Research Article

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Effect of cooling rate and Nb synergistic strengthening on microstructure and mechanical properties of high-strength rebar

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Abstract: Rebar is an extremely important building material. The cooling rate and the presence of niobium (Nb) element are key factors influencing the overall performance of rebars. In this work, the high-strength rebar's microstructure, precipitated phase, and mechanical properties were characterized using scanning electron microscopy, transmission electron microscopy, HVS-1000 hardness tester, and MTS810 universal tensile testing machine. The results showed that a shift in cooling rate from 0.3 to 3°C·s⁻¹ resulted in noticeable changes in the microstructures of rebars, particularly between Nb-free and Nb-containing variants. In the case of Nb-containing rebars, there was an increase of 8.26% in the proportion of pearlite, along with a decrease of 10.63 μm in the average grain size of ferrite. Furthermore, the lamellar spacing of pearlite experienced a decrease of 0.0495 μm, the proportion of low-angle grain boundaries saw an increment of 4.13%, and the size of the precipitated phase (Nb, Ti, V) C reduced by 18.9 nm. These changes collectively led to a significant increase in hardness (98.56 HV), yield strength (179.02 MPa), and ultimate strength (199.43 MPa). The resultant fracture morphology manifested as a dimple pattern.

Keywords: cooling rate, Nb, high-strength rebar, microstructure, precipitation behavior, mechanical properties

1 Introduction

Rebar, a crucial building material, and concrete exhibit excellent bonding performance and a similar temperature linear expansion coefficient, complementing each other. The rebar within a building bears the tensile forces of the concrete structure and increases the tensile strength of the concrete [1–3]. Acting as a strong and ductile material, rebars play the role of ridge beams in buildings, effectively bolstering the stability and seismic resilience of structures. Elevating the cooling rate and introducing the Nb element can notably enhance the overall performance of the rebar.

Within a specific range, as the cooling rate advances, there is an increase in the extent of supercooling. Consequently, the phase transition temperature from austenite to ferrite decreases, resulting in a reduction in the nucleation rate of ferrite, ultimately resulting in smaller ferrite grain sizes [4,5]. Liu et al. [6] thought that the grain size will affect the phase transformation process. Zheng et al. [7] discovered that the coarse ferrite grains cause the hot rolled plate containing niobium (Nb) to crack. As the cooling rate increases, the size of carbonitride precipitates decreases due to insufficient ripening and growth time [8]. As the most typical micro-alloying element, Nb primarily dissolves in the matrix or precipitates as carbides and carbonitrides. The dissolution of Nb within the austenite matrix has the potential to elevate the recrystallization temperature of austenite, enlarge the recrystallization zone of austenite, and defer the onset of recrystallization behavior in deformed austenite. This proves advantageous in facilitating the conversion of austenite to ferrite and achieving a more refined microstructure. The diminutive and widely scattered carbonitride formed during the cooling process acts as a deterrent at the grain boundary, obstructing the growth of ferrite grains, impeding dislocation movement, and contributing to the enhancement of steel strength [9,10]. Besides Nb, microalloying elements like Ti and V are commonly

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introduced for composite strengthening. The underlying principle involves leveraging the distinct solubility of various elements in austenite to fulfill diverse roles. Carbides of Ti and V are primarily employed to prevent grain coarsening and fortify the precipitation strengthening effect during the austenite reheating process. The synergistic presence of Nb, V, and Ti can lead to the formation of composite carbides or carbonitrides. The precipitation of these composite precipitates serves not only to refine austenite grain during high-temperature deformation but also to encourage the shift from austenite to ferrite, refine the dimensions of ferrite grains, and strengthen the robustness and resilience of the steel. Xin et al. [11] discovered that when subjected to a high cooling rate, the structure of ferrite grains in Nb-Ti high-strength steel transformed from polygonal to lath-shaped, resulting in finer pearlite and a significant enhancement in hardness. Shanmugam et al. [12] found that (Ti, Nb, V) C or (Nb, V) C precipitated during the cooling process of Nb-V-Ti microalloyed steel. Tianyou et al. [13] discovered that a longer isothermal time is beneficial to refine the microstructure of Nb-containing rebars and improve the mechanical properties. Olasolo et al. [14] found that the lower cooling rate is beneficial to obtain ferrite and pearlite microstructure in Nb/V microalloyed steel. Huang et al. [15] studied the effect of hot deformation on the microstructure and properties of microalloyed steel. Sheng et al. [16] analyzed the microstructure and properties of four kinds of rebars with different Nb contents, and found that the mechanical properties of rebars were improved best when the Nb content was 0.023%. Bansal et al. [17] designed a Nb-containing alloy to study its phase transformation behavior. Dong et al. [18] studied the transformation behavior of Nb-V-Ti microalloyed ultra-high strength steel during continuous cooling. The above studies primarily focused on the influences of alloying components and cooling rates on the comprehensive properties of steel, but there is still a gap in understanding the combined strengthening impact of cooling rate and Nb on rebars.

The aim of this study is to optimize the microstructure and precipitated phases of the rebar by modifying the cooling rate and Nb concentration of the rebar, thereby enhancing its mechanical properties. The study examines the influence of Nb and cooling rate on the microstructure, precipitation dynamics, and mechanical characteristics of high-strength Nb-Ti-V rebar. Employing diverse analysis techniques such as scanning electron microscopy (SEM), transmission electron microscopy (TEM), HVS-1000 hardness tester, and MTS810 universal tensile testing machine, the research identifies the most effective cooling rate and optimal Nb content. This exploration provides valuable theoretical insights for advancing high-strength rebar development.

2 Experimental materials and processes

2.1 Material preparation

The raw materials utilized for the test steel consisted of 500 MPa rebars, which were smelted by a steel group. Two groups of test steels were formulated: one excluding the Nb element, and the other including 0.023% Nb element. The re-melting process employed a medium-frequency induction furnace with a capacity of 50 kg. After the melting process was completed, the cylindrical billet, measuring 40 mm in height and 200 mm in diameter, was subjected to forging and air-cooled until it reached room temperature. The determination of Si, Mn, P, V, and Ti contents was conducted through inductively coupled plasma mass spectrometry (ICP-AES/MS), while C and S contents were analyzed using an inorganic carbon-sulfur analyzer. Testing for each set of test steel included five parallel samples, with the average values being recorded. The chemical composition of the test steel is presented in Table 1.

The formula for calculating the carbon equivalent (C_eq) is as follows [19]:

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

2.2 Experimental process

2.2.1 Austenite recrystallization critical temperature (T_{nr})

The critical temperature for austenite recrystallization (T_{nr}) has been elevated by several scholars through the

### Table 1: Chemical composition of the test steel (mass fraction, %)

<table>
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<tr>
<th>Steel number</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Nb</th>
<th>V</th>
<th>Ti</th>
<th>C_{eq}</th>
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<tr>
<td>1#</td>
<td>0.16</td>
<td>0.49</td>
<td>1.55</td>
<td>0.019</td>
<td>0.013</td>
<td>0</td>
<td>0.065</td>
<td>0.013</td>
<td>0.431</td>
</tr>
<tr>
<td>2#</td>
<td>0.17</td>
<td>0.50</td>
<td>1.53</td>
<td>0.019</td>
<td>0.012</td>
<td>0.023</td>
<td>0.064</td>
<td>0.012</td>
<td>0.438</td>
</tr>
</tbody>
</table>
incorporation of minute quantities of alloying elements such as Nb, V, and Ti [20–22]. The calculation formula for the austenite recrystallization critical temperature \( T_{nr} \) has been consolidated by Medina and Mancilla [23], as depicted in equation (2):

\[
T_{nr} = 887 + 464C + (6.445Nb - 644\sqrt{Nb}) + (732V - 230\sqrt{V}) + 890Ti + 363Al - 3,57Si,
\]

(2)

where \( T_{nr} \) represents the critical temperature of austenite recrystallization (°C), and Nb, V, Ti, C, Al, and Si denote the mass fractions of each element (wt%) in the steel, respectively.

According to equation (2), the \( T_{nr} \) of the 1# test steel is 884.58°C, and the \( T_{nr} \) of the 2# test steel is 937.69°C. This shows that the inclusion of Nb results in an increase in the recrystallization temperature of austenite in the test steel.

### 2.2.2 Thermal simulation process

Figure 1 illustrates the thermal simulation process. The experiments utilized a digitally regulated Gleeble-3800 thermal simulator. The thermal simulation specimen, with dimensions of 100 mm × 40 mm × 20 mm, was heated at a rate of 5°C·s\(^{-1}\) until it reached 1,150°C, where it was maintained for 5 min. Subsequently, it was cooled to 900°C at a rate of 10°C·s\(^{-1}\) for 40% rolling deformation, then held for 30 s to eliminate internal temperature gradients. Finally, the sample was gradually cooled to ambient temperature at rates of 0.3, 0.5, 1, 2, and 3°C·s\(^{-1}\).

### 2.3 Test method

Figure 2 is the schematic diagram of the experimental process of this study. The experimental raw materials were characterized after melting, forging, and thermal simulation. The specific characterization methods are as follows:

1. The critical point temperature of phase transition was determined through thermal expansion curve calculation. Thermal expansion data were documented, and the thermal expansion curve was generated using Origin software. The phase transition point was identified by applying the tangent method. Phase transformation point data and steel
composition data were imported into Jmatpro software, followed by the export of generated data into Origin software for acquiring the continuous transformation curve (CCT) of undercooled austenite.

The thermal simulation specimen was shaped into a cube with dimensions of 10 mm × 10 mm × 10 mm. Following this, it underwent cold inlay, rough grinding, and fine grinding, followed by polishing using a PG-IA polishing machine and corrosion using 4% nitric acid in alcohol. Subsequently, the microstructure was examined using a SUPRA40 field emission SEM.

For analysis, 100 metallographic micrographs of the test steel were selected. Quantitative analysis of pearlite and ferrite proportions was conducted using Ipwin32 image processing software. The ferrite grain size was determined with a Nano measurer software image analyzer. The morphology of pearlite and ferrite in the test steel was observed through 100 SEM scanning images, and the spacing of pearlite lamellae was measured using the same software.

The upper and lower surfaces of the thermal simulation specimens were leveled, and hardness was assessed using an HVS-1000 hardness tester under a load of 0.1 kg for 15 s. Twenty points were selected on each group of test samples for measurement, and the results were averaged.

Thermal simulation specimens were sectioned into 100 mm × 10 mm × 2 mm tensile samples along the rolling direction and elongated using an MTS810 universal tensile testing machine. Each set of test samples underwent three experiments, and the results were averaged. According to the obtained stress–strain curve, the energy absorption value of the tensile process of the sample is calculated. The fracture morphology perpendicular to the tensile direction was examined using a SUPRA40 field emission SEM.

The thermal simulation sample was processed into a 5 mm × 5 mm × 5 mm cube for EBSD analysis. The sample was mechanically polished, electropolished, and scanned with a step size of 0.65 μm over an area of 600 μm × 600 μm.

The thermal simulation specimen underwent a transformation into a wafer measuring 5 mm in diameter and 2 mm in thickness. Subsequently, it underwent coarse grinding to achieve a thickness of 0.2 mm, followed by mechanical thinning to 20 μm, and finally ion-thinning. The morphology of the Ni-Ti-V precipitated phase was observed under a TalosF200X field emission TEM, and the Smartedx energy dispersive spectrometer (EDS) was utilized to examine the composition of the precipitated phase containing Ni-Ti-V.

3 Results

3.1 CCT curve analysis

Figure 3 illustrates the CCT of undercooled austenite for both 1# and 2# test steels. When the cooling rate increased from 0.3 to 3°C·s⁻¹, ferrite transformation and pearlite transformation occurred in the tested steels. Figure 3(a) shows that with the increase in cooling rate from 0.3 to 3°C·s⁻¹, the starting transition temperature of Nb-free ferrite decreases from 820 to 810°C, the starting transition

Figure 3: CCT curve of test steel: (a) 1# test steel and (b) 2# test steel.
Figure 4: Microstructure and morphology of test steel under different cooling rates: (a)–(e) 1# test steel and (f)–(j) 2# test steel.
temperature and ending transition temperature of pearlite decrease from 690 and 640°C to 670 and 600°C, respectively, and the temperature range of pearlite transformation is expanded. Figure 3(b) shows that with the increase in cooling rate from 0.3 to 3°C·s⁻¹, the starting transition temperature of Nb-bearing steel ferrite decreases from 810 to 800°C, the starting transition temperature and ending transition temperature of pearlite decrease from 690 and 630°C to 660 and 580°C, respectively, and the temperature range of pearlite transformation is expanded. By comparing Figure 2(a) and (b), it is evident that compared with Nb-free steel, the austenite starting transformation temperature of the Nb-containing steel decreases, the ferrite starting formation temperature decreases, and the cooling rate range for pearlite transformation is expanded.

3.2 Microstructure analysis

The SEM morphology of five groups of test samples with different cooling rates after rolling for 1# and 2# test steels is showcased in Figure 4. The microscopic tissue proportion statistics, ferrite average size statistics, and pearlite lamellar spacing statistics for these groups are displayed in Figure 5.

Figure 4 illustrates that the predominant microstructures in the examined steels are randomly shaped ferrite and predominantly grayish-white pearlite. The pearlite lamellae are uniformly dispersed within the ferrite matrix, and the boundaries between grains are distinctly outlined. With an increase in cooling rate from 0.5 to 3°C·s⁻¹, the Nb-containing steel, compared to Nb-free steel, shows a more refined grain structure, increased grain boundaries, and denser pearlite lamellar arrangement. The ferrite grains and pearlite lamellar in the Nb-containing steel appear more consistently arranged.

To further validate the microstructure refinement, quantitative statistical analysis was conducted on the average grain size of ferrite and the interlamellar spacing of pearlite for two sets of experimental steels at various cooling rates. As shown in Figure 5(a), with cooling rates increasing from 0.3 to 3°C·s⁻¹, the proportion of ferrite in 1# and 2# test steels decreases from 82.89% to 74.63%, while pearlite increases from 17.11% to 25.37%. According to Figure 5(b), as the cooling rate increases from 0.3 to 3°C·s⁻¹, the average grain size of ferrite in 2# test steel decreases from 19.18 to 8.55 μm compared to 1# test steel. Additionally, the pearlite lamellar spacing reduces from 0.1321 to 0.1109 μm.

As the cooling rate increases, both sets of test steels exhibit a greater proportion of pearlite. Simultaneously, the average grain size of ferrite decreases significantly, and the interlamellar spacing of pearlite also decreases notably. In comparison to Nb-free steel, Nb-containing steel exhibits a higher proportion of pearlite, smaller ferrite grains, and reduced pearlite lamellar spacing.

The increased cooling rate induces greater supercooling, leading to a gradual reduction in ferrite nucleation. This process impedes grain boundary movement and ferrite growth, resulting in finer ferrite grains. The increased cooling rate hinders grain boundary movement and impedes ferrite growth, resulting in the refinement of ferrite grains. The lamellar spacing of pearlite primarily depends on the formation temperature of

![Figure 5](image_url)
pearlite. As the cooling rate increases, the increased supercooling decreases the austenite-to-pearlite transformation temperature, limiting the time available for cementite growth and consequently reducing lamellar spacing [24].

Additionally, the inclusion of Nb introduces carbonitrides around austenite grain boundaries during the cooling process. This precipitation reduces austenite's carbon content, aiding the transformation of undercooled austenite into pearlite. The presence of Nb composite carbides on austenite grain boundaries restricts grain boundary movement, leading to smaller ferrite grain sizes. Nb dissolved in the austenite matrix also lowers the pearlite transformation temperature and decreases lamellar spacing [25].

**Figure 6:** TEM images of precipitated phase in experimental steel: (a) 1# test steel at 0.3°C·s⁻¹ (V, Ti) C TEM, (a1) Enlarged TEM, (a2) EDS, and (a3) SAED; (b) 1# test steel at 3°C·s⁻¹ (V, Ti) C TEM, (b1) Enlarged TEM, (b2) EDS, and (b3) SAED; (c) 2# test steel at 0.3°C·s⁻¹ (Nb, Ti, V) C TEM, (c1) Enlarged TEM, (c2) EDS, and (c3) SAED; and (d) 2# test steel at 3°C·s⁻¹ (Nb, Ti, V) C TEM, (d1) Enlarged TEM, (d2) EDS (d3) SAED.
3.3 Analysis of Nb-Ti-V composite carbides

TEM images, EDS, and SAED patterns of two groups of test samples with different cooling rates after rolling 1# test steel and 2# test steel are presented in Figure 6. The precipitates exhibit an ellipsoidal or approximately spherical shape. Figure 7 illustrates the variation in the average precipitate size for 1# test steel and 2# test steel at different cooling rates ranging from 0.3 to 3°C·s⁻¹. In contrast to 1# test steel, 2# test steel shows a reduction in the average precipitate size, decreasing from 56.87 to 37.97 nm as the cooling rate increases.

The EDS data (Figure 6(a2)–(d2)) show that the precipitated phase in 1# test steel contains significant amounts of Ti and V. Notably, the high cooling rate sample exhibits elevated concentrations of Ti and V compared to the low cooling rate sample, along with smaller precipitate sizes. Besides the prominent Fe peak, the predominant precipitated phase primarily consists of (V, Ti) C. The atomic ratio of Ti to V is approximately 0.37:1.05 and 0.26:1.01, respectively. In 2# test steel, the precipitates contain Ni, V, and Ti, with higher Nb content and finer size in high cooling rate samples. Apart from the Fe peak, the precipitates consist mainly of (Nb, Ti, V) C, with atomic ratios of Nb, Ti, and V at 8.26:7.48:1.94 and 15.89:6.44:1.21. The precipitates exhibit a face-centered cubic structure. The lattice fringe spacing in Figure 6(a3)–(d3) measures 0.243, 0.211, 0.154, and 0.118 nm, respectively.

In Figure 6(a3)–(d3), the SAED patterns reveal a distinct orientation relationship between the (V, Ti) C precipitated phase and the matrix of 1# test steel. As the cooling rate increases from 0.3 to 3°C·s⁻¹, the size of precipitates in Nb-containing steel notably decreases compared to Nb-free steel, as observed in Figures 6(a1)–(d1) and Figure 7. Classical nucleation and growth theory suggest that faster cooling rates result in shorter aging times for precipitated phases, leading to smaller precipitate sizes, which contributes to the inhibition of grain growth [26,27].

Elevated concentrations of Ti and V compared to low cooling rate samples, along with smaller precipitate sizes, are observed in 2# test steel. Notably, the high cooling rate sample exhibits a reduction in the average precipitate size, decreasing from 56.87 to 37.97 nm as the cooling rate increases.

3.4 EBSD analysis

The EBSD scanning images in Figure 8 depict the microstructures of two distinct groups of test samples, namely, 1# and 2# experimental steels, subjected to varying cooling rates. When examining the inverse pole figure distribution (IPF) in Figure 8(a)–(d), it is evident that both 1# and 2# experimental steels exhibit a considerable presence of (111) ferrite grains, accompanied by a notable presence of (001) ferrite and (101) ferrite. The microstructure of both test steels reveals the presence of recrystallized and non-recrystallized deformed austenite, resulting in a substantial population of (111) α grains characterized by high-angle grain boundaries and a dense dislocation distribution. As the cooling rate is increased from 0.3 to 3°C·s⁻¹, a comparative analysis with 1# test steel indicates that the (111) α grains in 2# test steel exhibit a more refined structure, with an increased quantity and a more uniform distribution.

In Figure 8(a1)–(d1), the contrast in the grain boundary and diffraction band (GB + BC) reveals noticeable improvements in the microstructure of 2# test steel compared to 1# test steel as the cooling rate increases from 0.3 to 3°C·s⁻¹. The ferrite grains in the 2# samples exhibit a finer structure, showcasing a more uniform distribution and an
Figure 8: EBSD scanning image of the experimental steel: (a) 1# test steel at 0.3°C·s⁻¹ IPF, (a1) GB + BC, and (a2) KAM; (b) 1# test steel at 3°C·s⁻¹ IPF, (b1) GB + BC, and (b2) KAM; (c) 2# test steel at 0.3°C·s⁻¹ IPF, (c1) GB + BC, and (c2) KAM; and (d) 2# test steel at 3°C·s⁻¹ IPF, (d1) GB + BC, and (d2) KAM.
Figure 9: Distribution of ferrite dislocation angle and ferrite grain size in the experimental steel: (a) 1# test steel at 0.3°C·s⁻¹, (b) 1# 3°C·s⁻¹, (c) 2# test steel at 0.3°C·s⁻¹, (d) 2# test steel at 3°C·s⁻¹, (e) 1# 0.3 test steel at°C·s⁻¹, (f) 1# test steel at 3°C·s⁻¹, (g) 2# test steel at 0.3°C·s⁻¹, and (h) 2# test steel at 3°C·s⁻¹.
increased quantity. Simultaneously, there is an increase in high-angle grain boundaries. Additionally, a substantial presence of dislocations and sub-grain boundaries is dispersed along the ferrite grain boundaries.

From the local orientation difference (KAM) of Figure 8(a2)–(d2), with the cooling rate increasing from 0.3 to 3°C·s⁻¹, compared with 1# test steel, the lattice distortion area on the ferrite matrix of 2# test steel increases, the distribution is more dispersed, and the density is higher. The primary concentration of lattice distortion occurs at the boundaries of the matrix, establishing a specific coincidence relationship with dislocations and sub-grain boundaries.

The distribution diagram and fitting curve of ferrite misorientation angle and ferrite grain size for both test steels at cooling rates of 0.3 and 3°C·s⁻¹ are illustrated in Figure 8, as determined by EBSD analysis of the experimental steel. Traditionally, a misorientation angle falling within the range of 15°–45° is categorized as a high-angle grain boundary, while an angle less than 5° is designated as a low-angle grain boundary. In conjunction with the findings presented in Figure 8 and Figure 9(a)–(d), a comparison between the 1# and 2# test steels reveals an increase in the ratio of low-angle grain boundaries for the 2# test steel, rising from 2.69 to 10.88%. Simultaneously, the refinement of the ferrite matrix structure results in an escalation in the number of high-angle grain boundaries. When comparing Figure 9(a) and (c) to Figure 9(b) and (d), it becomes apparent that, under identical cooling rates, the 2# test steel exhibits a higher percentage of grain boundaries with shallow angles compared to the 1# test steel, featuring a more concentrated distribution of grain boundaries with steep angles. Further analysis by comparing Figure 9(a) and (b) with Figure 9(c) and (d) reveals that, for a given test steel, the specimen cooled at a rate of 3°C·s⁻¹ displays an elevated ratio of grain boundaries with small angles in contrast to the specimen cooled at 0.3°C·s⁻¹. Additionally, the distribution of grain boundaries with steep angles is more centered around 45°.

In summary, 2# test steel exhibits a greater number of high-angle grain boundaries, effectively hindering crack diffusion. Additionally, the distribution of low-angle grain boundaries in the 2# test steel becomes more dispersed, enhancing its strength. This observation aligns with the EBSD scanning results. The ferrite grains comprising these grain boundaries are formed by individual and multiple austenite grains [31,32]. These austenite grains may contain numerous high-density dislocations and deformation sub-structures, serving as potential nucleation sites in the γ → α phase transformation process. Figure 9(e)–(h) depict the ferrite grain size distribution and fitting curve for both test steels at 0.3 and 3°C·s⁻¹ cooling rates. It is visually evident that as the cooling rate increases, the ferrite size in Nb-containing steel significantly reduces compared to Nb-free steel. This observation, in conjunction with the information from Figure 5(b), reinforces the positive impact of increasing the cooling rate on reducing ferrite nucleation, decreasing ferrite grain size, and refining grains.

Table 2: Mechanical properties of the test steels

<table>
<thead>
<tr>
<th>Steel number</th>
<th>Cooling rate (°C·s⁻¹)</th>
<th>$R_{\text{dl}}$ (MPa)</th>
<th>$R_{\text{m}}$ (MPa)</th>
<th>$R_{\text{m}}/R_{\text{dl}}$</th>
<th>$A$ (%)</th>
<th>$A_{\text{eff}}$ (%)</th>
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<tr>
<td>1#</td>
<td>0.3</td>
<td>523.27 ± 3</td>
<td>655.06 ± 5</td>
<td>1.25</td>
<td>14.27 ± 2.4</td>
<td>12.24 ± 2.2</td>
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<tr>
<td></td>
<td>0.5</td>
<td>532.06 ± 5</td>
<td>668.99 ± 6</td>
<td>1.29</td>
<td>14.95 ± 2.8</td>
<td>12.15 ± 2.5</td>
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<td>1</td>
<td>541.95 ± 5</td>
<td>677.13 ± 4</td>
<td>1.25</td>
<td>13.45 ± 2.4</td>
<td>11.56 ± 2.3</td>
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<td></td>
<td>2</td>
<td>550.43 ± 4</td>
<td>696.82 ± 7</td>
<td>1.27</td>
<td>12.81 ± 2.5</td>
<td>11.18 ± 2.8</td>
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<td>3</td>
<td>577.42 ± 6</td>
<td>726.40 ± 5</td>
<td>1.26</td>
<td>12.60 ± 2.8</td>
<td>10.77 ± 2.7</td>
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3.5 Mechanical properties and fracture analysis

Figure 10 illustrates the correlation between cooling rates and microhardness of the test steel, ranging from 0.3 to 3°C·s\(^{-1}\). A significant increase in hardness is observed when comparing 1# test steel with 2# test steel, with hardness values increasing from 219.21 HV to 317.77 HV as the cooling rate escalates. This is because the increase in cooling rate promotes the transformation of undercooled austenite to lamellar pearlite, the content of pearlite in steel increases, and the hardness of pearlite itself is greater than that of ferrite, so the hardness is improved. The pearlite lamellar spacing is significantly reduced, and the phase interface between ferrite and cementite in unit volume steel increases, which hinders the movement of dislocations, that is, the plastic deformation resistance increases, and the hardness also increases. The ferrite grains are refined, the total grain boundary area increases, the more dislocations, the more grains of different orientations that need to be coordinated, and the hardness increases significantly [6,33,34]. Simultaneously, the refinement of ferrite grains, the augmentation of total grain boundary area, and the increased presence of dislocations among grains with varying orientations significantly contribute to the enhanced hardness. Furthermore, at equivalent cooling rates, 2# test steel exhibits greater hardness than 1# test steel. This disparity is attributed to the strengthening effect due to the precipitation of the second phase of Nb, which disperses fine carbides and carbonitrides throughout the steel, further augmenting its hardness.

Table 2 presents the mechanical properties data for five samples of test steel, comparing the cooling rates of 1# and 2# test steel. Figure 11 illustrates the stress–strain curves for these ten groups of test steel. Notably, the yield strength and tensile strength of the 2# test steel samples, across different cooling rates, exceed those of the 1# test steel. The simultaneous increase in yield strength and tensile strength of the test steel samples is correlated with the cooling rate. Specifically, at 3°C·s\(^{-1}\), the yield strength and tensile strength of 2# test steel reached 775.10 and 974.53 MPa, respectively. These values were 19.45 and 17.71% higher than those of 2# test steel at 0.3°C·s\(^{-1}\) and 34.24 and 34.16% higher than those of 1# test steel at 3°C·s\(^{-1}\). According to Figure 4, the precipitation of a second phase contributed to the refinement of austenite grains and hindered the movement of grain.

Figure 11: Stress–strain curves of the experimental steels: (a) 1# test steel, and (b) 2# test steel.

Figure 12: Relationship between different cooling rates and energy absorption.
Figure 13: Tensile fracture surface morphology of the experimental steels: (a)–(e) 1# test steel, and (f)–(j) 2# test steel.
boundaries and dislocations. Simultaneously, the higher cooling rate resulted in reduced ferrite grains, denser pearlite lamellae, and the generation of a significant number of dislocations, contributing substantially to the enhanced strength of the steel. These findings highlight that the inclusion of Nb and increased cooling rates substantially improve the mechanical properties of the steel.

Figure 12 is the energy absorption statistical diagram of ten groups of test steel from the beginning of tension to complete fracture, which is calculated by the stress-strain curve. It can be seen that when the cooling rate is $3{\degree}\text{C}\cdot\text{s}^{-1}$, the energy absorption value of Nb steel is $97.3 \text{ MJ}\cdot\text{m}^{-3}$, which is the highest among the ten groups of rebars. It shows good toughness. As the cooling rate increases from 0.3 to $3{\degree}\text{C}\cdot\text{s}^{-1}$, both groups of test steels maintain good energy absorption values, indicating that the yield strength and ultimate strength are greatly improved while maintaining good toughness [35]. The increase in cooling rate refines the ferrite grains and pearlite lamellae of the test steel, and the finer microstructure is an important factor to improve the toughness. At the same cooling rate, the energy absorption of Nb-containing steel is significantly higher than that of Nb-free steel. This is because the precipitation of $(\text{Nb, Ti, V})\text{C}$ at the grain boundary pins the grain boundary, hinders the movement of the grain boundary, refines the microstructure, increases the proportion of high-angle grain boundaries, and significantly improves the toughness of the rebar [35,36].

Figure 13 depicts the tensile fracture morphology of both test steels. It is evident that all samples exhibit ductile fracture, with numerous dimples distributed across the fracture surface. These dimples vary in size and depth, all exhibiting uniaxial stress tensile equiaxed dimples. Some individual dimples have a larger size with a diameter exceeding 20 $\mu\text{m}$. For samples (a), (b), (f), and (g), the number of fracture dimples is lower, and most of them are small and shallow, with an uneven distribution, indicating relatively poor plasticity. In contrast, samples (d), (e), (i), and (j) show more and deeper dimples with a more uniform distribution. This suggests that significant plastic deformation takes place during the stretching procedure, resulting in relatively high fracture toughness.

During the tensile process, the interlaced ferrite grains deform first. Due to the high strength of pearlite lamellae and the hindrance of dispersed dislocations within the matrix, plastic deformation of pearlite is not easily induced by applied stress. As the applied stress further intensifies and enters the necking stage, pearlite lamellae fracture, precipitated phase particles fall off, and tiny holes are formed. These micropores expand, connect, and gradually form dimples until sample fracture occurs. With an increase in cooling rate from 0.3 to $3{\degree}\text{C}\cdot\text{s}^{-1}$, a noticeable and substantial increase in both the number and size of dimples is observed at the fracture site of 2# test steel compared to 1# test steel. This trend indicates the positive effects of higher cooling rates and the introduction of Nb. These factors contribute to the refinement of ferrite grains, a reduction in pearlite lamellar spacing, and a more uniform distribution of ferrite grains. The strengthening effect of precipitates becomes evident, significantly enhancing both the strength and plasticity of the steel.

4 Conclusion

This study investigated the microstructure, precipitation patterns, and mechanical characteristics of high-strength rebars microalloyed with Nb, following various post-rolling cooling rates. The interplay between microstructural transformations, precipitation dynamics, and mechanical properties was systematically reviewed and analyzed, leading to the following key findings:

1. The microstructure and mechanical properties of high strength rebar can be controlled by adding Nb and adjusting the cooling rate after rolling. Under the condition of adding 0.023% Nb and $3{\degree}\text{C}\cdot\text{s}^{-1}$ cooling rate, high strength rebars with ferrite + pearlite, (Nb, Ti, V) C precipitated phase, and yield strength and tensile strength of 827.93 Mpa and 974.53 MPA can be obtained.

2. The synergistic effect of cooling rate and Nb refines the microstructure. The addition of Nb makes the precipitated phase of the test steel change from (V, Ti) C to (Nb, Ti, V) C, and the shape is ellipsoid. The microstructure of the examined steel consists of ferrite and pearlite. As the cooling rates increased from 0.3 to $3{\degree}\text{C}\cdot\text{s}^{-1}$, compared to Nb-free rebars, the Nb-inclusive rebars exhibited a reduction in average grain size from 19.18 to 8.55 $\mu\text{m}$. Simultaneously, the lamellar spacing of pearlite decreased from 0.1604 to 0.1109 $\mu\text{m}$. It is important to note that the proportion of low-angle grain boundaries increased from 6.75 to 10.88%, accompanied by a substantial rise in high-angle grain boundaries. The average size of the precipitated phase, decreasing from 56.87 to 37.97 nm. Additionally, the lattice fringe spacing also decreased, declining from 0.154 to 0.118 nm.

3. The refinement of microstructure and precipitated phase further improves the mechanical properties of rebars. With the increase in cooling rate from 0.3 to $3{\degree}\text{C}\cdot\text{s}^{-1}$, Nb-containing rebars exhibited a significant increase in hardness, rising from 219.21 to 317.77 HV compared to Nb-free rebars. The yield strength of Nb-containing rebars increased from 648.91 to 827.93 MPa, while the ultimate strength...
increased from 775.10 to 974.53 MPa. The energy absorption reaches 97.3 MJ·m⁻³ during the stretching process. The observed fracture in the tested steel displayed ductile characteristics, forming a dimple fracture morphology.

(4) In the production process of seismic rebar, a certain amount of Nb should be added to play its role of fine grain strengthening and precipitation strengthening. In the rolling process, a higher cooling rate should be adopted to refine the matrix structure and precipitated phase particles while controlling the structure of ferrite + pearlite, so as to obtain better mechanical properties.

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References


