Materials Research Express

PAPER

Growth behavior and kinetics of austenite grain in low-carbon high-strength steel with copper

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Keywords: low-carbon high-strength steel with copper, austenite grain growth, copper bearing precipitates, solid solution

Abstract

A low-carbon high-strength steel with copper has better antibiological corrosion property, more widely used in large marine projects and ships. In this study, an austenite grain growth model of a low-carbon ship plate steel with 1.6Cu was established by using Sellars equation at 900 °C–1200 °C and different holding times (30–120 min) to reveal its kinetics. The pinning effect of grain boundary precipitates was studied by scanning electron microscopy (SEM) and transmission electron microscopy (TEM), and a quantitative relationship between the solid solution and precipitation behavior of copper bearing precipitates in experimental steel and austenite grain growth was analyzed. The experimental results show that the austenite grains grow slowly, and the grains are fine grains when the heating temperature is below 1000 °C and the transition temperature of rapid grain coarsening is 1000 °C. The relative error of dynamic model can be controlled within 8.5%. During heat treatment, Cu atoms are segregated at the grain boundary to form a copper-rich region, providing a coating effect on the carbide and grain boundary and hindering the growth of grains. With the increase in temperature, the diffusion rate of copper atoms increases, and the precipitates containing copper are rapidly dissolved into austenite. Therefore, the pinning effect is weakened, and the austenite grains grow rapidly.

1. Introduction

Marine engineering steels are used in a complex marine environment, so they have higher requirements for plasticity, toughness, and seawater corrosion resistance. This type of steel is mainly used in the construction of ships and oil platforms [1–3]. Copper has corrosion resistance and good ductility and plasticity during processing [4, 5], so it is added to steel as an alloying element to improve the mechanical properties of steel, which not only improves the antibacterial property and corrosion resistance of steel, but also improves the strength of steel [6]. Therefore, low-carbon high-strength steels with copper are widely used in marine engineering steels. However, as the coarse grain will reduce the mechanical properties of high-strength steel with copper during heat treatment, the growth behavior of austenite grain in view of the effect of austenite grain size on steel properties has been studied [7, 8]. The homogenization temperature and homogenization time during austenitization are the main control variables during heat treatment, affecting the austenite grain growth and dissolution of precipitates [9]. The original austenite grain size and the degree of solid solution of alloy compounds and carbides in the heating process of steel directly affect the grain size during the deformation of steel, grain size after deformation, and precipitation state of alloy compounds and carbides [10, 11]. Mohamadi Azghandi et al [12] studied the grain growth trend of V–Ti microalloying steel at different heating temperatures. The grain size showed an increasing trend with the increase in austenitizing temperature, and the increase in grain size first increased and then decreased.

The processing technology of controlled rolling and controlled cooling of a continuous casting slab of ship plate steel affects the austenite grain size of steel and the solid solution behavior of microalloying elements and

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directly affects the properties of steel [13]. One of the core parameters that determine the mechanical properties of steel foundation is to control the austenite grain size of steel. Illescas et al [14] studied the austenite grain growth behavior of low-carbon high-strength steel and concluded that the value of grain size growth exponent decreases with the increase in heating temperature and holding time. J Fernandez et al [15] found that the addition of alloying elements is an important factor that affects the austenite grain growth. Therefore, it is essential to evaluate the effects of different heating temperatures, holding times, and alloying elements on austenite grain size [16–18]. In this study, the heating temperature, holding time, and solid solution behavior of Cu-containing phase on the austenite grain growth of 1.6Cu steel were studied. A kinetics equation of grain growth of copper bearing steel was obtained.

2. Materials and methods

A 200 kg vacuum smelting experimental steel obtained from the central testing institute was used in this experiment. The composition of experimental steel is shown in table 1. The steel was poured into a round billet of φ130–170 mm, and then it was cut to be rolled. Hot rolling was carried out using a 500 mm rolling machine at 1200 °C, and the surface oxide sheet was removed after being taken out of the furnace. The opening rolling temperature was 1150 °C, and the finishing rolling temperature was 825 °C. The roughing rolling was carried out for 6 passes, and the finishing rolling was carried out for 4 passes. A number of samples with a size of φ8 × 12 mm were taken from the center of the rolled steel plate for heat treatment experiment. The heat treatment process diagram is shown in figure 1. The samples obtained after heat treatment were sanded and

| C     | Si   | Mn   | P    | S    | Als  | Cu   | Mo   | Ti   | Nb   | Cr   | Ni   |
|-------|------|------|------|------|------|------|------|------|------|------|------|
| 0.056 | 0.320| 0.820| 0.011| 0.003| 0.040| 1.620| 0.200| 0.160| 0.032| 0.690| 1.030|

Figure 1. Heating temperature curve of sample.

Figure 2. Microstructure of experimental steels at different temperatures (a) 900 °C, (b) 1000 °C, (c) 1100 °C, and (d) 1200 °C.
polished, and the austenite grain boundary was etched with a saturated picric acid solution at 70 °C. The microstructure and the original austenite grain size were observed using Axio VerT. A1 Zeiss optical microscopy (OM), Evo MA 10 scanning electron microscopy (SEM), and JEM-2100 transmission electron microscopy (TEM). According to the national standard GB/T 6394-2002 method for the determination of average grain size of metal, the grain size of austenite was determined using the linear cut-off method.

3. Results and discussion

3.1. Effect of temperature on the growth behavior of austenite grains

Figure 2 shows the austenitic shape of experimental steel held for 30 min at different temperatures. When the temperature is 900 °C, the average grain size of experimental steel is 16.5 μm, and the austenite grain size is small because of the low heating temperature. When the temperature rises to 1000 °C, the average grain size of the test steel is 20.3 μm, and the distribution is uniform. When the temperature is 1100 °C, the average grain size is 57.5 μm, and the grain shape clearly changes. The grain size clearly grows. When the temperature is 1200 °C, the grain size is 100.1 μm, which increased by 6.25 times compared with that at 900 °C. The grain size of experimental steel was statistically plotted to obtain the variation trend of grain size with heating temperature at different temperatures, as shown in figure 3(a). According to figure 3(a), at the same holding time, the grain size increased with the increase in heating temperature, while at different temperatures, the grain size increased at different rates. When the temperature is 900 °C–1000 °C, the average grain size increased by 23%, and the grain size grows steadily without jumping or abrupt change. This is because the heating temperature is low; the grain boundary mobility is small; and the grain growth rate is low. However, when the temperature reached 1100 °C, abnormal grain growth occurred, and with further increase in temperature, the grain size increased faster. This is because
thermal activation and stress driving become the main reason for grain boundary migration under this condition [3]; the grain boundary mobility increases sharply; and the grain growth rate increases significantly. In the temperature range of 1000 °C–1100 °C, the average grain size increased by 183.3%, and some carbides and copper-bearing phases that hinder grain growth dissolved into austenite. This reduced the pinning and hindering effect on grain boundary, and most grains grow rapidly. In the temperature range of 1100 °C–1200 °C, the grain size increased by 74.1%, and the growth rate of grain size decreased. Therefore, the turning point temperature of 1.6Cu steel grain rapid growth is 1000 °C.

3.2. Effect of holding time on the austenite grain growth behavior
Figure 4 shows the microstructure of austenite grains of the experimental steel at 1200 °C and different holding times. As shown in figure 4, the overall change in grain size in the whole holding time range is small, and the grain presents polygons. With the increase in holding time, the grain size increases, while the number of grains decreases, and the total area of grain boundary decreases. The grain boundary tends to be flat, as shown in figure 4(c). At the junction of three grains, the grain boundary angle is 120°. Figure 5 shows the relationship between holding time and grain size of experimental steel at different temperatures. When the heating temperature is 900 °C, 1000 °C, 1100 °C, and 1200 °C, the grain size increases by 47.9%, 68.0%, 56.3%, and 42.0%, respectively, when the holding time increases from 30 min to 120 min. When the heating temperature is lower than 1000 °C, the slope of the line is small, and the increase in grain size is small. When the heating temperature exceeds 1000 °C, the slope of the straight line increases. With the extension of holding time, the grain size increases significantly. The results show that the growth behavior of austenite grains is more sensitive to the heating temperature than the holding time, and it can be determined that the transition temperature of the abnormal grain growth of experimental steel is 1000 °C. Grain coarsening temperature can be used as a reference for heat treatment process temperature, avoiding the influence of coarse grain size and microstructure defect on the mechanical properties of steel during the processing.

3.3. Characteristics of precipitates containing Cu
Figure 6 shows the morphology and EDS analysis of precipitated phase particles of experimental steel at 900 °C, 1000 °C, 1100 °C, and 1200 °C for 30 min. Figure 6 shows that the second phase is distributed mainly around the grain boundary, and the size and quantity of precipitated phase decreases with the increase in heating temperature. The EDS analysis results show that the precipitated phases contain various types of carbides and copper-rich phases, including Cu, Cr, Mn, Mo, Ti, and other alloy elements. Among them, Cr, Mn, Mo, and Ti form carbides with a high melting point, which are nailed at grain boundaries and hinder the growth of austenite grains. The statistical results of average size and quantity of precipitated phase in the experimental steel are shown in figure 7(a). When the heating temperature is 900 °C, 1000 °C, 1100 °C, and 1200 °C, the average size of precipitated phase is 30.7 nm, 28.1 nm, 21.8 nm, and 17.5 nm, respectively. The quantity was 525.5, 467.2, 267.5, and 187.3 pieces/μm², respectively. The results show that the average size and quantity of precipitates
gradually decreased with the increase in heating temperature, indicating that the large size and high density precipitates have an inhibiting effect on the growth of austenite grains.

Figure 7(b) shows the change curve of mass fraction of Cu in the precipitated phase. When the temperature is 900 °C–1200 °C, the mass fraction of Cu element in the precipitated phase is 11.49%, 17.02%, 2.03%, and 1.23%. Figure 7(b) shows that the content of Cu element in the second phase is the highest at 1000 °C. With the increase in heating temperature, Cu reaches the melting point and dissolves into the austenite, while the mass fraction of Cu in precipitated phase decreases.

Figure 6. Austenitizing morphology and corresponding EDS energy spectrum of test steel held at 900 °C (a), 1000 °C (c), 1100 °C (e), and 1200 °C (g) for 30 min.
TEM was used to further observe the morphology of precipitated phase and EDS analysis of 1.6Cu steel at $900^\circ$C–$1200^\circ$C for 30 min. As shown in figure 8, the heating temperature is $900^\circ$C. Figure 8(a) shows that the quantity and size of precipitated phase particles. Most particles are distributed near the grain boundary. A few particles are scattered in the intracrystalline region. The precipitation particles can hinder the expansion of grain boundary migration, so the austenitic grain size is very small. Figure 8(b) shows that the main precipitates in the EDS spectrum are Mn, Cr carbides, and copper-bearing phases. When the heating temperature rises to $1000^\circ$C, the number and size of precipitated phase particles in figure 8(c) decreased compared with those shown in figure 8(a). When the heating temperature reached $1100^\circ$C as shown in figure (c), compared with Figures (a) and (b), the precipitation particles had a significantly lower density. The precipitation particles are mainly distributed in the grain boundary, weakening the dissolution of precipitated phase on the pinning effect of grain boundary and leading to a rapid growth in the austenitic grain size. The EDS spectrum in figure 8(b) shows that the precipitate phase is rich in copper; this shows the existence of copper precipitated phase. The TEM diffraction image in figure 8(c) shows that the precipitate at point B has the FCC structure of $\varepsilon$-Cu phase, and the copper atoms are continuously enriched and evolved to form a copper-rich segregation area, which is mainly distributed near the grain boundaries. Most of the precipitated particles in figure 8(d) are dissolved at $1200^\circ$C.
The dispersed precipitated particles gradually converge towards the grain boundary, and almost no obvious precipitated phase can be observed in the grain.

3.4. Austenite grain growth model of 1.6Cu steel

Based on the austenite grain evolution, a model was established to predict the grain size and control the mechanical properties of experimental steel. Banerjee [19] Adrian [20] and Manohar et al [21] derived a grain growth kinetics model of microalloying steel considering the precipitated phase. These models dynamically evolved from Beck equation. Under isothermal conditions, Beck [22, 23] established the relationship between austenite grain size and holding time based on the research results, which can be expressed as follows:

\[ D = D_0 + Kt^n \]  

where D is the average grain size (μm) of austenite grains grown after heating for a certain time; \( D_0 \) is the initial average grain size of austenite (μm). K is the austenite grain growth rate; n is the austenite grain growth exponent. Both are functions of temperature T. t is the holding time (s). Take the natural logarithm of both sides of equation (1).

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

The experimental data were added to equation (2) for linear regression processing. Under different heating conditions, the linear relationship between lnD and ln t is shown in figure 9.

As shown in figure 9, when the heating temperature is 1173–1473 K, an approximate linear relationship exists between lnD and ln t, indicating that the austenite grain growth trend of 1.6Cu steel conforms to Beck equation. The slope of each line in the figure represents the austenite grain growth exponent n at different heating temperatures, and its intercept is lnK. The calculation results are shown in table 2. As shown in table 2, when the heating temperature is 1173–1273 K, the austenite grain growth exponent n and growth rate K change slightly, indicating that the austenite grain size does not increase significantly with the extension of holding time.

| Temperature (K) | n   | K    |
|----------------|-----|------|
| 1173           | 0.27| 2.16 |
| 1273           | 0.27| 2.93 |
| 1373           | 0.28| 7.49 |
| 1473           | 0.25| 15.41|

Figure 9. Relationship between lnD and ln t under different heating conditions.

Table 2. Growth index and growth rate of copper-containing ship plate steel at different heating temperatures.
in this heating range, and it has a good anticoarsening ability. The coarsening trend of grain during thermal
deformation negatively affects the final mechanical properties [24, 25]. When the heating temperature rises to
1373 K, the growth exponent n continues to increase to 0.28. Meanwhile, the average grain growth rate K rapidly
increases to 7.49, indicating that the austenite grains grew rapidly in the heating temperature range of
1273–1373 K. When the heating temperature was increased to 1473 K, the growth exponent n significantly
decreased to 0.25. However, the average grain growth rate K rapidly increased to 15.41, indicating that the
austenite grain size reached a plateau when the heating temperature was 1473 K.

The austenite grain growth model of 1.6Cu steel during heating can be expressed by Sellars equation [26].

\[ D^k - D_0^k = At \exp \left( -\frac{Q}{RT} \right) \] (3)

where D is the average grain size (\( \mu m \)) of austenite grains after their growth; D_0 is the average grain size of
original austenite (\( \mu m \)). A and k are material constants. Q is the activation energy of austenite grain growth
(J mol\(^{-1}\)). R is the gas constant, and its value is 8.314 J mol\(^{-1}\). T is the heating temperature (K); t is the holding
time (s). Because the original austenite grain is much smaller than the grown austenite grain, the effect of D_0 can
be ignored. Therefore, equation (3) can be simplified as follows:

\[ D^k = At \exp \left( -\frac{Q}{RT} \right) \] (4)

Taking the logarithm of both sides of equation (4), we can obtain:

\[ \ln D = \frac{\ln A}{k} + \frac{\ln t}{k} - \frac{Q}{kRT} \] (5)

To improve the accuracy of this model, the statistical data in figure 3 and the regression analysis method were
used to build a segmented model for different temperature ranges of austenite grain growth in the test steel.
When the holding temperature is lower than 1273 K, the austenite grain growth rate is slow, and the austenite
grain size change range is small. When the temperature is higher than 1273 K, the austenite grain size varies
significantly with the heating temperature and holding time. Therefore, 1173 K \( \leq T \leq 1273 \) K and 1273 K \( < T \leq 1473 \) K are discussed in this study. The modeling process and results of \( T \leq 1473 \) K:

(1) In the range of 1173 K \( \leq T \leq 1273 \) K, substituting the experimental data into equation (5), k = 6.57 and
A = 2.3 \times 10^9 and Q = 165383.9. Substitute the values of k, A and Q into equation (3) to obtain the
model:

\[ D^{3.6} - D_0^{3.6} = 2.3 \times 10^9 t \exp \left( -\frac{165383.9}{RT} \right) \] (6)

(2) In the range of 1273 K \( < T \leq 1473 \) K, put the experimental data into equation (5), k = 4.70 and
A = 6.3 \times 10^{18} and Q = 371431.8. The obtained values of k, A and Q are substituted into equation (3) to
obtain the model:

\[ D^k = At \exp \left( -\frac{Q}{RT} \right) \] (4)
interfacial energy and the total grain boundary area. Austenite grain size and the high grain boundary energy because grain growth always tends to reduce the driving force of austenite grain growth in experimental steels originates from the inhomogeneity of the small grains. If the temperature is further increased or the holding time is extended, the grains will continue to grow. Grain growth is carried out by the continuous outward migration of grain boundaries. The nature of precipitation particles

\[ D^{4.7} - D_0^{4.7} = 6.3 \times 10^{19} \exp \left( -\frac{371431.8}{RT} \right) \]  

(7)

3.5. Model validation
The relationship between linear regression \( \ln D \) and \( T^{-1} \) is shown in figure 10(a). In the temperature range from 1073 K to 1473 K, figure 10(a) shows that \( \ln D \) and 1000/T are approximately linear during continuous heating and isothermal austenitizing, proving that the model is valid. The experimental data was fitted with the curve predicted by model calculation, as shown in figure 10(b). As shown in figure 10(b), the relative error between the calculated value and the experimental value is between 8.5%, and the experimental data is near the model curve. It shows that the model can accurately predict the grain growth of test steel in the temperature range of 1173 K \( \leq T \leq 1473 \) K, and the model can better predict the experimental results. A constitutive equation constructed in this study can effectively define the relationship between austenitic grain size and austenitizing temperature with time, and it can be used to predict the low carbon high strength steel in copper under different heating temperatures and holding times after the heat treatment of austenitic grain size as other low carbon high-strength steel austenitic grain growth kinetics of copper research has a reference value.

4. Results and discussion
The driving force of austenite grain growth in experimental steels originates from the inhomogeneity of austenite grain size and the high grain boundary energy because grain growth always tends to reduce the interfacial energy and the total grain boundary area [27–29]. Therefore, the grains grow up and gradually absorb the small grains. If the temperature is further increased or the holding time is extended, the grains will continue to grow. Grain growth is carried out by the continuous outward migration of grain boundaries. The nature of grain boundary migration is that atoms pass through the diffusion interface. In addition, the diffusion coefficient at the interface has an exponential relationship with temperature [30]. Therefore, the grain growth process has the characteristics of general thermal activation process. Under the effect of temperature [31, 32], when the experimental steel was heated to the critical temperature, the second phase formed by low-alloy carbide and copper precipitating to a large extent prevented the growth of austenite grain size and refined the austenite grains [33]. This critical temperature is known as grain coarsening temperature. Figures 3 and 5 show that heating temperature plays a dominant role in the grain growth, and the austenite grain size grows the fastest at 1000 °C, indicating that the trend of austenite grain growth is the strongest at this temperature. Thus, the grain coarsening temperature of experimental steel is 1000 °C.

Compared with the ordinary steel, the grain growth behavior of austenite in high-strength steel is different, and the composition of steel determines the dissolution temperature of precipitated phase, thus determining the grain coarsening [34]. A large number of studies indicate that the second-phase particles significantly affect the growth behavior of austenite grains [35–37]. The principle that the second-phase particles of steel can prevent grain growth is the combined action of driving force of grain growth and the resistance of the second relative grain [38]. The effect of precipitated phase particles on grain boundary migration is similar to the effect of friction resistance, reducing the driving force of grain growth [39]. The relationship between the pinning force of precipitated particles and grain boundary migration can be expressed as follows [40, 41]:

\[ d_r = 3kf / 2r \]  

(8)

In equation (8), \( d_r \) is the grain growth resistance; \( k \) is the interface energy; \( r \) is the radius of second-phase precipitation particles (μm); \( f \) is the volume of precipitated particles. Equation (1) shows that the larger the proportion of the volume of the second-phase precipitated particles, the greater the resistance of grain growth, the stronger the nailing effect on grain boundary, and the smaller the grain size. Figure 7(a) shows that at the early stage of heating, due to a low temperature, the activation and migration of grain boundary itself is small, and the precipitated grain boundary particles have a large size and large number, clearly exhibiting nailing and hindrances on the grain boundary and making the grains smaller. With the increase in temperature, the fine dispersed second phase dissolves into austenite continuously, but a small amount of high melting point carbides contain Cr and Mn in the precipitated phase, reducing the nailing on grain boundaries and leading to rapid grain growth. The decrease in the number and size of precipitated particles is the main reason for the austenite grain growth [42]. As the temperature continues to rise, the driving force of grain growth decreases; the nailing force almost disappears; the grain boundary is in the state of unnailing; and the grain growth rate reaches the plateau stage and maintains a stable growth.

In this experiment, the steel contains high Cu microalloying elements, and the Cu-rich phase is enriched at grain boundaries during heating, limiting the growth of austenite grains. Among them, the solid solubility of copper in austenite occurs the temperature range of 900 °C–1200 °C [43]:

\[ \text{Mater. Res. Express} 8 \text{ (2021) 096504} \]
\[ \log(Cu) = 2.652 - 2462/T \] (9)

In equation (9), T is the heating temperature (K). According to the formula of solid solubility of Cu in austenite, with the increase in heating temperature, the solid solubility of Cu in austenite increases significantly. At the early stage of heating, diffusion of Cu atom is slow; a Cu-rich phase and carbide exist in grain boundary and ingrain; a part of Cu is dissolved into the matrix; and most of the Cu-containing phase is coated in grain boundary, acting together with carbide, effectively preventing the growth of austenite grains and leading to grain refinement. At this time, the growth rate of austenite grains is not obvious. As shown in figure 7(b), when the heating temperature reaches 1000 °C, the mass fraction of Cu in the precipitated phase reaches the highest level. As the heating temperature increases, the mass fraction of Cu gradually decreases. As the temperature increases, the diffusion rate of Cu increases significantly [44–46], and the precipitated copper phase dissolves in large quantities and enters austenite. The high-temperature diffusion of Cu in steel mainly occurs along the grain boundaries [47, 48]. The number of Cu-bearing phase enrichment areas at the grain boundaries decreases, and the coating effect on grain boundaries decreases. Cu is a noncarbide forming element, which is conducive to carbide diffusion, reduces the obstruction of grain growth, and promotes the growth of austenite grains. In conclusion, copper precipitates affect the austenite grain growth of low-carbon high-strength steel containing copper.

5. Conclusion

The grain growth behavior of low-carbon low-alloy high-strength steel with copper during austenitization in 900 °C–1200 °C temperature range for 30–120 min was studied. The austenitization grain growth model of low-carbon steel with copper was established, and the dynamic mechanism of copper grain growth in the process was explored. The following conclusions can be drawn from this study:

1. With the increase in heating temperature and the extension of holding time, the austenite grain size gradually increases. When the heating temperature is lower than 1000 °C, the grain growth rate is slower. When the heating temperature is higher than 1000 °C, the grain growth rate increases rapidly, and the effect of heating temperature on austenite grain growth is more significant.

2. A kinetic model of austenite grain growth of 1.6Cu high-strength steel under different heating conditions was established:
   Within the range 1173 K ≤ T ≤ 1273 K:
   \[ D^{1.6} - D_0^{1.6} = 2.3 \times 10^{9} t \exp \left( -\frac{165383.9}{RT} \right) \]
   Within the range 1273 K < T ≤ 1473 K:
   \[ D^{1.7} - D_0^{1.7} = 6.3 \times 10^{9} t \exp \left( -\frac{371431.8}{RT} \right) \]

Moreover, the model was verified by the experimental data, and the verification results showed that the relative error between the experimental value and the model simulation value is less than 8.5%. Therefore, the constitutive equation constructed can effectively define the relationship between austenite grain size, austenitizing temperature, and time with high accuracy.

3. During the heating, when the temperature is lower than 1000 °C, the number and size of copper-rich precipitates and the second phase of carbide are larger, and the effect of hindering grain growth is more obvious. When the temperature is higher than 1000 °C, the Cu-rich phase and carbide size decrease, and the austenite grain grows rapidly.

Acknowledgments

This work are supported by the National Natural Science Foundation of China(U1860112); Joint fund of University of Science and Technology Liaoning and Ansteel State Key Laboratory of offshore engineering and steel(HGSKLUSTLN2020-01); Outstanding youth fund of University of Science and Technology Liaoning (2019RC10); Youth Fund Project of Liaoning Provincial Education Department(2020LNQN19).
Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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