Effect of final cooling temperature on the microstructure and mechanical properties of high-strength anti-seismic rebar

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Abstract

Rebar is an important material in the major structural engineering, and its fine structure has a very important effect on the performance of the rebar. In this work, the Gleeble-3800 thermal simulator was used to simulate and control the final cooling temperature process to explore the effect of the precipitation behavior of the microalloying elements on the microstructure and mechanical properties of the rebar. The electron backscatter diffraction (EBSD), high-resolution transmission electron microscope (TEM), and universal tensile testing machine were used to characterize the microstructural transformation and mechanical properties of high-strength anti-seismic rebar. The results shows that under the conditions of different final cooling temperatures, the microstructure of the rebar were mainly composed of ferrite and pearlite. When the final cooling temperature decreased from 750 °C to 650 °C, the ferrite grain size decreased from 0.01237 mm to 0.00678 mm and the pearlite lamellar spacing decreased from 0.226 μm to 0.114 μm. The EBSD results found that the most of ferrite grains with larger misorientation angle (20° ~ 60°) formed by the different austenite grains. The TEM results found that the main precipitates were (Nb, Ti, V) C, which precipitated on the ferrite matrix, and the shapes were oval, and the average particle sizes were about 20 ~ 30 nm. When the final cooling temperature was 650 °C, the tensile strength and yield strength of the rebar reached 712.94 MPa and 562.97 MPa, respectively, and strength yield ratio was 1.27. With the decreases in the final cooling temperature, the tensile strength and yield strength of the rebar gradually increased.

1. Introduction

With the rapid development of the construction industry, people pay more and more attention on the durability of high-strength rebar in the building structures, the higher requirements are put forward for the mechanical properties of high-strength rebar to ensure the safety of the building structure [1–3]. The combination of the microalloying technology and controlled rolling and cooling technology is a method to develop the microalloyed high-strength rebar. The excellent and ideal mechanical properties can be obtained by the optimizing alloy design and thermo-mechanical control processing (TMCP) [4–6]. The microalloying elements, such as Nb, V, and Ti are usually used to produce the ultra-high strength steel, which obtain the required microstructure and mechanical properties. These microalloying elements can improve the strength of steel by the grain refinement, solid solution, dislocation, and precipitation hardening [7–9].

There are many studies that the controlled rolling and controlled cooling can obtain the best mechanical properties of the microalloyed hot rolled steel. Jang et al [10] studied the interface precipitation of Ti–Nb and Ti–Nb–Mo microalloyed steels at 700 °C and analyzed the coarsening behavior of carbides after the aging treatment under the conditions of different temperatures and times. Huang et al [11] investigated the effect of Nb on the microstructure and properties of Ti–Mo microalloyed high-strength ferritic steel, which found that when the cooling temperature was 550 °C, the tensile strength, yield strength, and elongation of Ti–Nb–Mo
ferrite steel were 755 MPa, 712 MPa, and 22%, respectively. Ghosh and Mula [12] found that the combination of high yield strength and plasticity of the forced air cooling (FAC) and quenched specimens rolled in the $\alpha$ - $\gamma$ region was attributed to the high dislocation angle of the matrix, the formation of sub crystalline ferrite ($2 \mu m$) + large ferrite ($35 \mu m$), and the precipitation of NbC (<10 $\mu m$). Sanz et al. [13] analyzed the effect of thermo-mechanical treatment and cooling temperature on the strengthening mechanism of Nb microalloyed steel, which found that the increase of Nb content has little effect on the grain size of the microstructure, whereas the cooling temperature greatly influenced the mechanical properties. Karmakar et al. [14] studied the effect of composition and thermal mechanical processing on the microstructure, precipitation, and strengthening of Nb microalloyed steel, which found that the precipitation, strengthening, and hardness of six pass hot rolling were better than that of two pass hot rolling. In the thermo-mechanical control machining process, the large deformation of austenite below the recrystallization temperature ($T_m$) transformed the equiaxed austenite grains into the cake grains with high density near plane crystal defects, which effectively acted as the nucleation centers of ferrite and resulted in the refinement of ferrite grain [15, 16]. The solid solution and precipitation of Nb promoted the ferrite transformation, which was attributed to (I) the removal of solute Nb from the matrix, thereby reducing the resistance effect of solute Nb; and (II) the precipitation of Nb effectively provided a heterogeneous nucleation site for ferrite transformation [17–19]. Hu et al. [20] showed that the microstructure of the two-stage continuous cooling test of steel was composed of the polygonal and acicular ferrite, which exhibited the excellent strength, ductility, and toughness.

Howere, the Gleeble-3800 thermal simulator plays an important effect in the material thermo-mechanical simulation process, which can simulate the heating process, soaking process, rolling process, temperature control process, cooling process of the material. The Gleeble-3800 thermal simulation tester can perform the dynamic process simulation tests including the rolling and forging process, continuous casting smelting process, welding process, metal heat treatment process, mechanical thermal fatigue, and can measure the high temperature mechanical properties of metal materials, metal thermophysical properties, CCT curve, stress-strain curve, etc. Akhtar et al. [21] used the Gleeble-3800 thermal simulator simulate the single/multi-pass welding to bears the detail investigations on physically simulated sub-HAZs. Dong et al. [22] used the Gleeble-1500 thermal mechanical simulator systematically investigated the transformation behavior of Nb–V–Ti microalloyed ultra-high strength steel during continuous cooling. Ghosh and Mula [12] used the Gleeble-3800 thermo-mechanical simulator performed the dilatometry test to obtain the useful critical temperatures such as austenite to ferrite start transformation temperature ($Ar_s$) and austenite to ferrite finish transformation temperature ($Ar_f$), which investigated the effect of deformation temperature and cooling rate on microstructural features and mechanical properties of Nb–Ti stabilized microalloyed steel. Chen et al. [23] used the Gleeble-3800 thermal simulator mainly simulate the controled rolling and controled cooling process to study the isothermal precipitation behavior of precipitates in the tested steel.

The aforementioned studies in high-strength steel mainly focused on the aspects of controlled rolling and cooling, but the effect of final cooling temperature on the microstructure and mechanical properties of high-strength anti-seismic rebar were less studied. In this work, under the condition of controlled rolling, the microstructural transformation and mechanical properties of high-strength anti-seismic rebar under the conditions of different final cooling temperatures were studied. The relationship between microstructure, secondary phase, and mechanical properties of high-strength anti-seismic rebar was discussed. Moreover, the optimal final cooling temperature was obtained.

### 2. Experimental procedures

#### 2.1. Experimental materials

The raw materials of the experimental steel came from a 500 MPa high-strength anti-seismic rebar produced by the microalloying technology and controlled rolling and controlled cooling processes. Considering the microalloying technology, the composition design of the experimental steel mainly considered two aspects: the one was the conventional elements, namely C, Mn, and Si; and the other was the microalloying elements, namely, Nb, V, and Ti. Considering the controlled rolling and controlled cooling processes, on the one hand, the controlled rolling of the experimental steel mainly considered two aspects: the one was the hot rolling deformation and the second was the hot rolling temperature; on the other hand, the controlled cooling of the experimental steel mainly considered two aspects: the one was the cooling rate and the second was the final cooling temperature. The process route of high-strength anti-seismic rebar was as follows: 100tLD converter (hot metal + scrap) converting $\rightarrow$ Argon blowing treatment (stirring) $\rightarrow$ Ladle processing (compound microalloying) $\rightarrow$ Billet continuous casting (the section size was 160 mm $\times$ 160 mm, the length was 12.05 m) $\rightarrow$ Heating furnace heating (the soaking temperature was 1050–1180 $^\circ$C) $\rightarrow$ Nip roller (removing the oxide scale on the surface of the hot billet) $\rightarrow$ Continuous rolling mill rolling (rough rolling, intermediate
rolling, finishing rolling) \(\rightarrow\) Controlled cooling (weak penetration of water) \(\rightarrow\) Cooling bed air cooling (the upper cooling bed temperature was 830 \(\sim\) 860 \(^\circ\)C) \(\rightarrow\) Cutting, packaging \(\rightarrow\) high-strength anti-seismic rebar.

The alloy materials mainly included ferro niobium, ferro vanadium, ferro titanium, ferro manganese, and ferro silicon, the experimental steel were melted into 15 kg steel ingots in the medium-frequency induction furnace and forged into a \(\Phi\)40 mm \(\times\) 40 mm steel bar. The forging process of the steel ingots were as follows: the steel ingots were heated to 1200 \(^\circ\)C at 20 \(^\circ\)Cs for 30 min by the Gleeble-3800 thermal simulator to homogenize the austenite grains. The Gleeble-3800 thermal simulator with the nip roller were used to compress and deform by 80\% and performed the four passes of the continuous hot rolling, so that the austenite grains were repeatedly refined and compacted, and then air cooled formed a forged samples.

The chemical composition of the experimental steel was analyzed by using a carbon sulfur, nitrogen hydrogen oxygen, and ICP analyzers. The chemical composition of the experimental steel was shown in table 1.

The calculation equation of carbon equivalent (Ceq) was as follows

\[
C_{eq} = C + \frac{Mn}{6} + \frac{(Cr + V + Mo)}{5} + \frac{(Cu + Ni)}{15} \tag{1}
\]

2.2. Thermal simulation process

The forged samples were processed into a rectangular samples (100 mm \(\times\) 40 mm \(\times\) 20 mm) by wire cutting, and the plane compression test was carried out on the Gleeble-3800 thermal simulator. The thermal simulation of the experimental steel was shown in figure 1.

According to the industrial production of high-strength anti-seismic rebar, forging parameters and related reference results [3, 13, 14], the thermal simulation process route of the experimental steel was set as follows: All samples were heated to 1150 \(^\circ\)C at 20 \(^\circ\)Cs for 5 min, thereby homogenizing the austenite grains and fully dissolving the solute Nb/V/Ti. In the recrystallized and non-recrystallized zones of austenite, the temperature was reduced to the corresponding rolling temperature at 5 \(^\circ\)Cs \(\sim\) 5 \(^\circ\)Cs, and the five passes of the hot rolling were carried out. The temperature of the first pass of the hot rolling was reduced to 1050 \(^\circ\)C at 5 \(^\circ\)Cs \(\sim\) 1, the deformation was 0.3, and the deformation rate was 2 \(s^{-1}\). The temperature of the two passes of the continuous hot rolling were reduced to 950 \(^\circ\)C at 5 \(^\circ\)Cs \(\sim\) 5 \(s^{-1}\) for 5 s, the deformation and deformation rate were 0.2 and 1 \(s^{-1}\), respectively. The temperature of the three passes of the continuous hot rolling were reduced to 900 \(^\circ\)C at 5 \(^\circ\)Cs \(\sim\) 5 \(s^{-1}\) for 5 s, the deformation and deformation rate were 0.2 and 1 \(s^{-1}\), respectively. Subsequently, the hot rolled samples were cooled rapidly to the final cooling temperature (650 \(^\circ\)C, 700 \(^\circ\)C, 750 \(^\circ\)C) at 15 \(^\circ\)C–20 \(^\circ\)C \(s^{-1}\) for 30 s, and finally cooled slowly to 200 \(^\circ\)C at 0.5 \(^\circ\)Cs \(\sim\), and then air cooled.

| Steel number | C  | Si  | Mn  | P  | S  | Nb | V  | Ti | Ceq |
|--------------|----|-----|-----|----|----|----|----|----|-----|
| 1#           | 0.18 | 0.35 | 1.34 | 0.018 | 0.012 | 0.018 | 0.085 | 0.020 | 0.53 |
| 2#           | 0.18 | 0.38 | 1.40 | 0.018 | 0.012 | 0.024 | 0.086 | 0.012 | 0.52 |

Figure 1. Thermal simulation process route of the experimental steel.

Table 1. Chemical composition of the experimental steel (mass fraction, \%).

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2.3. Test methods
The thermal simulation samples were processed into a rectangular samples (10 mm × 10 mm × 10 mm). After rough and fine grinding with sandpaper, the surface of the samples were smoothed by the metallographic polishing machine (PG-IA) and were evenly corroded by 4% nitric acid alcohol. The metallographic microscope (OLYMPUSGX 71) and field emission scanning electron microscope (SUPRA40) were used to observe the microstructural morphology of the experimental steel. Selecting 100 metallographic microstructure photos of 1# ~ 2# experimental steels and the ferrite grain size were measured by Nano measurer software image analyzer. Selecting 100 SEM morphology photos of 1# ~ 2# experimental steels and the pearlite lamellar spacing were measured by Nano measurer software image analyzer.

The middle part of the thermal simulation samples were used as the tensile samples, and the samples were processed into a tensile samples (length (120 mm) × width (15 mm) × thickness (2 mm)). According to the national standard GB/T 228.1–2010 of room temperature tensile test methods, the gauge length of the samples were 120 mm. Under the condition of room temperature, the samples were stretched at 10⁻² s⁻¹ strain rate by the universal tensile testing machine (MTS810).

The samples used for EBSD were obtained by the centers of the thermal simulation samples. The EBSD samples were processed into a rectangular samples (5 mm × 5 mm × 5 mm). The samples were polished, mechanically polished, and then electropolished (perchloric acid and ethanol solution). Finally, the FEI Quanta 650 F+ HKL Channel 5 were used to scan the samples. The scanning step size was 0.65 μm, and the scanning area was 385 × 385 μm².

To observe the precipitation behavior of Nb/V/Ti, the thermal simulation samples were processed into a small circular plates (Φ 5 mm × 2 mm), which was ground to 0.01 mm. First, the mechanical thinning was performed to 30 μm, then following the ion thinning to prepare samples for TEM. The high-resolution transmission electron microscopy (TECNAI G2 F30 S-TWIN) was used to analyze the precipitation behavior and electron diffraction zone. The working voltage was 200 kV. The composition of Nb/V/Ti precipitates was analyzed by EDS equipped with TEM.

3. Results and analysis
3.1. Analysis of microstructural transformation
3.1.1. Effect of microstructural transformation
Under the observation of the metallographic microscope, the metallographic microstructures of 1# and 2# experimental steels were given in figure 2. Figures 2(a)~(f) shows that the metallographic microstructures of 1# and 2# experimental steels were mainly composed of pearlite and ferrite under the conditions of different final cooling temperatures (650 °C, 700 °C, 750 °C), and the pearlite was evenly distributed on the ferrite matrix.

Figure 2. Metallographic microstructure (OM) under the conditions of different final cooling temperatures (650 °C, 700 °C, 750 °C) (a)–(c)—1#; (d)–(f)—2#.
Under the observation of the field emission scanning electron microscope, the microstructural morphology of 1# and 2# experimental steels were given in Figure 3. Figures 3(a)–(f) shows that under the conditions of different final cooling temperatures (650 °C, 700 °C, 750 °C), the ferrite of 1# and 2# experimental steels were polygonal, and the pearlite layers were distributed on the ferrite matrix. With the decrease in the final cooling temperature, the polygonal ferrite zone reduced, and the pearlite lamellar spacing gradually refined. During the continuous hot rolling, the strain-induced precipitation of carbonitride may occur around the austenite grain boundaries [24]. With the decrease in the final cooling temperature, the precipitation of carbonitride reduced the carbon content in austenite, which was beneficial to the pearlite transformation and ferrite transformation.

Under the conditions of different final cooling temperatures, the deformed austenite cooled slowly to form the lamellar pearlite (as shown in figures 3(a)–(f)). The nucleation of pearlite needed enough time to establish the synergistic growth between ferrite and cementite sheets, and the cooperative growth mainly depended on the diffusion of carbon atoms. The sufficient time was essential to ensure the adequate carbon diffusion, which formed the continuous lamellar and established a balanced growth of the two lamellar phases. Therefore, when cooling slowly under the conditions of different final cooling temperatures after the finishing rolling, the nucleation rate of pearlite lamellar increased with time. Shanmugam et al [25] also explained that the formation of lamellar pearlite in V and Nb microalloyed steel. They also explained that the equilibrium growth of pearlite structure required the sufficient time, and the faster cooling rate deviated from the normal nucleation rate of ferrite and cementite due to the insufficient carbon diffusion, resulting in the incomplete transformation of pearlite.

3.1.2. Effect of recrystallization critical temperature ($T_{nr}$)

The recrystallization critical temperature ($T_{nr}$) is a key factor for determining the controlled rolling and cooling process and the subsequent cooling process (phase transformation process), which is very important to analyze the microstructural transformation of high-strength seismic rebar. In this work, $T_{nr}$ needs to be considered. Medina et al [26] reported that the recrystallization critical temperature of austenite ($T_{nr}$) in microalloyed steels was shown:

$$T_{nr} = 887 + 464C + (6445Nb - 644\sqrt{Nb}) + (732V - 230\sqrt{V}) + 890Ti$$

where $T_{nr}$ is the recrystallization critical temperature (°C), Nb(wt%), V(wt%), Ti(wt%), C(wt%) is the content of Nb, V, Ti, C in steel, %.

According to the equation (2), the recrystallization critical temperature of austenite ($T_{nr}$) in 1# and 2# experimental steels were 1007 °C and 1027 °C, respectively. The hot rolling temperature of first pass (1050 °C) was higher than the $T_{nr}$, and the deformation was 30%. At this time, the austenite occurred a large amount of the repeated recrystallization to form the fine austenite grains. During the continuous hot rolling and time delay, the
coarsening austenite grain was hindered due to the existence of fine carbide/nitride induced by the thermal deformation, which inhibited the growth of austenite grain and maintained the recrystallized structure of fine austenite [27, 28].

The hot rolling temperature of two pass and three pass (950 °C and 900 °C) belonged to the non-recrystallized zones of austenite, and the each pass was deformed twice (10%). At this time, the austenite occurred the non-recrystallization and was elongated along the rolling direction, which formed in a flat shape. The high-density of crystal defects, such as dislocations were found in the elongated austenite grains with large deformation. When the fine carbides/nitrides and carbonitrides nucleated on these defects, the ferrite nucleation rate greatly increased during the γ → α phase transformation. Under the conditions of different final cooling temperatures, the supercooled austenite transformed into the fine ferrite grain during the slow cooling process, and the remaining supercooled austenite transformed into the pearlite at the ferrite grain boundaries.

According to the previous research [29–33], Tnr was usually increased by the addition of microalloying elements (such as Ti and Nb), which can delay the static recrystallization of austenite by forming the carbides, nitrides and/or carbonitrides under the conditions of low temperatures. Under the condition of thermal deformation above Tnr, the austenite repeatedly occurred the recrystallization and refinement, and the area and number of austenite grain boundaries increased. Under the condition of thermal deformation below Tnr, the austenite occurred the non-recrystallization, and the increases of dislocation density significantly increased the nucleation density. During the γ → α phase transformation, the favorable conditions were provided for the nucleation of ferrite, which eventually led to the obvious reduction of ferrite grain sizes. Therefore, in this work, the addition of 0.018%Nb and 0.024%Nb significantly increased the Tnr of the experimental steel, which was consistent with the previous research results.

3.2. Analysis of grain size
The grain size statistics of 1# and 2# experimental steels were given in figure 4. Figure 4 shows that when the final cooling temperature were 750 °C, 700 °C, 650 °C, respectively, the ferrite grain sizes of 1# experimental steels were 0.01237 mm, 0.01037 mm, 0.00709 mm, respectively, and the ferrite grain sizes of 2# experimental steels were 0.01036 mm, 0.01018 mm, 0.00678 mm, respectively. The solid solution and precipitation of Nb significantly refined the ferrite grains. Due to the lattice mismatch between Nb and Fe decreased the solubility limit of Nb in the Fe-rich solid solution, which enabled the segregation of Nb at the austenite grain boundaries and reduced the free energy of austenite grain boundaries. In addition, the addition of Nb can promote the precipitation of Nb under the condition of high temperature. The precipitation particles in steel can effectively inhibit the growth of austenite grains by pinning on the grain boundaries, which can effectively refine the austenite grains, thereby resulting in the significant refinement of ferrite grains [34]. Therefore, the average ferrite grain sizes of 2# experimental steel were less than that of 1# experimental steel.

During the equilibrium phase transformation, the proeutectoid ferrite and cementite were first precipitated from the supercooled austenite. When the cooling temperature was lowered to the A1 temperature, the untransformed supercooled austenite transformed into the pearlite. However, the A3 and Acm lines can be extended to the two-phase α and Cm regions, and the supercooled austenite can be transformed into all pearlite by the form of pseudo-eutectoid transformation [35] under the conditions of high cooling rates. According to
the theory of thermodynamics, the minimum interlayer spacing of possible values was derived, as shown in equation (3) [36]

\[
S_{\text{MIN}}^{0} = \frac{2\gamma_{\alpha\theta} T_{E}}{\Delta H \Delta T}
\]

where \(\gamma_{\alpha\theta}\) is the interface energy between ferrite and cementite, \(T_{E}\) is the equilibrium temperature \(A_{1}\), \(\Delta H\) is the change in the enthalpy, and \(\Delta T\) is the degree of subcooling.

According to the equation (3), increasing the degree of subcooling (\(\Delta T\)) can effectively refine the pearlite lamellar spacing, and decreasing the final cooling temperature can effectively increase the degree of subcooling (\(\Delta T\)) [37].

According to the equation (3) combined with figure 4, when the final cooling temperature were 750 °C, 700 °C, 650 °C, respectively, the pearlite lamellar spacing of 1# experimental steels were 0.226 \(\mu m\), 0.164 \(\mu m\), 0.134 \(\mu m\), respectively, and the pearlite lamellar spacing of 2# experimental steels were 0.198 \(\mu m\), 0.156 \(\mu m\), 0.114 \(\mu m\), respectively. Therefore, with the decrease in the final cooling temperature, the pearlite lamellar spacing in 1# and 2# experimental steels significantly refined, and the pearlite lamellar spacing in 2# experimental steels clearly refined relative to that in 1# experimental steel. The decreases of final cooling temperature can increase the degree of subcooling, thereby enhancing the driving force of phase transformation. Therefore, the combination of thermal deformation and final cooling temperature can effectively refine the pearlite lamellar spacing.

3.3. Analysis of EBSD

The EBSD scanning maps of the experimental steel under the condition of final cooling temperature of 650 °C were illustrated in figure 5. The IPF (X(a) axis, Y(b) axis, Z(c) axis) maps of the RD-TD plane (figures 5(a)~(d)) shows a large number of \{111\} \(\alpha\) grains and scattered existence of \{001\} \(\alpha\) and \{101\} \(\alpha\) in the microstructure of 1# and 2# experimental steels. The low-density and dispersed existence of \{001\} \(\alpha\) grains of microstructural transformation controlled by the \{001\} \langle 100\rangle \(\gamma\) diffusion indicated that the less austenite occurred the recrystallization before the \(\gamma \rightarrow \alpha\) phase transformation began. The dispersed distribution and low-density of \{001\} \(\alpha\) grains in the non-recrystallized zones of austenite decreased the effective grain size of \{001\} oriented in ferrite. The above analysis results shows that when the final cooling temperature was 650 °C, the \{111\} \(\alpha\) grains in 2# experimental steel was obviously larger than that of 1# experimental steel.

According to the EBSD analysis of the experimental steels, the frequency distribution diagram of the average misorientation angle of 1# and 2# experimental steels were given in figures 6(a) and (c). The reference results illustrated that the misorientation angles larger than 15° were defined as high-angle grain boundaries and the
misorientation angles larger than 5° but smaller than 15° were defined as low-angle grain boundaries [11, 38–40], while the misorientation angle range of 20° ~ 60° in the frequency distribution diagram was selected as high-angle grain boundaries. Figures 6(a) and (c) found that the density of high-angle grain boundaries in 1# experimental steel was significantly higher than that of 2# experimental steel. The distribution proportion of ferrite grain boundaries was larger, the misorientation angle was between 20° and 60°, whereas the distribution proportion of ferrite grain boundaries was small, and the misorientation angle was between 0° and 20°, which made the distribution curve tend to the tail. Generally, the formation of ferrite grain summarized two types, the one was that the ferrite grains formed by the single austenite grain in the misorientation angle range of 0° ~ 20°, and the second was that the ferrite grains formed by the different austenite grains in the misorientation angle range of 20° ~ 60°.

The experimental results show that the most of ferrite grains with larger misorientation angle (20° ~ 60°) formed by the different austenite grains. These austenite grains may occur a large number of high-angle crystallization, high-density dislocation, and deformation substructure, which can be used as potential nucleation points during the γ → α phase transformation. Typically, a large number of austenite grains repeatedly occurred the recrystallization, and a large number of flattened austenite grains were formed in the non-recrystallized zones. The misorientation angle of ferrite in 1# experimental steel were mainly between 40° and 50°, whereas those in 2# experimental steel were mainly concentrated in 45°, indicating that the ferrite in 2# experimental steel has high-angle grain boundaries, which can prevent the crack growth. The average grain size distribution of ferrite were given in figures 6(b) and (d), the ferrite grain sizes of 1# and 2# experimental steels were mainly concentrated in 3–12 μm, which was consistent with the ferrite grain size statistics in figure 4.

3.4. Analysis of Nb/V/Ti composite precipitation
3.4.1. Precipitation behavior of Nb/V/Ti composite
Under the observation of TEM, the dislocation and pearlite lamellar distribution of the experimental steel were given in figure 7.
Figure 7 shows that when the final cooling temperature was 650 °C, the ferrite in 1# and 2# experimental steels was polygonal under the observation of TEM, the grain boundaries were clearly visible, the pearlite lamellar were clearly distributed on the ferrite matrix, which were neatly arranged on the ferrite matrix. Figure 7 also shows that a large number of dislocations were distributed on the ferrite matrix and grain boundaries in 1# and 2# experimental steels. Due to the deformed austenite occurred the recrystallization and non-recrystallization, the composite precipitation of microalloying elements during the continuous hot rolling increased the grain boundaries and produced a large number of dislocations.

Under the observation of TEM, the (Nb, V, Ti) C precipitates of 1# and 2# experimental steels under the condition of final cooling temperature of 650 °C were given in figures 8 and 9.

Nb, V, Ti microalloying elements have strong binding affinity with C and N, and the addition of Nb, Ti, V can lead to the precipitation of Nb, Ti, V carbides and nitrides. Figures 8(a) and 9(a) shows that the precipitates of 1# and 2# experimental steels were (Nb, V, Ti) C, which precipitated on the ferrite matrix, and the average particle sizes were about 30 nm and 20 nm, and the shapes were oval. With the decrease in the final cooling temperature, the average particle size of the (Nb, V, Ti) C precipitates in 1# and 2# experimental steels...
gradually decreased. A large number of dislocations and other defects in the ferrite matrix were observed (as shown in figures 7(a) and (b)), which were conducive to the precipitation of (Nb, V, Ti) C precipitates. Therefore, the precipitates in 2# experimental steel slightly refined than that in 1# experimental steel. The EDS results of (Nb, V, Ti) C precipitates in figures 8(b) and 9(b) shows that the (Nb, V, Ti) C precipitates in 1# and 2# experimental steels mainly contained Nb, V, Ti, where Nb was the main peak. The Nb peak in 2# experimental steel was higher than that in 1# experimental steel. The high-resolution images of (Nb, V, Ti) C precipitates in figures 8(c) and 9(c) shows that the (Nb, V, Ti) C precipitates in 1# and 2# experimental steels were the face-centered cubic structures, and their maximum aspect ratios were 1.43 and 1.08, respectively, showing that the (Nb, V, Ti) C precipitates of 2# experimental steel relatively refined. The electron diffraction images of (Nb, V, Ti) C precipitates in figures 8(d) and 9(d) shows that the (Nb, V, Ti) C precipitates have a certain B-N orientation relationship with ferrite matrix [4, 16, 17, 23], namely, (Nb, V, Ti) C|| (100)α and [478] (Nb, V, Ti) C|| (001)α (figure 8(d)) as well as (321)(Nb, V, Ti) C|| (100)α and [133](Nb, V, Ti) C|| (001)α (figure 9(d)). Therefore, the (Nb, V, Ti) C precipitates in 1# and 2# experimental steels not only existed by the form of strain-induced precipitation in deformed austenite, but also formed in ferrite during and after the γ → α phase transformation, which can be expected in [41–43].

3.4.2. Effect of Nb/V/Ti Composite Precipitation
The precipitation of nano-carbides has an important effect on the nucleation and grain refinement of ferrite, and the precipitation of carbides is mainly related to the final cooling temperature. Figures 8 and 9 shows that when the final cooling temperature was 650 °C, the main precipitates of the experimental steel were Nb-rich carbides ((Nb, V, Ti) C), and the average particle sizes were about 20 ~ 30 nm. The references [44, 45] pointed out that the precipitated phase of nano-carbides mainly formed during the rolling and coiling process, which was the main reason for increasing the strength of high-strength steel. After the final rolling temperature of 900 °C, the Nb-rich carbides precipitated on the ferrite matrix and dislocations under the conditions of different final cooling temperatures.

Figures 10(a) and (b) shows that with the decrease in the final cooling temperature, the average particle sizes and proportions of Nb-rich carbides ((Nb, V, Ti) C) in 1# and 2# experimental steel gradually decreased. When the final cooling temperature was 650 °C, the average particle sizes of Nb-rich carbides ((Nb, V, Ti) C) in 1# and 2# experimental steels were about 28.5 nm and 18.5 nm, respectively and the proportions were 48.5% and

Figure 9. (Nb, V, Ti) C precipitates of 2# experimental steel under the condition of final cooling temperature of 650 °C (a) Morphology of (Nb, V, Ti) C precipitates, (b) EDS of (Nb, V, Ti) C precipitates, (c) High-resolution images of (Nb, V, Ti) C precipitates, (d) Selected area electron diffraction of (Nb, V, Ti) C precipitates.
58.5%, respectively, indicating that the average particle size of Nb-rich carbides ((Nb, V, Ti)C) in 2# experimental steel was significantly refined relative to that in 1# experimental steel.

As well known, when the final cooling temperature decreased, the degree of supercooling of the precipitated phase increased, which led to an increase in the driving force for the nucleation of the precipitated phase, thereby reducing the critical size of nucleation. The ferrite and pearlite in Nb–Ti–V steel formed during the \( \gamma \rightarrow \alpha \) phase transformation under the conditions of lower final cooling temperatures, which can provide a large number of non-uniform nucleation centers for carbides, reducing the activation energy of nucleation \([46]\), and the precipitated phase of carbides was easy to precipitate on the ferrite matrix and the dislocation line, and the ferrite grain size and the pearlite lamellar spacing were refined. The previous studies \([47]\) showed that the addition of Nb can increase the supersaturation of Nb/Ti/V in the matrix, thereby increasing the driving force of precipitation and significantly increasing the strain-induced precipitation. Therefore, the particle size of precipitates in 2# experimental steel was slightly smaller than that in 1# experimental steel.

Due to the strain-induced precipitation occurred in austenite, under the conditions of high temperatures, with the help of dislocation tube diffusion and bulk diffusion, the precipitates was easy to coarsen \([48]\). The grain size of the precipitated phase in austenite was generally larger than that in ferrite. In addition, although the strain-induced precipitation will inhibit the growth of austenite grains, the precipitation will consume the content of microalloying elements in steel, resulting in the reduction in the number of microalloying elements precipitated on the ferrite during the final cooling process and the reduction in the average particle size of precipitates.

The above analysis shows that the final cooling temperature has a greater impact on the precipitation of carbides. These precipitates mainly precipitated on the matrix during or after the \( \gamma \rightarrow \alpha \) phase transformation under the conditions of different final cooling temperatures after finishing hot rolling at 900 °C, which provided a favorable conditions for ferrite nucleation and grain refinement. With the decrease in the final cooling temperature, the average particle size of precipitates in 1# and 2# experimental steels gradually decreased.

3.5. Analysis of mechanical properties

The tensile curves and the tensile data of 1# and 2# experimental steels under the conditions of different final cooling temperatures were given in figure 11 and table 2.

Figure 11 and table 2 shows that with the decrease in the final cooling temperature, the tensile strength and yield strength of 1# and 2# experimental steels gradually increased. When the final cooling temperature was 650 °C, the tensile strength and yield strength of 1# experimental steel were 655.62 MPa and 500.28 MPa, respectively, and the strength yield ratio was 1.31; the tensile strength and yield strength of 2# experimental steel were 712.94 MPa and 539.24 MPa, respectively, and the strength yield ratio was 1.32. During the hot rolling process, a large amount of deformation caused the dynamic recrystallization and non-recrystallization of austenite, and the fine (Nb, V, Ti)C precipitates were distributed in the austenite grain boundaries, which promoted the pearlite transformation and ferrite transformation during the slow cooling process. In the stretching process, the ferrite first assumed the deformation, which finally broke on the pearlite structure. Due to the microstructure of 2# experimental steel was obviously refined relative to that of 1# experimental steel, resulting in the tensile strength of 2# experimental steel was greater than that of 1# experiment steel.
Figure 11. Tensile curves of 1# and 2# experimental steels under the conditions of different final cooling temperatures. (a) Stress-strain curve, (b) Yield platform.

Figure 12. Effect of ferrite grain size on the tensile strength and strength yield ratio.

Table 2. Tensile data of 1# and 2# experimental steels under the conditions of different final cooling temperatures.

| Steel number | Tensile strength/MPa | Yield strength/MPa | Strength yield ratio | Elongation after breaking/% | Maximum force total elongation/% |
|--------------|----------------------|--------------------|----------------------|-----------------------------|---------------------------------|
| 1# 650 °C    | 655.62               | 500.28             | 1.31                 | 24                          | 21.3                            |
| 700 °C       | 617.09               | 453.22             | 1.36                 | 23                          | 20.8                            |
| 750 °C       | 605.58               | 447.03             | 1.35                 | 25                          | 22.4                            |
| 2# 650 °C    | 712.94               | 539.24             | 1.32                 | 24                          | 20.3                            |
| 700 °C       | 638.75               | 464.71             | 1.37                 | 23                          | 22.0                            |
| 750 °C       | 612.07               | 451.23             | 1.36                 | 26                          | 22.1                            |

Figure 12 shows that the ferrite grain size of the experimental steel has a more obvious effect on the tensile strength, but a small effect on the strength yield ratio. With the increase in the ferrite grain size, the tensile strength of the experimental steel gradually decreased, and the strength yield ratio varied between 1.310 and 1.363. Due to the strength of the experimental steel depended on the ferrite grain size, the smaller the ferrite grain size, the higher the strength. However, the degree of grain refinement was determined by the combination of the precipitation of microalloying elements and the hot rolling process. In the experimental steel, a large amount of thermal deformation occurred in the recrystallized and non-recrystallized zones of austenite to form...
the high-density crystal defects and the fine precipitates of microalloying elements, which pinned the austenite grain boundaries and hindered the growth of austenite grains. During the $\gamma \rightarrow \alpha$ phase transformation process, the fine ferrite grains were formed, and the higher strength was obtained during the stretching process.

Figure 11 and table 2 also shows that the plasticity of 1# and 2# experimental steels was better under the conditions of different final cooling temperatures. When the final cooling temperature was 750°C, the tensile fracture elongation of 1# experimental steel was 22.4%, and the plasticity was best. Honeycombe et al.[49] showed that the precipitates was usually located on the 100 plane of ferrite, which was closest to being parallel to the austenite/ferrite interface. Therefore, when the transformation front passes through the austenite, the repeated nucleation of precipitates formed this linear microstructure[50]. The linear distribution of precipitates can reduce the strain concentration, thereby improving the plasticity. Therefore, under the conditions of different final cooling temperatures, the plasticity of 1# experimental steel was greater than that of 2# experimental steel.

4. Conclusions

In this work, the Gleeble-3800 thermal simulator was used to simulate and control the final cooling temperature process to study the microstructural transformation and the precipitation characteristics of microalloying elements. The conclusions were as follows:

(1) The microstructure of 1# and 2# experimental steels were mainly composed of ferrite and pearlite. The ferrite was polygonal, and pearlite lamellar spacing was distributed on the ferrite matrix. With the decrease in the final cooling temperature, the ferrite grain size and pearlite lamellar spacing gradually decreased.

(2) The EBSD results shows that when the final cooling temperature was 650°C, the \{111\}$_\alpha$ grains in 2# experimental steel was obviously larger than that of 1# experimental steel. The most of ferrite grains with larger misorientation angle (20°–60°) formed by the different austenite grains, which hindered the crack propagation.

(3) The main precipitates in 1# and 2# experimental steels were (Nb, Ti, V) C, which precipitated on the ferrite matrix and the average particle sizes were about 30 nm and 20 nm, respectively. The shapes of the (Nb, Ti, V) C precipitates were oval, and the crystal structure was the face-centered cubic structure.

(4) When the final cooling temperature was 650°C, the tensile strength and yield strength of 1# experimental steel were 655.62 MPa and 522.76 MPa, respectively, and the strength yield ratio was 1.26; the tensile strength and yield strength of 2# experimental steel were 712.94 MPa and 562.97 MPa, respectively; and the strength yield ratio was 1.27. With the decrease in the final cooling temperature, the tensile strength and yield strength of 1# and 2# experimental steels gradually increased.

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Data availability statement

No new data were created or analysed in this study.

Conflicts of 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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