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Effect of Heating Temperature on the Grain Size and Titanium Solid-Solution of Titanium Microalloyed Steels

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Abstract

Through studying on the heating process of titanium microalloyed steels, the influence of heating temperature on the austenite grain size and the solid dissolution, precipitation law of Ti microalloying element were analyzed, and the results showed that, the austenite grain size increased with the increase of heating temperature, When the heating temperature reached 1050˚C and 1250˚C, the austenite grains appeared the obvious coarsening process twice. TiC particles dissolved gradually as the heating temperature increased. When the heating temperature rose to 1100˚C, TiC particles disappeared basically, When the heating temperature rose to 1250˚C, TiN particles began to be dissolved and grow up.

Keywords

Titanium Microalloying, Heating Temperature, Austenite Grains, Solid-Solution, Precipitation

1. Introduction

In the rolling process of titanium microalloyed steels, the heating system has an important effect on the original austenite grains and solid solution of Titanium microalloying. Due to the inheritance of austenite grains, the heating process will affect the size of austenite grains and distribution of titanium particles during rolling process, thus affecting the microstructure and properties of as-rolled products. And so the study on the heating process of titanium microalloyed steels and the establishing of a reasonable heating system has an important influence on the controlled rolling and cooling processes.

The main factors affecting the size of austenite grains during the heating process, include the heating temperature, the holding time and the contents of alloying elements. Through further analysis on the Zener model [1], Gladman [2] And Hillert [3] studied the effect of precipitation particles on the size of austenite grains, and proposed a method for predicting the size of austenite grains.

Through analysis on the size of austenite grains during the heating process of continuous casting slabs, some scholars [4] derived the empiric formula, the formula of austenite grain growing related to chemical composition was derived, but the effect of Ti on the activation energy of grain growth was not analyzed.

The second-phase particle precipitation of Ti element is usually divided into two stages: the precipitation of TiN particles occurs at high temperatures and the strain-induced precipitation of TiC particles occurs at low temperatures. Similarly, during the heating process, the influence of Ti-bearing precipitated particles on the grain coarsening also has two stages. In this paper, the growth of austenitic grain size for the microalloyed steels with Ti content of 0.05% were studied in the paper.

2. Test Materials and Methods

The test steel is titanium microalloyed Q345D, its chemical composition is detected by photoelectric direct reading spectrometer (M12). Its chemical composition was shown in **Table 1**.

The test steel was taken from 1500 mm continuous rolling production line of laigang group steel mill, A sample of 16 mm × 16mm × 10 mm in size was processed. Then the sample was heated to a set temperature (850˚C, 900˚C, 950˚C, 1000˚C, 1050˚C, 1100˚C, 1150˚C, 1180˚C, 1200˚C, 1250˚C), and immediately quenched with ice brine after holding for 30 minutes. After grinding and polishing, the quenched specimen were treated with a solution of saturated picric acid and a small amount of detergent, with the constant temperature of 60˚C, to display the original austenite microstructure, and then the austenite grain size was measured by truncation in the Image-pool software, and the number of grains measured was more than 300 to ensure the accuracy of the data.

A thermodynamic formula was used to calculate the solid solution law of Ti during austenization process of the test steel under equilibrium conditions. The size, morphology and distribution of the particles under different heating temperatures were observed by transmission electron microscopy. And the components of the particles were analyzed by EDS.

3. Effect of Heating Temperature on Austenite Grain Size

Figure 1 shows the original austenite microstructure of test steel after holding for 30 minutes at different heating temperatures.

As can be seen from **Figure 1**, when the heating temperature reaches 850˚C and 950˚C, the austenite grains are small and uniform, and as the heating

Table 1. Chemical compositions of test steel (Wt %).

C	Si	Mn	P	S	Als	Ti	N
0.15	0.28	0.65	0.013	0.004	0.023	0.048	0.0028

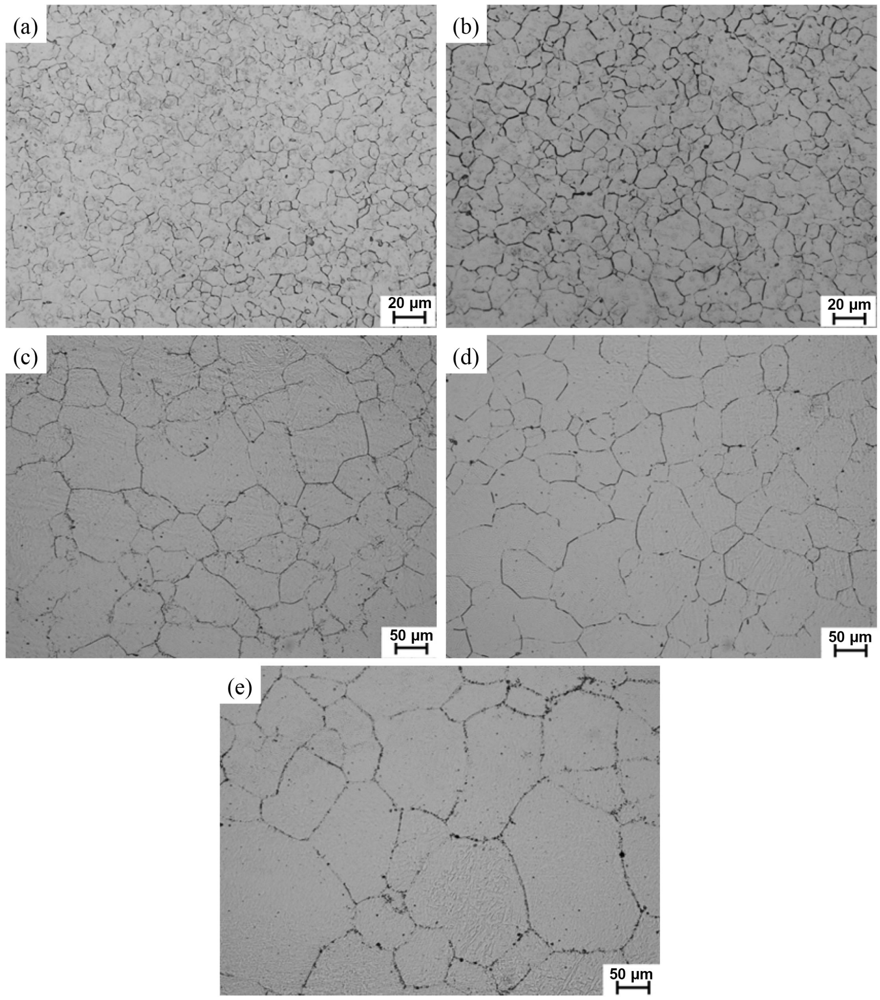

Figure 1. The original austenite grain microstructure under different heating temperatures. (a) 850˚C; (b) 950˚C; (c) 1050˚C; (d) 1150˚C; (e) 1250˚C.

temperature increases, the austenite grains grow slowly. When the heating temperature is lower, the fine TiC particles in the test steel are dispersed in austenitic grains, preventing austenitic grains from growing. When the heating temperature rises to 1050˚C, the austenite grains grow significantly, and some large-sized grains appear, the non-uniform distribution of grain size is observed, and there is a tendency for large grains begin to swallow small grains. At the moment, the TiC precipitated particles in austenite grains are basically dissolved, and thus its pinning force on austenite grain growth is greatly reduced, and a few grains grew abnormally. At heating temperature of 1150˚C, the growth of austenite grains are not more significant due to the pinning effect of TiN particles at the boundary of austenite grains. When the heating temperature rises to 1250˚C, the TiN particles at the austenitic boundary are dissolved, and its pinning force is greatly reduced, so the austenitic grains grow abnormally.

Table 2 shows the average size of austenitic grains in test steel after holding for 30 minutes at different heating temperatures.

Figure 2 shows the tendency of austenitic grain size changing with temperature. It can be seen from **Figure 2**, with the increase of temperature, there are two coarsening temperatures for the test steel, which are 1050˚C and 1250˚C, respectively,. When the heating temperature is lower than 1000˚C, the austenite grains grow slowly. When the temperature rises to 1050˚C, the austenite grains grow up suddenly, the size increased from 24.7 μm to 63.7 μm. When the temperature rises from 1050˚C to 1200˚C, the austenite grain growth tends to slow down again. When the temperature rises to 1250˚C, the austenite grains grow rapidly, the size increased from 80.4 μm to 107.9 μm.

Figure 3 shows the distribution of austenite grain size after holding for 30 minutes at different heating temperatures. It can be seen from **Figure 3**, the size distribution of austenite grains is close to the normal distribution. The normal distribution can well describe the grain size during grain growth [5]. When the heating temperatures are 850˚C and 1050˚C, the austenitic grain size distribution is in line with the normal distribution. When the heating temperature is 1250˚C, the austenitic grain size distribution is relatively dispersed, and some grains grow up abnormally. When the heating temperature is 850˚C, the austenite grains are basically less than 20 μm, and most grains are between 5 μm and 15 μm in size; When the heating temperature rises to 1050˚C, most austenite grains are between 40 μm and 80 μm in size, but due to the dissolution of TiC particles, The large-sized grains larger than 100 microns, which account for 10% of the total grain size, appears. When the heating temperature rises to 1250˚C, the austenite grains are coarsening, and more than 50%. Of the grainssize is larger than 100 μm.

4. The Effect of Heating Temperature on the Solid Dissolution and Precipitation of Ti Element

During the controlled rolling and cooling processes of titanium microalloyed steel, Ti precipitated particles mainly include TiN, Ti4C2S2, and TiC [6]. Because the sulfur contentl is relatively lower, the typical particles are mainly TiN and TiC particles, as shown in **Figure 4**. **Figure 4(a)** shows a dispersed fine precipitated particle with size less than 10 nm. Analysis on the selected region through the EDS spectrum, these fine precipitated particles are TiC; **Figure 4(b)** is a precipitated particle with size between 100 and 200 nm. EDS spectroscopic

Table 2. Austenite grain sizes at different heating temperatures.

Heating temperature/˚C	850	900	950	1000	1050	1100	1150	1180	1200	1250
Average grain size/μm	10.03	11.42	12.71	24.68	63.69	68.11	72.63	75.88	80.4	107.9

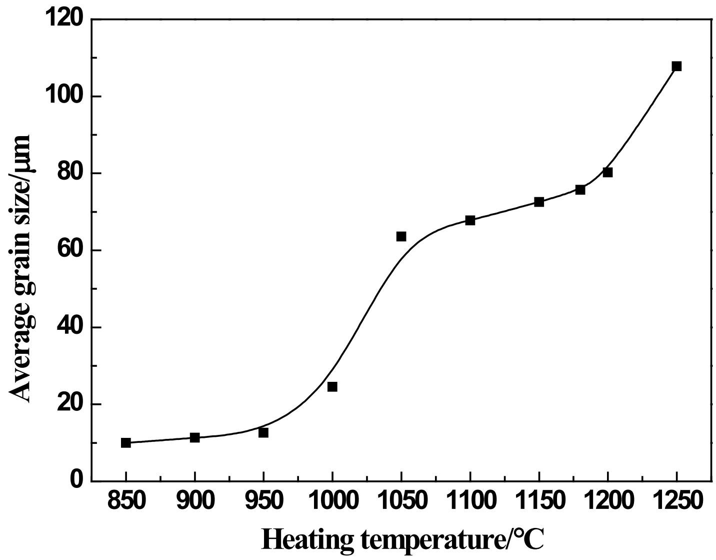

Figure 2. Effect of heating temperature on the size of austenite grains.

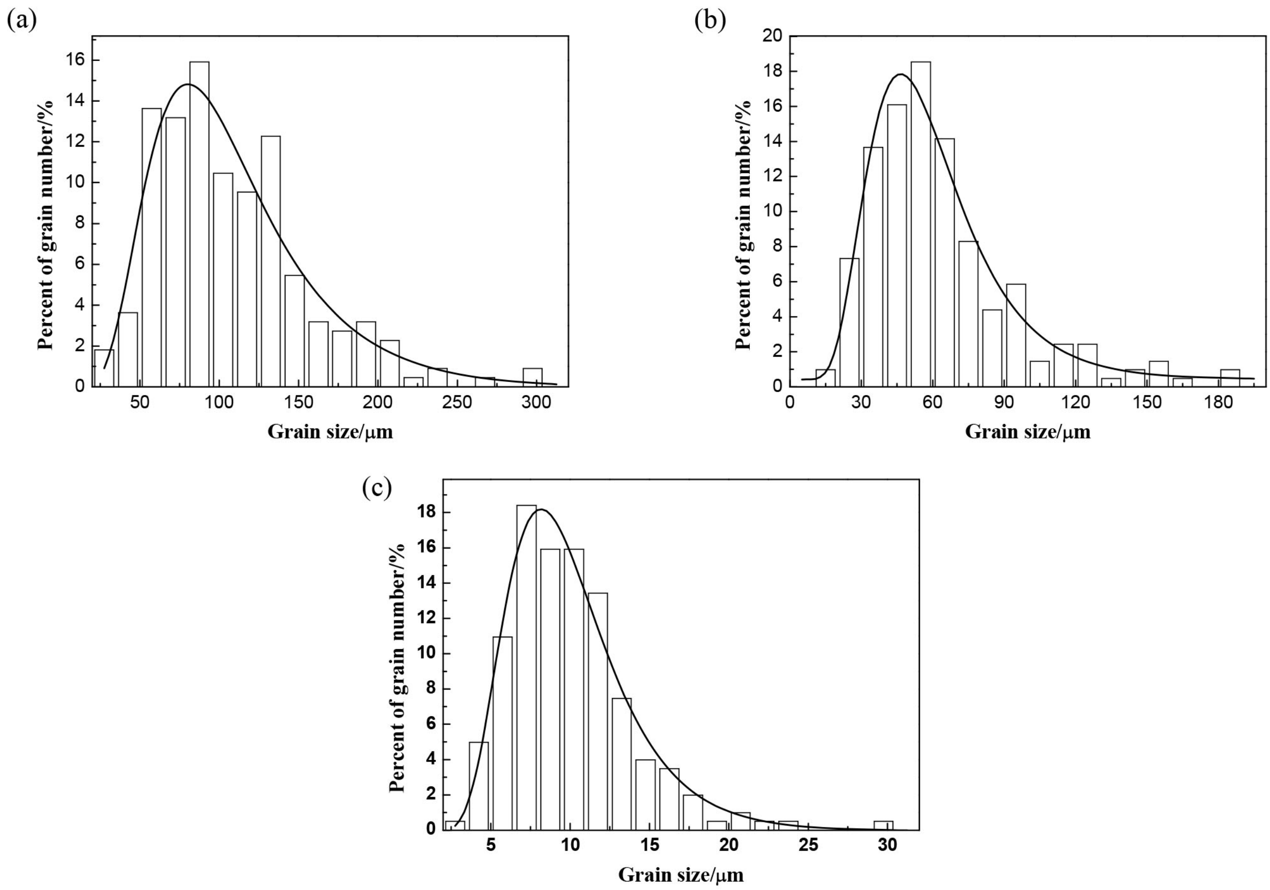

Figure 3. Austenite grain size distribution at different temperatures. (a) 850˚C; (b) 1050˚C; (c) 1250˚C.

analysis shows that the precipitated particles are carbonitrides of Ti, and further analysis by EDR calibration shows that the precipitated particles are TiN particles with face-centered cubic.

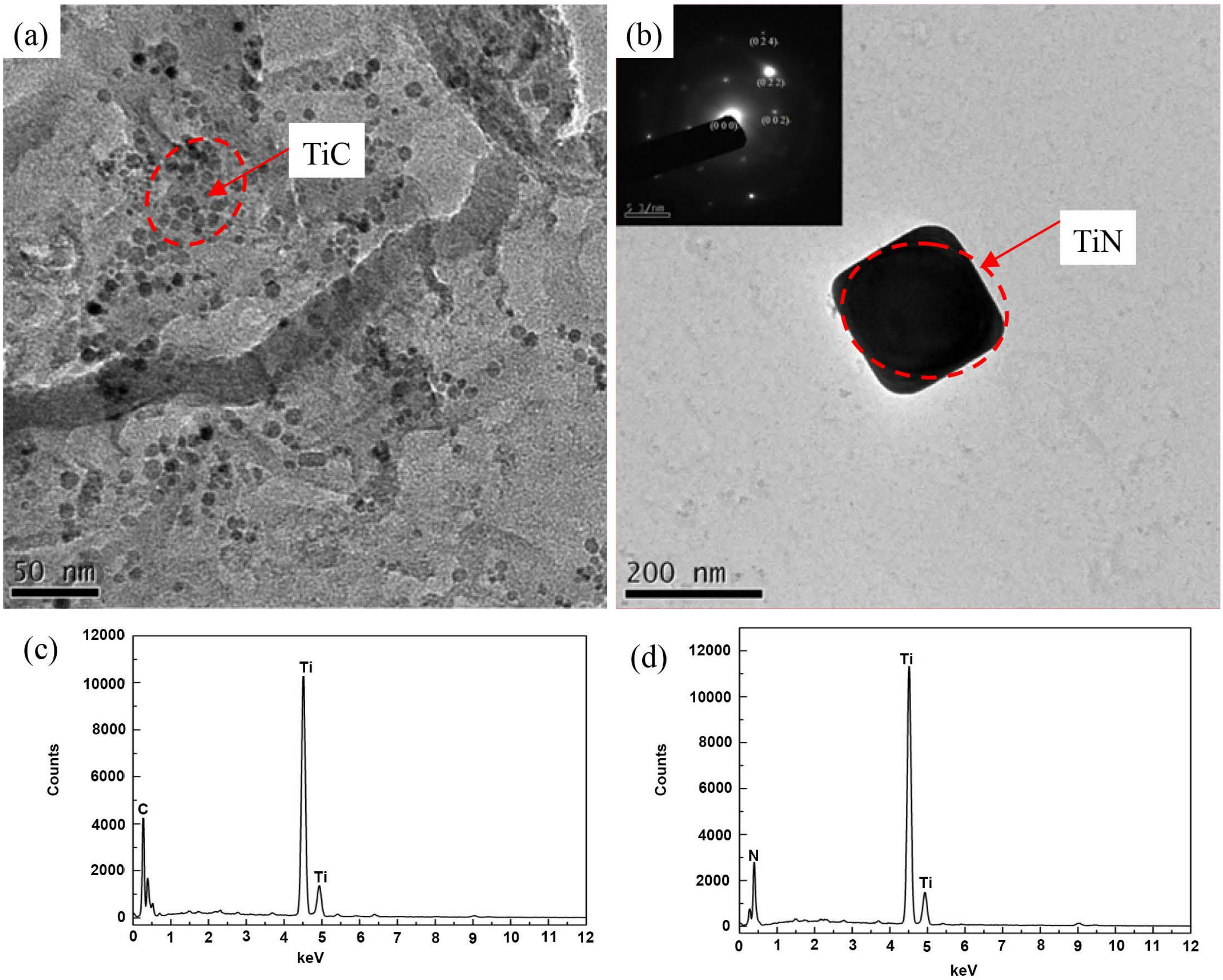

Figure 4. Two typical particles precipitated by Ti. (a) Smaller particles; (b) Bigger particles; (c) and (d) are EDS analysis of corresponding regions of (a) and (b).

Figure 5 is the distribution of the size and morphology of Ti-precipitated particles prepared by extraction complex at different heating temperatures. It can be seen from **Figure 5** when the heating temperature reaches 850˚C, there are a large number of TiC precipitate with size less than 10 nm. which have a strong effect on grain coarsenization. When the heating temperature rises to 1000˚C, the spherical fine TiC particles are significantly reduced and the size increases to 10 - 20 nm; When the heating temperature rises to 1100˚C, the small spherical TiC particles basically disappear and the austenite grains are coarsened, but there are still TiN particles with size between 50 nm and 100 nm, and these particles still have some hindrance to the growth of austenite grains.; When the heating temperature rises to 1200˚C, the precipitated TiN with size mainly between 50 nm and 200 nm; When the heating temperature rises to 1250˚C, the number of TiN particles is significantly reduced, and the precipitated particles mostly grows to 200 - 500 nm, whose effect of preventing austenite grain growth is greatly weakened, and the austenite grains are significantly coarsened.

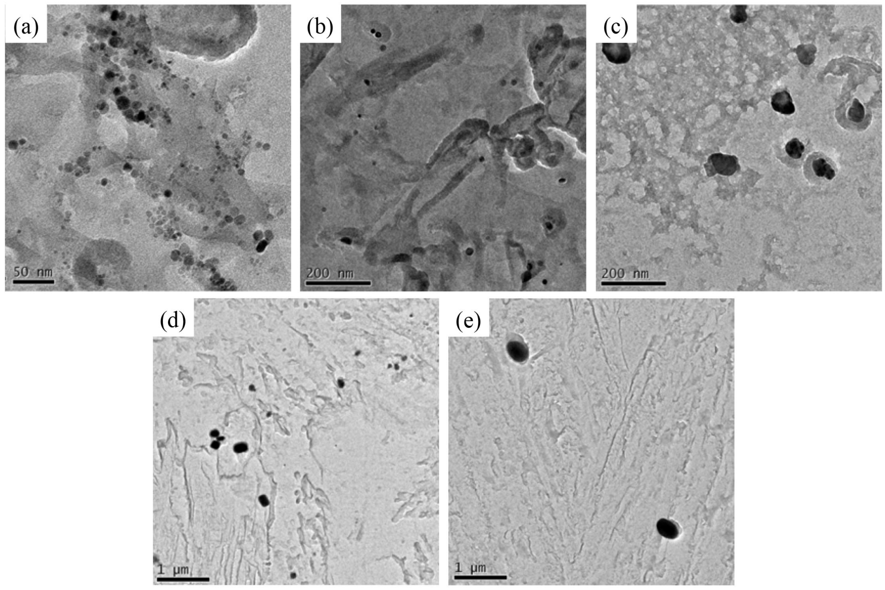

Figure 5. The morphology of precipitated particles at different heating temperatures. (a) 850˚C; (b) 1000˚C; (c) 1100˚C; (d) 1200˚C; (e) 1250˚C.

5. Results Analysis and Discussion

The coarsening process of austenite grains during heating process is related to the alloying elements in steel. The precipitated particles of alloy elements have a strong hindrance effect on the growth of austenite grains. When the driving force of grain growth reaches a thermodynamic equilibrium with the pinning force of precipitated particles, Austenitic grains grown normally. Therefore, the austenite grain size prediction model can be expressed by the following formula:

$$R = A \cdot r / f \tag{1}$$

In the formula: R is the austenite grain size, A is the constant, R is the average size of the precipitated particles, and f is the the volume fraction of precipitated particles. At present, many scholars have analyzed the value of the constant A, The value of A in the Zener model [1] is 0.75; The value of A in Gladman model [2] is between 0.05 and 0.26; The value of A in Hillert model [3] is 0.44, the austenite grains grow normally, the value of A is 0.44 to 0.67, and the austenite grains grow abnormally.

Austenite grain size is related to the size and volume fraction of the precipitated particles. Both TiC and TiN particles precipitated in the test steel after being rolled.. As the heating temperature increases, the dispersed fine TiC particles are gradually dissolved. When the heating temperature rises to 1100˚C, the TiC particles are basically dissolved, the pinning effect of TiC on austenite grains disappears and the austenite grains change coarsened. With the further increase of heating temperature, the growth of austenite grains is not clear due to Due to the nail effect of TiN precipitates particles. When the temperature rises to 1250˚C, the volume fraction of TiN precipitated particles is significantly reduced, their size is increased, and the nail effect on austenite grains is significantly reduced, the austenite grains are coarsened.

At present, many scholars have studied the solid solution behavior of Ti element in austenite. Among them, the solid solubility product of TiN in austenite can be expressed by the formula proposed by Kunze *et al.* [7], The solid solubility product of TiC in austenite can be expressed by the formula proposed by Irvine *et al.* [8], they are as follows:

$$\lg\left([\%Ti][\%N]\right)_{\gamma} = 5.19 - 15490/T \tag{2}$$

$$\lg\left([\%Ti][\%C]\right)_{\gamma} = 2.75 - 7000 \tag{3}$$

In the formula: $[Ti]$ $[N]$ And $[C]$ is the amount of element dissolved in austenite. T is the solid solution temperature (K). Based on the formula, the complete solution temperature of TiN and TiC is respectively 1450.7˚C and 1132.3˚C. which are all higher than the coarsening temperature of austenitic grains. When the precipitated particles are dissolved and grow to a certain extent, the pinning force will be reduced to a certain critical value, so that the austenite grains will grow, which is consistent with the study results from Cuddy *et al.* [9].

The austenite grain size in the heating process can also be expressed by the Arrhenius-type formula [10]:

$$d = A\exp\left(-\frac{Q}{RT}\right)t^{n} \tag{4}$$

In the formula: D is the average grain size, A, N are the constant, Q is the grain growth activation energy (J/mol), and R is the gas constant (8.314 $J{\cdot}mol^{-1}{\cdot}K^{-1}$), T is the holding time(s). Considering that the heating times of the test steel in this paper are the same, the austenite grain size in this paper is only related to the heating temperature and alloying elements. Therefore, the empirical formula can be simplified as follows:

$$d = B\exp\left(-\frac{Q}{RT}\right) \tag{5}$$

For general carbon steels, the grain growth activation energy Q is a constant. However, for microalloyed steels, Q is a value related to the content of alloy elements. The influence of alloying elements on Q is mainly by the pinning precipitated particles which is based on the migration of austenite grain boundary. Therefore, the main factor affecting Q value is basically the precipitation particles in steel. In this paper, there are two kinds of precipitated particles in titanium microalloyed steel. The solution temperatures of the two precipitating particles are quite different, and there is a significant two-stage precipitating process. Therefore, the influence of precipitated particles on the growth of austenitic grains also has obvious two-stage effect, and this is consistent with the results in **Figure 2**. And Formula 5 was used to fit the normal growth process of austenite grains in two stages, Further analysis of the influence of precipitated particles on Q value, as shown in **Figure 6**. It can be seen from the figure that, when the heating temperature is lower, the grain growth activation energy of the

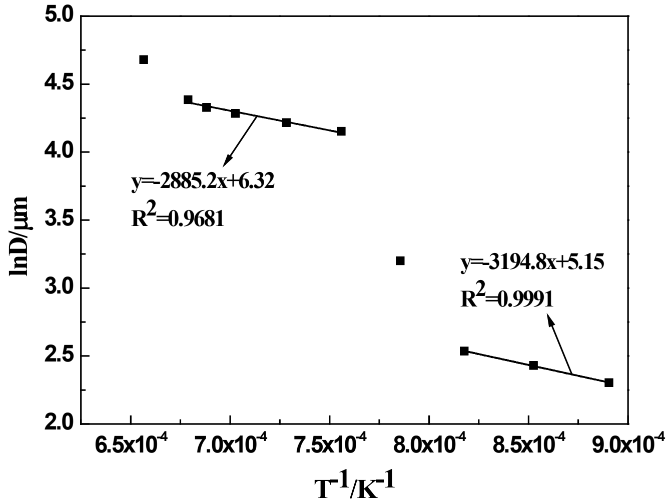

Figure 6. Relationship between reheating temperature and austenite grain size.

test steel is higher, which is 26,561 J/mol; When the heating temperature is higher, TiC particles have been dissolved and the activation energy value Q is reduced to 23,987 J/mol. Therefore, it can be determined that it is not the alloy itself that can affect growth activation., but the precipitated particles in the steel. Among them, TiC particles contribute 2574 J/mol to the activation energy.

During the heating process of titanium microalloying steel, the choice of heating temperature is very important. If the heating temperature is too high, the original austenite grains will be coarsening, and TiN precipitated particles also can fully grow up. During the rolling process, The effect of restraining austenite grain growth is greatly reduced, resulting in the coarsening of grains. If the heating temperature is too low, the precipitated TiC particles can not be fully dissolved. It greatly weakens the pull and nail action of solute atoms in the subsequent rolling process and the inhibition of austenite grain crystallization.. From the above experimental analysis results, it can be seen that, when the heating temperature is between 1180˚C and 1200˚C, the TiC particles are fully dissolved and the austenite grains are not obviously coarsened.

6. Conclusions

Through the study on the heating process of titanium microalloyed Q345D steel, the effect of heating temperature on the size of austenite grains in the test steel and the solid dissolution and precipitation law of Ti microalloying element were analyzed, and the following conclusions can be drawn:

1) The size of austenite grains in the test steel increases with the increase of the heating temperature. When the heating temperature is 1050˚C and 1250˚C, the austenitic grains appear to be significantly coarsened, and the austenite grains grow up. The growth process of austenite grains has obvious two-stage characteristics. The heating temperature of the test steel should be controlled at 1180˚C - 1200˚C.

2) There are two types of Ti precipitated particles in the test steel. With the increase of heating temperature, the TiC particles are gradually dissolved. When the heating temperature rises to 1100˚C, the TiC particles basically disappear; When the heating temperature rises to 1250˚C, TiN particles begin to be dissolved and grow up. The two-stage solid solution of the precipitated particles corresponds to the two-stage growth process of austenite grains.

3) Fitting the grain size law of austenite in two stages respectively, it was obtained that, the activation energies of grain growth in the two stages were 265.6 KJ/mol (at low temperature) and 239.8 KJ/mol (at high temperature), respectively, and the contribution of TiC particles to the activation energies was 25.8 KJ/mol.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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