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Article

In-Situ Observation of the Austenite Grains Growth Behavior in the Austenitizing Process of Nb-Ti Micro-Alloyed Medium Manganese Steel

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Abstract

In this paper, the austenite grains growth behavior in the austenitizing process of Nb-Ti micro-alloyed medium manganese steel was studied through in-situ observation by high temperature laser confocal microscope. The results show that the average austenite grain sizes change from about 3 μm at 1050°C to over 50 μm at 1250°C. When the grain boundary is a small angle grain boundary, one grain boundary will split into several dislocations. With the extension of heating time, the lattice orientation difference further decreases, and the remaining dislocations may merge into new grain boundaries. The most suitable heating temperature for the medium manganese steel in this paper is from 1100°C to 1150°C. This takes into account influences such as grain size, grain boundary damage, and deformation resistance.

Keywords: medium manganese steel; austenite; precipitate; grain size distribution; Nb-Ti micro-alloyed

1. Introduction

In recent years, manganese steel has emerged as a prominent research area owing to their potential to achieve a wide range of mechanical properties [1–5]. Medium manganese steel, containing manganese ranging from 3% to 12%, has become a leading candidate for the next generation of advanced high-strength steels due to its optimal balance between mechanical performance and cost efficiency. [6–10]. The superior balance of high strength, toughness, and ductility in medium manganese steels originates from the transformation induced plasticity effect, which is activated by the austenite transform to martensite during deformation. The microstructures of medium manganese steel are mainly composed of tempered martensite and retained austenite (RA). The ductility was improved by introducing metastable austenite via the enrichment of C and Mn during inter-critical annealing [11–13]. From the perspective of rolling process, the hot rolling temperature has a significant effect on the microstructure and properties, which are essentially caused by grain boundaries. Yu et al. found that with the decrease of hot rolling temperature from 1150 °C to 900 °C, the average austenite grain size was decreased from 40 μm to 20 μm and average width of martensite lath was refined from 400 nm to 250 nm [14]. Therefore, it is very important to study the austenite grain size control of medium manganese steel at high temperature.

Grain boundary engineering is an advanced materials science aimed at optimizing the properties of polycrystalline metals by deliberately modifying their grain boundaries [15–17]. Kokkula et al. studied the effect of grain boundary engineering on the microstructures of high Mn steel, and found the grain boundary engineering specimen exhibited higher fraction of $\Sigma 3$ boundaries and its variants $\Sigma 9$ and $\Sigma 27$, larger twin related domain size, and greater number of grains than other specimens [18]. This is attributed to the greater extent of multiple twinning through the activation of

strain induced grain boundary migration. The austenite grain size control during hot rolling heating process is the critical first step for grain boundary engineering, and the size and distribution of austenite grains have a significant influence on the microstructure and properties during subsequent rolling and cooling. Especially for medium manganese steel that has a low phase transformation temperature, a high tendency for austenite grain growth, and easy damage to grain boundaries, controlling the heating process is very important.

Grain growth is an important research direction in the field of materials, and the driving force for grain growth is centered on the reduction of grain boundary energy according to existing research results [19–21]. Rohrer reviewed the important role of the grain boundary energy in complexion transformation, and found the change in grain boundary energy causes a change in the grain boundary character distribution [22]. There is also evidence that higher energy grain boundaries transform to the lower energy complexions at a lower temperature than low energy grain boundaries. In recent years, the influence of solute segregation at the grain boundaries on grain boundary energy has received much attention. Lejček and Hofmann studied the grain boundary segregation using the standard enthalpy and the standard entropy through thermodynamic model, and of grain boundary segregation [23]. The iron-based binary system and predicted examples were compared with the data published in the literature, and good consistency was obtained. At present, research on the kinetics of grain growth mainly focuses on the promoting effect of temperature and the hindering effect of precipitation on grain growth of steady state growth stage. However, the research on the mechanism of grain growth in the early growth stage is still insufficient, i.e., just after the austenite phase transformation and the impingement of grains. To control microstructures with multiple co-existing complexions in a predictive way, it is necessary to better understand, at a theoretical and experiment level.

This paper uses high temperature laser confocal microscope to in-situ observation of the high temperature austenite grains growth behavior in the austenitizing process of Nb-Ti micro-alloyed medium manganese steel. Thermodynamic calculations were performed on the content and average diameter of Nb-Ti precipitates. The diameter frequency and area percentage of austenitic grains with different heating temperatures and time were statistically counted to study recrystallization, grain growth, and grain merging. The growth behavior and mechanism of austenite grains in medium manganese steel was discussed.

2. Experimental Materials and Methods

The forged billet of medium manganese steel with a size of 120×130×130 mm³ was heated to 1200°C and isothermal for 120 minutes. Then, the steel billet was hot rolled to 30mm thick in a Φ450 mm hot rolling mill and online quenched to room temperature, with the initial and finish rolling temperature being 1050°C and 900°C, respectively. The chemical composition of the medium manganese steel is listed in Table 1. The phase zone and temperature at which precipitates dissolve is calculated using Thermo-Calc software.

Table 1. Chemical composition of the experimental steel (wt.%).

C	Si	Mn	P	S	Nb	Ti
0.08	0.25	4.52	0.011	0.0019	0.018	0.036

To study the austenite grain growth during heating and isothermal processes of medium manganese steel, high temperature laser confocal microscope (HTLCM, VL3000DX-SVF17SP/15FTC) was used for in-situ observing the dynamic migration and growth of austenite grains in high temperature. The HTLCM specimens were cut from the 30mm thick plate with a size of Φ5×3 mm³. The specimens were manually and mechanically polished to ensure the flatness and cleanliness of the observed surface. The specimens were heated to 1050°C, 1100°C, 1150°C, 1200°C, and 1250°C and isothermal for 600 s with the heating rate of 10°C/s, respectively. Typical images were selected to statistical calculate the average grain size and area percentage with different temperature and time.

3. Results

3.1. Growth Behavior of Austenite Grains During Heating Process

Figure 1 shows the austenite grains heated to 1050°C then isothermal for 600 s at a heating rate of 10°C/s, and the average austenite grain size is very small, only about 3 μm . The reason why the grains are so small under these conditions is that the phase transformation has just ended, and the grains have collided, so they did not have time to grow at this temperature.

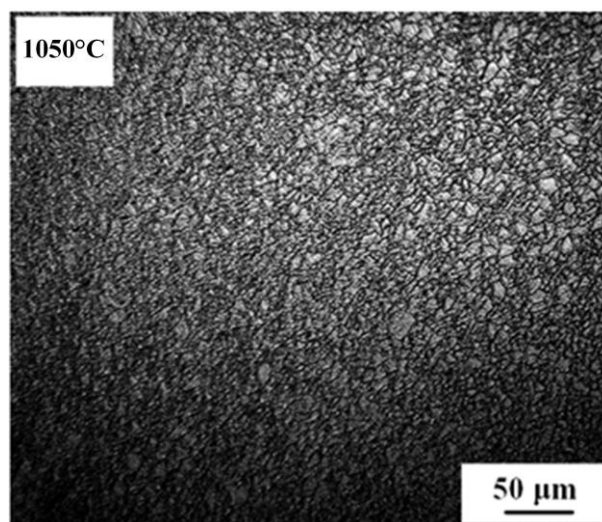


Figure 1. Images of austenite grains isothermal at 1050°C for 600 s.

Figure 2 shows the austenite grain image at high temperature, which was heated to 1100°C, 1150°C, 1200°C, and 1250°C then isothermal for 200 s, 300 s, 400 s, and 600 s at a heating rate of 10°C/s. The 4 images Figure 2 (a), (e), (i), and (m) show the austenite grain size of isothermal for 200 s, and the austenite grain boundaries become clear until the temperature reaches 1200°C. However, the austenite boundaries have become clear when the isothermal time increase to 300 s, and the grain size at 1100°C is still very small, as shown in Figure 2 (b), (f), (j), and (n). When the isothermal time is increased to 400 s, the austenite grain boundaries have fully emerged as shown in Figure 2 (e), (g), (k), and (o), and obvious coarsening phenomena appear at the austenite grain boundaries when the insulation time is increased to 600 s, as shown in Figure 2 (d), (h), (i), and (p). From the results of HTLCM, it can be seen that the austenite grain boundaries become increasingly clear and coarsen with the extension of isothermal time, and the austenite grain size continuously increases simultaneously. It can also be seen in Figure 2 (d), (h), (i), and (p) that the austenitic grain size increases with the isothermal temperature and changes significantly at 1100°C, and this change is reflected in the transformation from small grains to large equiaxed grains. Meanwhile, the traces of austenite grain merging and grain boundary migration become increasingly apparent above 1150°C, while the austenite grain boundaries gradually become straight.

To investigate the influence of isothermal temperature on the austenite grain size distribution, the austenite grain size distributions and area percentage of different specimens were statistically analyzed, as shown in Figure 3 and 4. Figure 3 shows the specimens of 1100°C and 1150°C, as the grain size of the specimens at these two temperatures has significantly increased. Figure 3(a) and (b) show that the grain sizes of the specimen isothermal at 1100°C for 200 s and 300 s are all smaller than 30 μm . Meanwhile, some grains are larger than 30 μm appeared and less than 3% grain sizes are above 80 μm isothermal at 1100°C for 400 s and 600 s, as shown in Figure 3(c) and (d). Although some large grains appeared, the reason why the average grain size did not increase significantly is that the weight of large grains is small, and the grain size is mainly determined by small grains. Figure 3(e) shows that the grain size of the specimen isothermal at 1150°C for 200 s is all smaller than 80 μm . As

for Figure 3(f), (g), and (h), the grain size distributions of the specimen isothermal at 1150°C for 300 s, 400 s, and 600 s are similar, and mainly between 20 μm and 50 μm with a few exceeding 80 μm .

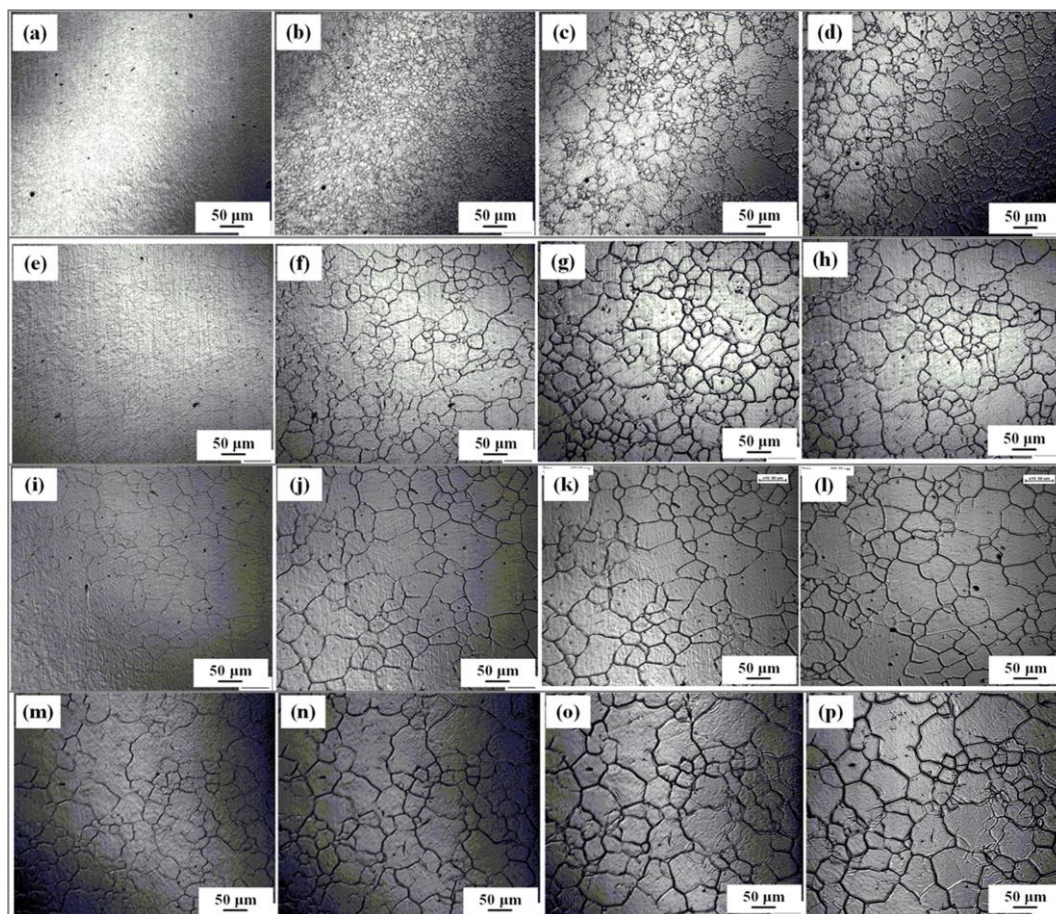


Figure 2. Influence of isothermal time on austenite grain size. (a~d) 200 s, 300 s, 400 s, 600 s for 1100°C, (e~h) 200 s, 300 s, 400 s, 600 s for 1150°C, (i~l) 200 s, 300 s, 400 s, 600 s for 1200°C, (m~p) 200 s, 300 s, 400 s, 600 s for 1250°C.

Figure 4 shows the specimens of 1200°C and 1250°C, as the grain size of the specimens at these two temperatures remains stable without significant increase. It is shown in Figure 4(a) that the austenite grain size is mainly concentrated in the range of 30-35 μm of the specimen isothermal at 1200°C for 200 s, and the grain size is between 20 μm and 80 μm when the isothermal time were 300 s, 400 s, and 600 s, as shown in Figure 4(b), (c), and (d). Finally, When the isothermal temperature is 1250°C, The range of grain size concentration is similar, but the dispersion is more uniform, as shown in Figure 4(e), (f), (g), and (h). This indicates that the rapid grain growth stage was completed within 200 s at temperatures of 1200°C and 1250°C, and the grain size became relatively stable.

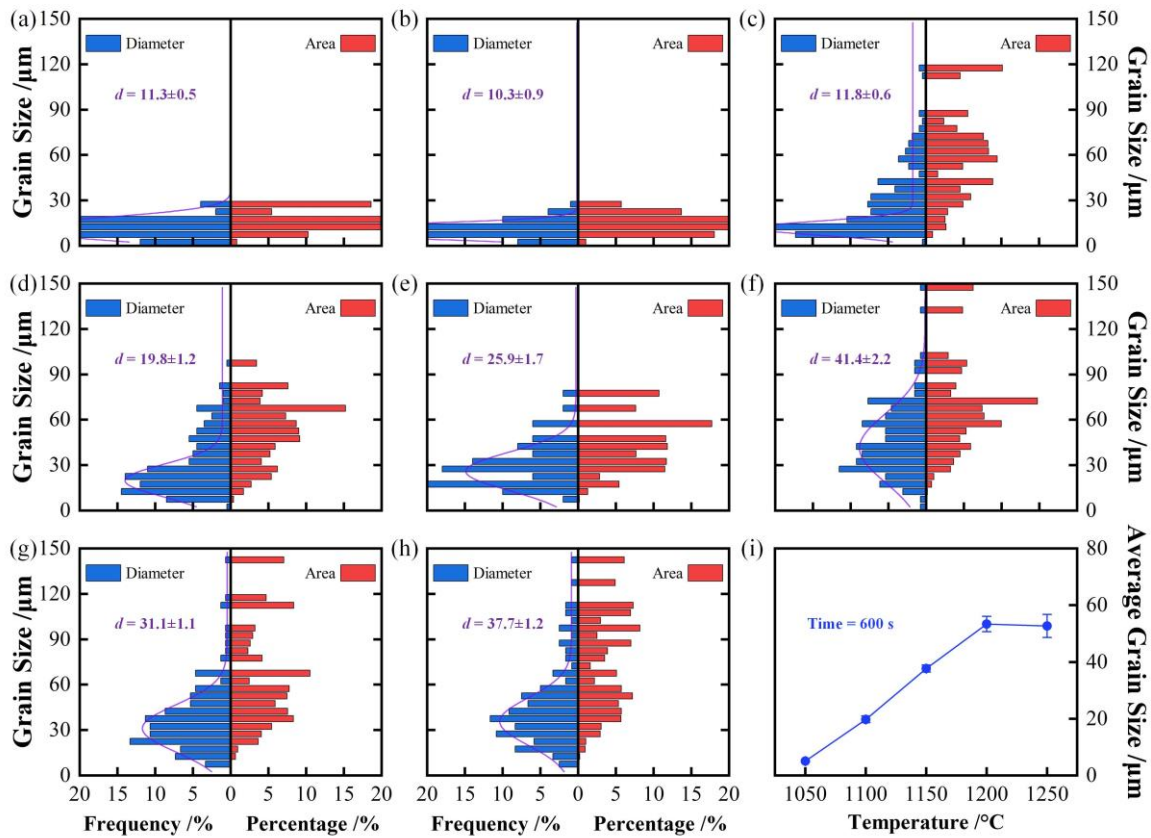


Figure 3. Austenite grain size distribution at 1100°C and 1150°C.(a) 1100-200, (b) 1100-300, (c) 1100-400, (d) 1100-600,(e) 1150-200, (f) 1150-300, (g) 1150-400, (h) 1150-600, (i) Average grain size.

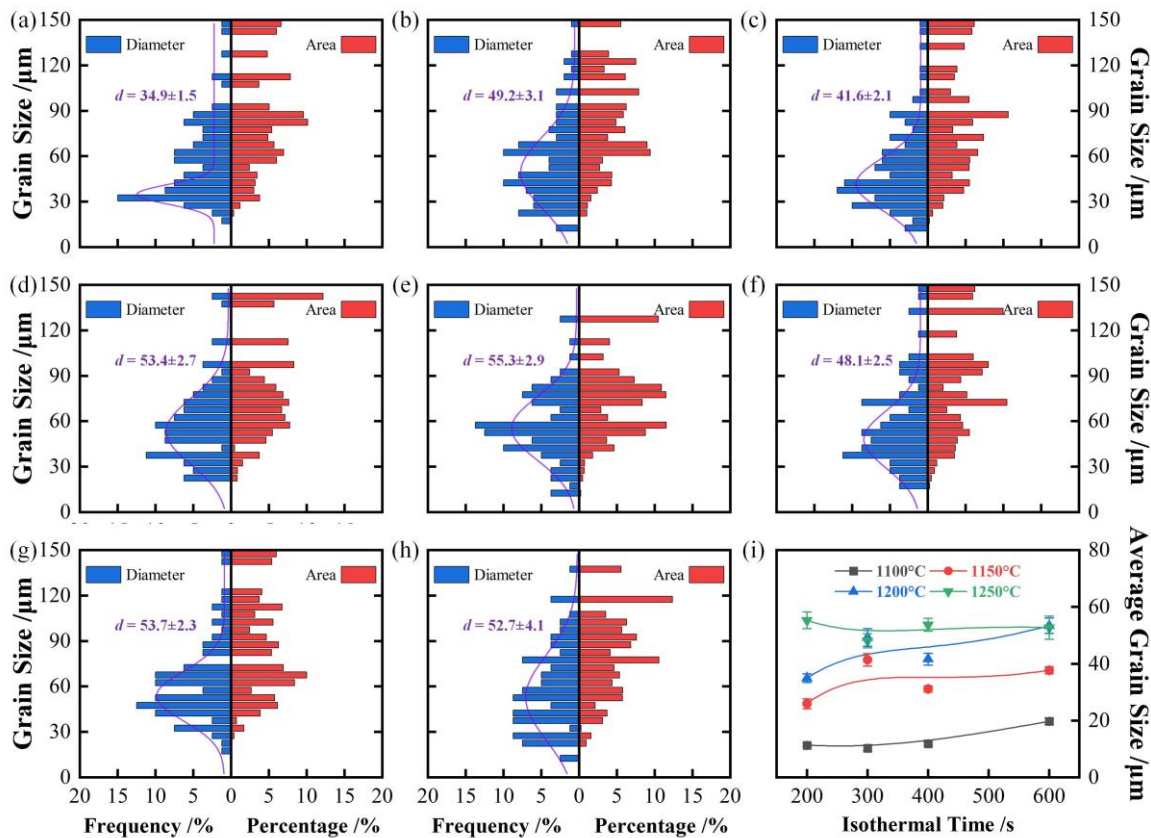


Figure 4. Austenite grain size distribution at 1200°C and 1250°C.(a) 1200-200, (b) 1200-300, (c) 1200-400, (d) 1200-600,(e) 1250-200, (f) 1250-300, (g) 1250-400, (h) 1250-600, (i) Average grain size.

The average austenite grain sizes statistics from Figure 3 and Figure 4 are listed in Table 2. The average austenite grain sizes of 1100°C are about 10 μm within 400 s and about 20 μm at 600 s. However, the average austenite grain sizes of 1150°C are about 25 μm at 200 s and about 35 μm between 300 s and 600 s. Besides, However, the average austenite grain sizes of 1200°C are about 35 μm at 200 s and about 45 μm between 300 s and 600 s. Finally for the specimen isothermal at 1250°C, the average austenite grain sizes remain stable at around 50 μm.

Table 2. Average austenite grain size of different specimens (μm).

	200 s	300 s	400 s	600 s
1100°C	11.3±0.5	10.3±0.9	11.8±0.6	19.8±1.2
1150°C	25.9±1.7	41.4±2.2	31.1±1.1	37.7±1.2
1200°C	34.9±1.5	49.2±3.1	41.6±2.1	53.4±2.7
1250°C	55.3±2.9	48.1±2.5	53.7±2.3	52.7±4.1

From the above results, it can be seen that 80 μm is a clear demarcation point to distinguish between large and small grains. Therefore, we calculated the proportion of grain sizes and the percentage of grain area greater than 80 μm, and listed them in Table 3. It can be seen that there are almost no grains larger than 80 μm at 1100°C, and the proportion of grain sizes and the percentage of grain area larger than 80 μm are increase with the isothermal time, which are from 0% to 13.3% and to 49.7%, respectively. However, the proportion of grain sizes and the percentage of grain area larger than 80 μm are almost the same for 1200°C and 1250°C, which are all about 20% and 50%, respectively.

Table 3. The diameter frequency and area percentage of the grain size larger than 80 μm (%).

	200 s		300 s		400 s		600 s	
	Diameter	Area	Diameter	Area	Diameter	Area	Diameter	Area
	Freque	Percentag	Freque	Percentag	Freque	Percentag	Freque	Percentag
	y	e	y	e	y	e	y	e
1100°C	0.0	0.0	0.0	0.0	2.7	22.6	2.0	11.0
1150°C	0.0	0.0	6.9	28.0	5.3	31.1	13.3	49.7
1200°C	21.3	53.7	19.0	51.1	16.0	49.3	17.5	46.5
1250°C	17.5	41.1	20.0	53.5	22.5	53.3	21.3	51.8

3.2. Thermodynamics Calculation of Phase Zones and Carbides Average Diameter

Figure 5 (a) shows the phase zone of the steel in the temperature range from 0°C to 1400°C. FCC phase disappears at 296°C and reappears at 430°C, BCC phase disappears at 752°C. and cementite disappears at 605°C. Furthermore, the weight fraction of precipitates below 1000°C is about 0.065% and precipitate reduce to disappear from about 1000°C to 1200°C, as shown in Figure 5 (b). The original specimen was on-line quenched to room temperature at 900°C after hot rolling, therefore, most of the precipitates have already precipitated at this temperature and will remain in the microstructure.

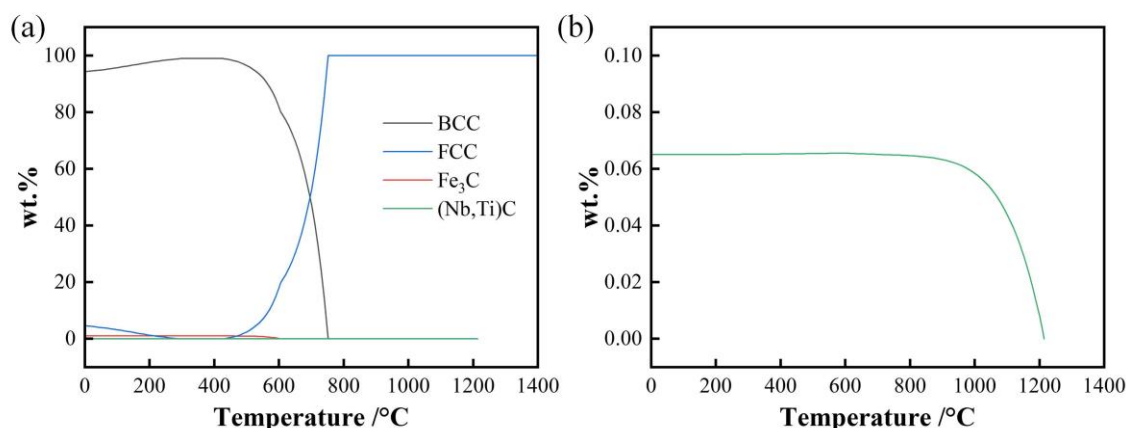


Figure 5. Phase zones of the steel calculated by Thermal-Calc(a) Full range of the steel, (b) Weight fraction range of precipitates.

Figure 6 shows the average diameter of precipitates during heating process at different isothermal temperature. Figure 5 shows that the dissolution temperature of precipitates is approximately around 1200°C. At a holding temperature of 1050~1150°C, a portion of the precipitates nucleate and grow during the heating and holding stage, resulting in a continuous increase in their average diameter. However, during the cooling process, fine precipitates begin to precipitate extensively, leading to a rapid decrease in the average diameter. When the holding temperature exceeds 1200°C, the precipitates dissolve completely during heating, and no precipitation occurs during subsequent cooling.

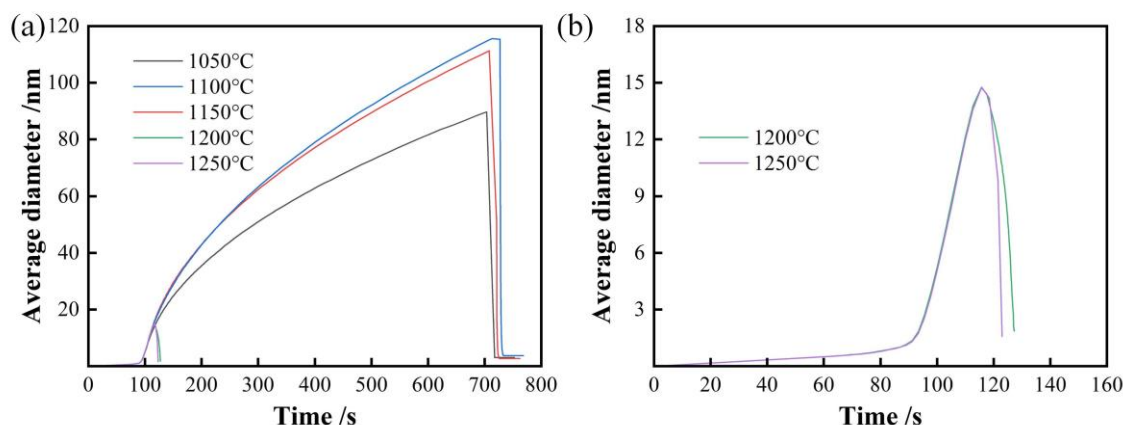


Figure 6. Average diameter of precipitates during heating process at different isothermal temperature(a) The specimens of all isothermal temperature, (b) magnification of 1200°C and 1250°C.

4. Discussion

4.1. Effect of Isothermal Temperature on Austenite Grain Growth Behavior

It is well known that temperature has a significant influence on grain growth, i.e., higher temperatures lead to faster growth rates. One primary reason is that elevated temperature accelerates atomic diffusion, leading to increased grain boundary mobility. On the other hand, the free energy difference between impurities in the grain interior and at the grain boundaries decreases, weakening solute segregation at the boundaries and thereby reducing the solute drag effect on grain boundary migration. Both of these influences increase the grain boundary energy, destabilizing the grains and driving their tendency to grow [24–27]. Based on the above results, the change in austenite grain of

different isothermal temperature changes under the same isothermal time is significantly greater than that of different isothermal time at different isothermal temperatures. For instance, the average austenite grain sizes change from about 3 μm at 1050°C to over 50 μm at 1250°C, but increasing the isothermal time from 200 s to 600 s did not result in a significant increase in austenite grain size, as shown in Figure 3 (i) and Figure 4(i). At 1200°C and 1250°C, the proportion of large grains with a diameter greater than 80 μm did not increase significantly, indicating that the large grains became relatively stable after 200 s for the specimens heating over 1200°C.

Refining the grain size is one of the most successful processing strategies to improve the properties of polycrystalline solids, and the fact that precipitates play a significant role in grain refinement has also been extensively studied by many researchers. The current research results indicate that the interaction between precipitates and grain boundaries hinders grain boundary movement, thereby hindering grain growth. Assuming that the dispersed phase particles are spherical, the maximum tensile force $F_{\max}$ exerted by the precipitates in the opposite direction of grain boundary movement on grain boundary movement is:

$$F_{\max} = \pi r \gamma_b \quad (1)$$

Assuming that precipitates are uniformly distributed in the metal with a density of N per unit volume, which means the grain boundary intersects with $2rN$ precipitates per unit area. The total constraint force $F'_{\max}$ acting on grain boundary movement per unit area is:

$$F'_{\max} = 2rN F_{\max} = 3f\gamma_b/2r \quad (2)$$

where r is the radius of precipitates, γ_b is the grain boundary energy per unit area, and f is the volume percentage of precipitates [28,29].

It can be clearly seen from Eq. (2) that the total constraint force increases with the increase of volume percentage of precipitates f and with the decrease of precipitates radius r . According to the calculation of precipitates content and diameter in this paper, the volume percentage of precipitates decrease with the increase of isothermal temperature from 1050°C to 1250°C (Figure 5), and the precipitates radius at 1050°C is smaller than 1100°C and 1150°C (Figure 6). From these two aspects, the pinning effect of the precipitate is strongest at 1050°C, weakened at 1100°C and 1150°C, and basically lost at 1200°C and 1250°C. This conclusion also corresponds to the phenomenon that the grain size increases linearly between 1050°C and 1200°C, while between 1200°C and 1250°C remain basically unchanged.

Moreover, the grain boundary width increases with the temperature, especially for 1250°C, indicating that grain boundaries are damaged at high temperatures. Although the size of grains increases with temperature, the proportion of large grains stabilizes at around 50% at 1150°C. This indicates that under this microalloying composition condition, the optimal isothermal temperature range for the medium manganese steel is between 1100°C and 1150°C.

4.2. Austenite Grain Growth Mechanism During Isothermal

Medium manganese steel was composed of martensite after quenching due to the shear transformation of austenite, and martensite transform to austenite during heating. Therefore, it is necessary to first understand the relationship between these two transformations. Both Dong et al. [30,31] and Yu et al. [14] found that austenite nucleates between martensitic lath and at grain boundaries during heating for medium manganese steel, with many nucleation sites and very small austenite grains. It can also be seen in Figure 1 that the austenite grain sizes are very small, indicating that there are a large number of nucleation sites in the phase transformation, which belongs to the position saturation phase transformation. Besides, Yang et al. found that martensite blocky transform into austenite at a heating rate of 10°C/s to 50°C/s [32]. Moszner et al. intensively studied the role of Mn redistribution in the reverse transformation of the binary Fe-10Mn alloy, and spotted that the formation of austenite proceeds in an interface-dominated manner on fast heating [33]. On slow heating, austenite reversion takes place in a dual-step process, where diffusional mechanisms

dominate the first stage and the second stage is controlled by interface migration. For the specimens in this study, the orientation difference between the austenite generated by these two transformations and the nearby martensitic matrix should be small. Furthermore, the newly generated grain boundaries are mainly small angle grain boundaries. In general, when the heating temperature is low, a large part of the grain boundaries between the transformed austenite grains are small angle grain boundaries, which determines the characteristics of austenite growth during the isothermal process and provides conditions for merging.

The isothermal time can also strongly affect grain growth, and different grains have different growth mechanisms. It can be clearly seen from Figure 2 (b) that the microstructure was all composed of small austenite grains, and many large austenite grains appeared after 100 s as shown in Figure 2 (c). This indicates that the mechanism of austenite grain growth is through merging. Not all grains can merge into one large grain, and the prerequisite for merging into a large grain is that the orientation difference between these grains before merging cannot be large. Another way of grain growth is one grain engulfs another grain, which is the result of grain boundary migration, as shown in Figure 2 (n) and (o). This is because the orientation difference of grains is relatively large, and can only rely on grain boundary migration. The schematic diagrams of these two grain growth mechanisms are shown in Figure 7. When the grain boundary is a small angle grain boundary, there is a strong tendency for multiple grains to merge into one large grain. The merged large grain will generate many dislocations at the original grain boundary position, which means that one grain boundary will split into several dislocations, and then it will appear as a complete grain when observed. With the extension of heating time, the lattice orientation difference further decreases, and the dislocations have not completely disappeared. The remaining dislocations may merge into new grain boundaries, resulting in the appearance of some shallow grain boundaries inside the large grains. When the grain boundary is a large angle grain boundary, only the phenomenon of annexing can occur, that is, grain boundary movement.

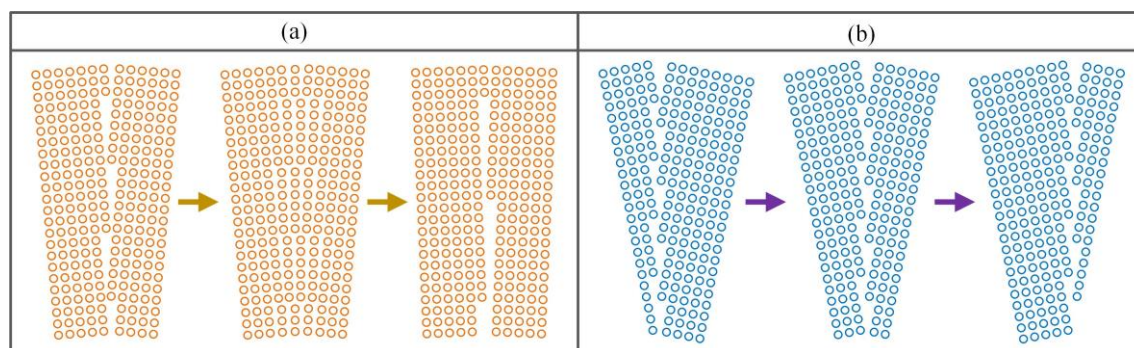


Figure 7. The schematic diagrams of two grain growth mechanisms (a) Multiple grains merge into one grain, (b) One grain annexes with another grain.

From the specimens for 1200°C, it can be seen more clearly that both mechanisms coexist simultaneously, as shown in Figure 8. As the isothermal time extending, some austenite grains become larger through mutual annexation, and traces of grain boundary migration and disappearance can also be seen from Zone 1, 2, and 3. However, it can also be observed from Zone 4, 5, and 6 that recrystallization nucleation occurs within some austenite grains as the isothermal time increases. Also, a small grain is gradually annexed by adjacent grains over isothermal time, as shown in Zone 7, 8, and 9. With the migration of grain boundaries, they eventually reaching an equilibrium state, that is flat grain boundaries and hexagonal grains.

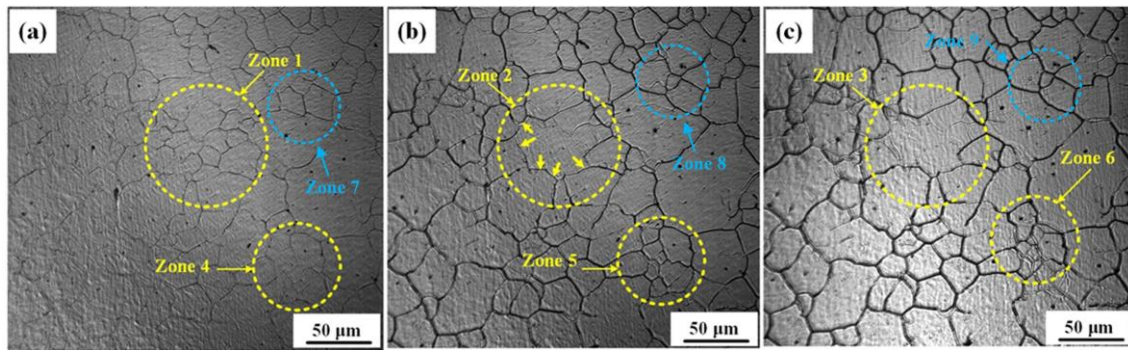


Figure 8. The austenite grain growth for 1200°C with the extension of isothermal time.(a) 200 s, (b) 300 s, (c) 400 s.

According to the above results, large-sized grains are relatively stable. Because large grain boundaries have low energy, small grain boundaries have high energy, and small grains far away from large grain boundaries. It is possible that not every boundary surrounding the large grain has transformed to the high mobility complexion. Further, boundaries around the smaller grains might have transformed, but did not create an abnormally large grain either because not enough time elapsed for the grain to increase in size to differentiate it from the others, or not enough of the boundaries surrounding that grain had transformed and allowed it to grow abnormally large. The increase in average grain size is due to the growth of small grains, at which point the size of large grains remains largely unchanged.

4.3. Consideration of Heating Process for Medium Manganese Steel Continuous Casting Billet

From the perspective of toughness requirements, strict control of the heating process is needed for medium manganese steel. Medium manganese steel has high hardenability and is also martensitic when slowly cooled, so the microstructure of the continuous casting billet is martensitic. Medium manganese steel has special characteristics during the heating process due to the low austenite transformation temperature, because the increase in manganese content can significantly reduce the austenite transformation temperature. During the heating process, carbon and manganese tend to segregate at the grain boundaries, further reducing the austenite transformation temperature.

From the experimental results, it can be seen that 1050°C is composed entirely of small grains, 1100°C to 1150°C is composed of a mixture of large and small grains, while grain sizes above 1200°C are relatively large. Moreover, an increase in grain boundary width indicates damage to the grain boundary when the heating temperature exceeds 1200°C. The heating temperature also affects the of processing difficulty due to the low temperature and high deformation resistance, and it is not possible to choose a temperature that generates very small grains. In summary, the most suitable heating temperature for the medium manganese steel in this paper is from 1100°C to 1150°C.

5. Conclusions

- (1). The average austenite grain sizes of the specimen isothermal at 1050°C, 1100°C, 1150°C, 1200°C, and 1250°C for 600 s are 3.2, 19.8, 37.7, 53.4, and 52.7 μm, respectively. The average austenite grain sizes change from about 3 μm at 1050°C to over 50 μm at 1250.
- (2). When the grain boundary is a small angle grain boundary, one grain boundary will split into several dislocations. With the extension of heating time, the lattice orientation difference further decreases, and the remaining dislocations may merge into new grain boundaries. When the grain boundary is a large angle grain boundary, only grain boundary movement can occur.
- (3). The weight fraction of precipitates below 1000°C is about 0.065% and precipitate reduce to disappear from about 1000°C to 1200°C. A portion of the precipitates nucleate and grow during the heating and holding stage at a holding temperature of 1050~1150°C, and the precipitates dissolve completely during heating when the holding temperature exceeds 1200°C.

- (4). The most suitable heating temperature for the medium manganese steel in this paper is from 1100°C to 1150°C. This takes into account influences such as grain size, grain boundary damage, and deformation resistance.

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Conflicts of Interest: Authors declare no conflicts of interest.

Declaration of Competing Interest: The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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