and ferrite phases more intuitively. Figure 8(b) shows the structure along the observation angle perpendicular to the cooling direction. Since this observation angle was convenient for measuring the size of austenite and ferrite dendrites, the microstructures of specimens treated at 1100 °C are presented along an observation angle perpendicular to the cooling direction.

![Figure 12](image1.png)

Figure 12. The ferrite size distribution of the structure after solution treatment at 1100 °C.

![Figure 13](image2.png)

Figure 13. SEM images of the structure after holding at 1100 °C for 8 h (a) ×500, (b) ×2000.
Figure 14. Structure after holding at 1150 °C for: (a) 2 h; (b) 4 h; (c) 6 h; (d) 8 h.

Figure 15. Structure after holding at 1200 °C for: (a) 2 h; (b) 4 h; (c) 6 h; (d) 8 h.
Figure 11 presents the microstructures of specimens treated at 1100 °C with holding times of 4, 6, and 8 h. Increasing the holding time to 6 or 8 h led to the partial dissolution of dendrite arms and a decrease in the density of the dendrites. Figure 12 shows the size distribution of ferrites in specimens treated at 1100 °C. The size distribution gradually became decentralized from a highly concentrated state with increasing holding time. With 2 h holding time, the ferrite size was concentrated at 60 μm. When the holding time was increased to 8 h, the ferrite size was mainly distributed around 100 μm, but with a wider size distribution range. The ferrites increased in size with increases in the holding time. This occurred because the phase transition temperature of δ-ferrite was very high (over 1300 °C), and the decomposition rate of dendrites was slow during the temperature holding process at 1100 °C. Although some of the fine dendrite arms melted, many large primary and secondary dendrite arms were retained, as indicated by the increase in average size. As shown in figure 13, partial recrystallization occurred in the microstructure held at 1100 °C for 8 h, with obvious grain boundaries appearing among the austenite (figure 13(a)). Meanwhile, κ-carbide precipitates in the form of pale white dots could be observed in austenite (figure 13(b)).

As shown in figures, the microstructure after heat treatment underwent significant changes when the temperature increased to 1150 °C and above. The previous dendrites transformed into massive grains and a small number of equiaxial crystals. Unlike the specimen treated at 1100 °C where only dendrites were observed, two distinct phases could be distinguished in the specimens treated at 1150 °C and above. Specifically, the phases were the austenite matrix of light color and large volume and the ferrites of dark color and small volume distributed at the grain boundaries in the austenite matrix. During holding at 1150 °C for 2 h, the austenite dendrites recrystallized and transformed into similar equiaxial grains, and part of the austenite was polygonized. The δ-ferrites retained a certain dendrite morphology with a tendency to evolve into bar and equiaxial crystals and were distributed at the austenite grain boundaries. When the holding time was extended at 1150 °C to 6 h, the ferrites disappeared, and the austenite was polygonized, forming grain boundaries of 120°. When the solid solution temperature and holding time reached or exceeded 1200 °C and 2 h, respectively, a large amount of austenite was polygonized and the δ-ferrites formed finer blocks and were distributed at the austenite grain boundaries. When the solid solution temperature and holding time reached or exceeded 1250 °C and 2 h respectively, the austenite was fully polygonized and δ-ferrite was no longer presented in the microstructure.

The austenites observed at 1150 °C–1250 °C all contained white κ-carbide precipitates, as well as precipitation-free zones at the grain boundaries. As shown in figure 17, after holding at 1150 °C for 6 h, the austenite was completely
polygonized, with fine white rod-like particles precipitating inside the austenite and at the grain boundaries. The particles can be identified as $\kappa$-carbides based on previous studies by Lu et al, Zhang et al, Mapelli et al and moon et al [23–26]. When the temperature was elevated to 1200 °C, $\kappa$-carbides were observed in the austenite after only 2 h. Extending the holding time to 6 h, $\delta$-ferrites disappeared completely, and obvious precipitation-free zones were observed near the grain boundaries. When the solid solution temperature and holding time reached or exceeded 1250 °C and 2 h, respectively, $\delta$-ferrites disappeared from the microstructure, accompanied by the growth of $\kappa$-carbides at the grain boundaries and wider precipitation-free zones.

Compared with the carbides precipitated at 1100 °C, the $\kappa$-carbides were finer in size and diffusely distributed. A certain angle formed between the carbides, which was basically the same in the same grain. Because the grains differed in orientation, the angle varied to some extent, but still presented strong regularity. These angles were between 60° to 90°, indicating that the austenite underwent spinodal decomposition during heat treatment. Spinodal decomposition mostly occurred in the process of austenite recrystallization. Supersaturated $\kappa$-carbides aggregate first in the parent phase and, upon reaching a certain concentration, precipitate from the parent phase to form a new precipitation phase. The phase interface was not obvious in the initial stage of spinodal decomposition, compared with the pale white dotted carbides observed in the specimens held at 1100 °C for 8 h. Thus, it could be deduced that an increase in temperature led to a more completed spinodal decomposition.

Figure 17. SEM images of $\kappa$-carbide structure after holding at: (a) 1150 °C for 6 h; (b) 1200 °C for 2h; (c) 1200 °C for 8 h; (d) 1250 °C for 2h; (e) 1250 °C for 8 h.
3.2. Austenite crystal growth kinetics

The grain sizes of austenite and ferrite were recorded. The relationship between austenite grain size and holding time under isothermal conditions is expressed by the Beck equation, and equation (1) states the original Beck equation:

\[ D = D_0 = K t^n \]  

where \(D\) is the average grain size after austenite grain growth (\(\mu m\)); \(D_0\) is the average size of the original austenite grain (\(\mu m\)); \(K\) is the growth rate of austenite grains; \(n\) is the growth index of austenite grains; \(t\) is the holding time (s). \(K\) and \(t\) are both functions of temperature \(T\). Under the condition that the original austenite grain size is much smaller than the grain size after growth, the Beck equation can be simplified to the form of equation (2).

\[ D = K t^n \]  

Take logarithms from both sides of equation (2) to obtain equation (3).

\[ \ln D = \ln K = n \ln t \]  

The relationship between the holding time \(t\) and the grain size \(D\) is represented by the curve in figure 18(a). Through linear regression of the experimental data, the relationships between \(\ln D\) and \(\ln t\) under different heating conditions were obtained, as shown in figure 19(b). If \(\ln D\) is linearly related to \(\ln t\), \(n\) can be determined from the slope of the line and \(K\) from the intercept of the line (the intercept is \(\ln K\)).

The values of \(n\) and \(K\) at four groups of temperatures were obtained from the relationship between \(\ln D\) and \(\ln t\), as shown in table 5. The average of \(n\) value was 1.06, and the calculated variance was 0.016, which showed that \(n\) fluctuated little in the range of 1100–1250 °C. Therefore \(n\) could represent the grain growth index of the whole temperature range. The value of \(K\) was very small over the whole temperature range (no greater than 0.05), indicating that the grain growth of low-density steel was slow at high temperature. However, the austenite grain size after heat treatment was large because the experiment used a longer holding time to eliminate \(\delta\)-ferrite. According to equation (2), when the holding time was too long, \(t\) mainly contributed to the growth behavior of austenite, and \(K\) had little effect on the average grain size. Unlike \(n\), \(K\) changed obviously with increasing temperature. When the temperature was in the range of 1100 °C–1150 °C, the order of \(K\) was only 0.001, indicating that austenite had a strong coarsening resistance at this temperature. When the temperature reached 1200 °C or 1250 °C, the order of \(K\) increased to 0.01, indicating that the coarsening resistance of low-density steel decreased significantly at this temperature. Especially at 1250 °C, austenite grains were significantly larger than those at other temperatures, as shown in figure 19(a). Therefore, 1250 °C could be considered as the critical temperature for austenite growth in Fe-15Mn-10Al-5Ni-0.8C steel. To sum up, the overall growth rate of austenite was slow during solution treatment. However, when the temperature rose to 1200 °C–1250 °C, the growth rate increases significantly.

The heterogeneity of austenite grain size was the main driving force in promoting austenite growth. Due to the high energy at the grain boundary, the grain growth behavior always develops in the direction of reducing the
During grain growth, grain boundaries migrate by atomic diffusion. On the one hand, this behavior was characterized by thermal activation under the influence of temperature. On the other hand, grain boundary migration was related to the interaction with other phases. In this research,  δ-ferrite remained stable at high temperature and was mostly distributed at the austenite grain boundary, which had an important effect on austenite grain boundary migration. At 1100 °C, δ-ferrite was mainly distributed on the matrix in the form of dendrites, which seriously limited austenite growth. Only when the dendrite arm of ferrite was dissolved could part of the austenite grow by the migration of the grain boundary to form a polygonal grain boundary, as shown in figure 11(c). Therefore, curve of 1100 °C in figure 19(a) changed little with time increasing, and the overall grain size was obviously smaller than for the other three curves. As the temperature rose, δ-ferrite dendrites were broken and became dissolved, and distributed along the austenite grain boundary in a strip shape, which weakened the hindrance to the grain boundary movement. Especially at 1150 °C and 1200 °C, after holding for 6 h, ferrite was completely dissolved, and austenite could grow rapidly. As a result, the curve shown in figure 19(a), the curve rises sharply when t = 6. When the temperature rose to 1250 °C, the ferrite had changed into a needle shape or slender rod shape after holding for 2 h and had been completely dissolved after holding for 4 h. Thus, the K value for this temperature increased significantly, and curve of 1250 °C showed a steep upward trend over the whole range.

According to the Sellars model, as shown in equation (4), the growth model of austenite grains in C-Mn steel can be described as:

\[ D^k - D_0^k = At \exp\left(-\frac{Q}{RT}\right) \]

where \( D \) is the average size of an austenite grain after growth (μm); \( D_0 \) is the average grain size of the original austenite (μm); \( T \) is the temperature (K); \( t \) is the holding time (s); \( R \) is the gas constant \( (R = 8.314 \text{ J mol}^{-1}) \); \( Q \) is the activation energy of austenite crystal growth (J/mol); \( A \) and \( k \) are constants of the material.

Since the austenite grains after growth are much larger than the original austenite grains, \( D_0 \) can be ignored. The simplified equation (5) is then obtained.

\[ D^k = At \exp\left(-\frac{Q}{RT}\right) \]

Take logarithms of both sides of equation (5) to obtain equation (6).

\[ \ln D = \ln A + \frac{\ln t}{k} - \frac{Q}{kRT} \]

The relationship between lnD and 1/T under different holding times is shown in figure 18.
Perform linear regression on the curve to calculate the values of \( k, A \) and \( Q \). During continuous heating solution treatment, \( \ln D \) and \( 1/T \) have a linear relationship, which proves that the model is valid, in which case \( k = 27.75; Q = 365 \text{ KJ mol}^{-1}; A = 2.01 \times 10^{20} \). The calculation results of \( k, A, \) and \( Q \) are substituted into the simplified Sellars model equation to obtain equation (7).

\[
D^{27.75} = 2.01 \times 10^{20}t \exp \left( -\frac{365}{RT} \right) 
\]

(7)

4. Conclusion

(1) The \( \delta \)-ferrite could be completely eliminated by solid solution treatment under the following conditions: water cooling after holding at 1150 \(^\circ\)C for 6 h, or holding at 1250 \(^\circ\)C for 2 h.

(2) The \( \kappa \)-carbide precipitation could be observed in solid solution microstructure. At the same holding time, \( \kappa \)-carbides grew with the increase of temperature; At the same temperature, \( \kappa \)-carbides grew up with the extension of holding time.

(3) The value of the austenite growth index \( n \) for the Fe-15Mn-10Al-5Ni-0.8C low-density steel ingots stabilized at 1. The austenite growth rate \( k \) increased with higher heat treatment temperature and had a maximum value at 1250 \(^\circ\)C. In this study, the activation energy for austenite growth was 365 \text{ KJ mol}^{-1}, which was significantly higher than the diffusion activation energy of C in austenite (155 \text{ KJ mol}^{-1}), indicating that austenite growth during heat treatment of low-density steel was not only influenced by the diffusion behavior of elemental C but is also the result of the combined effect of the diffusion of C, Mn, Al, and other elements.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Author contributions

Conceptualization, Z W (Zhongjun Wang) and W L (Weijuan Li); methodology, K F (Kuijun Fu); software, Z G (Zhihe Guo); validation, F H (Fengya Hu), and J W (Jiaji Wang); formal analysis. All authors have read and agreed to the published version of the manuscript.

Conflicts of interest

The authors declare no conflict of interest.

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