(Nb, Ti)C Precipitation During Post-Rolling Coiling and Its Effect on Micromechanical Characteristics of Ferrite Phase (Postprint)

Authors: Xu Yang, Sun Mingxue, Zhou Yanlei, Liu Zhenyu

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Abstract

Microalloyed steel with combined additions of Nb and Ti was investigated using thermal simulation, microhardness testing, transmission electron microscopy, and nanoindentation techniques to observe and analyze the effects of cooling rate during continuous cooling and coiling processes on microstructure evolution and microhardness, as well as to study the precipitation behavior of (Nb, Ti)C during coiling and its influence on the micromechanical properties of the ferrite phase. The results indicate that increasing the cooling rate during both continuous cooling and coiling processes promotes the transformation from ferrite-pearlite microstructure to bainite microstructure in the Nb-Ti experimental steel, accompanied by ferrite grain refinement. Under continuous cooling conditions, the microhardness of the experimental steel gradually increases with increasing cooling rate; however, during the coiling process, a lower cooling rate promotes nucleation and growth of (Nb, Ti)C in ferrite, resulting in a large number of uniformly dispersed nano-precipitates that strengthen the matrix, and consequently the microhardness of the experimental steel exhibits a decreasing trend with increasing cooling rate during coiling. The nanoindentation hardness of the ferrite phase in the Nb-Ti experimental steel is 4.13 GPa with a Young' s modulus of 249.3 GPa, whereas that in conventional C-Si-Mn steel is 2.64 GPa with a Young's modulus of 237.4 GPa, and the contribution of nano-precipitates to the nanoindentation hardness of the ferrite phase reaches 1.49 GPa.

Full Text

Preamble

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Precipitation Behavior of (Nb, Ti)C in Coiling Process and Its Effect on Micro-Mechanical Characteristics of Ferrite

XU Yang, SUN Mingxue, ZHOU Yanlei, LIU Zhenyu
(State Key Laboratory of Rolling and Automation, Northeastern University, Shenyang 110819)

Abstract
High-strength micro-alloyed steel has been widely used in automobiles and machinery because of its remarkable strength and formability, which are attributed to nano-precipitates and microstructural refinement. Since nano-precipitates nucleate primarily during the austenite-to-ferrite transformation or in supersaturated ferrite after transformation, investigating precipitation behavior during the coiling process is crucial. Nanoindentation technology provides an opportunity to study the specific influence of nano-precipitates on the micro-properties of ferrite. The effects of cooling rate during continuous cooling and coiling processes on microstructural evolution and micro-hardness of Nb-Ti micro-alloyed steel were studied using a thermal-mechanical simulator, micro-hardness tester, TEM, and nanoindentation instrument. The precipitation behavior of (Nb,Ti)C during coiling and its effect on the nano-hardness of ferrite were investigated. Experimental results indicated that increasing the cooling rate in both continuous cooling and coiling processes promoted the microstructural transition from ferrite and pearlite to bainite. The micro-hardness of the tested steel increased with cooling rate during continuous cooling, but decreased with cooling rate during coiling due to the formation of numerous dispersive nano-precipitates in ferrite that strengthened the matrix. Smaller cooling rates promoted the volume fraction of (Nb, Ti)C particles in ferrite because sufficient time was available for nucleation and growth of (Nb, Ti)C precipitates. When the cooling rate during coiling was 0.1 ℃/s, precipitates were dispersively distributed in the ferrite matrix with diameters less than 10 nm. The nanohardness and Young' s modulus of ferrite were 4.13 GPa and 249.3 GPa for Nb-Ti micro-alloyed steel, and 2.64 GPa and 237.4 GPa for C-Si-Mn steel. The contribution of nano-precipitates to the nano-hardness of ferrite reached 1.49 GPa.

Keywords: nano-precipitate, cooling rate, nanoindentation, nanohardness, Young' s modulus

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Introduction

The frequent occurrence of "haze" weather in recent years has highlighted its threats to society and human health. Since vehicle exhaust accounts for over 20%

of haze composition, reducing automotive emissions and energy consumption has become critical. Using high-strength, high-formability steel sheets for automotive structural components to reduce vehicle weight, fuel consumption, and exhaust emissions has become a primary approach for automakers. Research has shown that adding trace amounts of Nb, V, Ti, and Mo to conventional C-Si-Mn steel not only effectively refines austenite and ferrite grains but also forms numerous nanoscale carbonitride precipitates during or after transformation, significantly improving steel strength and stability. Therefore, obtaining fine, uniformly distributed precipitates with high volume fraction has become an important research direction in steel materials.

Funakawa et al. developed high-strength automotive steel with tensile strength exceeding 780 MPa based on Ti-Mo micro-alloyed steel, utilizing nanoscale precipitates of Ti and Mo while maintaining good hole expansion and thermal stability. Yen et al. and Okamoto et al. observed regular interphase precipitation in Ti, Ti-Mo, and Nb micro-alloyed steels using transmission electron microscopy. They found that lower isothermal temperatures resulted in smaller inter-precipitate spacing and particle size, leading to higher micro-hardness. Using the Orowan dislocation bypassing theory, they calculated that strengthening from these nanoscale interphase precipitates could exceed 400 MPa. Mao et al., Lu and Wang, and Duan et al. found that controlling rolling and cooling processes could obtain dispersively distributed nano-precipitates in the matrix. They concluded that these strength-contributing nanoscale precipitates formed primarily in ferrite, as precipitates formed in austenite were larger, provided weaker dislocation pinning, and contributed less to strength enhancement. Therefore, controlling the process to obtain nanoscale precipitates in ferrite is particularly important.

The fastest precipitation temperature for Nb and Ti carbonitrides in ferrite is between 600-700 ℃. In industrial production of micro-alloyed high-strength steel, high cooling rates are typically used with relatively high coiling temperatures, making transformation and precipitation occur mainly during the coiling process. Thus, studying the precipitation behavior of micro-alloyed carbonitrides during coiling is necessary. Based on the arrangement of precipitates in ferrite, precipitation can be classified as regular interphase precipitation or randomly distributed precipitation. Both types can significantly increase steel strength if precipitate size is controlled below 20 nm. However, few studies have used nanoindentation technology to directly investigate the effect of micro-alloying elements and their carbonitride precipitates on the nanomechanical properties of ferrite. This work systematically studied the effects of different cooling rates during coiling on microstructure and nano-(Nb, Ti)C precipitation in Nb-Ti experimental steel using a thermal simulator, micro-hardness testing, and transmission electron microscopy. The effect of (Nb, Ti)C precipitation on the micromechanical properties of ferrite was analyzed through nanoindentation.

Experimental

The experimental steel had a main chemical composition (mass fraction, %) of: Fe-0.08C-0.21Si-1.76Mn-0.06Nb-0.11Ti-0.0045N, hereinafter referred to as Nb-Ti experimental steel. The steel was melted and cast using a ZGJL0.2-200-2.5H vacuum furnace, then forged into billets measuring 80 mm wide, 80 mm high, and 120 mm long for hot rolling. The forged billets were heated to 1200 ℃ for 2 h, then hot-rolled to 12 mm thickness on an F450$×$450 rolling mill. Samples measuring 8 mm in diameter and 15 mm in length were machined for thermal simulation experiments, which were conducted on an MMS-300 thermal-mechanical simulator. To ensure that transformation and precipitation occurred during coiling, the continuous cooling transformation (CCT) curve of the experimental steel was first determined. The coiling temperature was set based on the transformation start temperature, and different cooling rates during coiling were controlled to study the precipitation behavior of micro-alloyed carbonitrides. The specific process routes are shown in [FIGURE:1].

Samples after thermal simulation were sectioned radially at the thermocouple location using wire cutting. After mechanical grinding and polishing, they were etched with 4% nital solution (volume fraction) and observed under a LEICA-DMIRM optical microscope (OM) to examine the microstructure under different processes. The micro-hardness of the polished and etched samples was measured using a Vickers hardness tester with a 50 g load and 10 s dwell time, with 20 measurements averaged per sample. Thermal simulation samples were cut into 0.5 mm thick slices using wire cutting, then mechanically ground and twin-jet electropolished to prepare 3 mm diameter TEM specimens for observation in a Tecnai G2 F20 field-emission transmission electron microscope. Micro-mechanical property testing of the ferrite phase was performed on a TRIBOINDENTER in-situ nanomechanical testing system. Sample preparation involved mechanical grinding followed by electropolishing in a solution with volume ratio 13:2:1 of alcohol, perchloric acid, and water to remove surface residual stress. Nanoindentation experiments used a 5-5-5 loading mode (5 s loading, 5 s holding, 5 s unloading) with a load of 2000 μN. Nanoindentation tests were conducted at different locations on each sample, and the results were statistically averaged.

Results and Discussion

2.1 Microstructural Evolution and Micro-Hardness Under Continuous Cooling Conditions

[FIGURE:2]a-d show OM images of the experimental steel under different cooling rates during continuous cooling. At a cooling rate of 0.5 ℃/s, the matrix consisted primarily of ferrite with a small amount of pearlite. When the cooling rate increased to 1 ℃/s, bainite began to appear in the matrix. With increasing cooling rate, ferrite grain size gradually decreased while the volume fraction of bainite increased. The experimental steel underwent two compression deformations at 1050 ℃ and 840 ℃, causing austenite recrystallization and deformation that increased austenite grain boundary area. The deformation also increased nucleation sites such as deformation bands in the matrix, enhancing ferrite nucleation rate and refining the ferrite structure. The austenite-to-ferrite transformation is a diffusion-type transformation, and ferrite growth is mainly controlled by carbon atom diffusion. As cooling rate increased, austenite undercooling increased, suppressing carbon atom diffusion and preventing long-range diffusion. Under these conditions, austenite transformed to bainite through a shear mechanism. When the cooling rate exceeded 5 ℃/s, the experimental steel matrix consisted mainly of bainite.

Based on metallographic observations and dilatometric curves measured at different cooling rates, the CCT curve of the experimental steel was constructed (austenitizing temperature 1250 ℃), and the matrix micro-hardness at different cooling rates was measured, as shown in [FIGURE:3]a and b, respectively. Increasing cooling rate lowered the transformation start temperatures for both ferrite and bainite. At cooling rates greater than 10 ℃/s, the start temperature of undercooled austenite transformation was already below 600 ℃. As cooling rate increased, the micro-hardness of the experimental steel matrix gradually increased due to refined ferrite grain size, bainite structure refinement, and increased volume fraction of bainite.

2.2 Effect of Cooling Rate During Coiling on Microstructure and Micro-Hardness

In hot rolling production, nano-precipitates that contribute significantly to yield strength nucleate and grow primarily during the austenite-to-ferrite transformation or in supersaturated ferrite after transformation. To accurately investigate the precipitation of micro-alloyed carbonitrides during coiling, the undercooled austenite transformation must be completed entirely during the coiling process, and the coiling temperature must be higher than the actual transformation start temperature. Based on the CCT curve, a coiling temperature of 640 ℃ was selected for the experiments, followed by cooling to room temperature at different rates. [FIGURE:4] shows typical microstructures at different cooling rates during coiling and the variation curve of micro-hardness with cooling rate.

At cooling rates of 0.05-0.2 ℃/s, the microstructure consisted mainly of ferrite with small amounts of pearlite and bainite. When the cooling rate exceeded 0.5 ℃/s, the microstructure was primarily bainite, which became progressively finer with increasing cooling rate. As seen in [FIGURE:4]a-c, increasing cooling rate during coiling had some refining effect on ferrite grains, and using smaller cooling rates after coiling could still ensure a fine ferrite structure. [FIGURE:4]f shows the micro-hardness at different cooling rates during coiling. The micro-hardness exhibited a decreasing trend with increasing cooling rate during coiling. When the cooling rate increased from 0.5 ℃/s to 1 ℃/s, the matrix micro-hardness dropped sharply, then increased slightly with further cooling rate increases.

Figure 5

Figure 1: Figure 5

2.3 Precipitation of (Nb, Ti)C During Coiling

shows typical precipitate morphologies in the matrix at cooling rates of 0.1, 0.5, 1, and 2 ℃/s during coiling. At a cooling rate of 0.1 ℃/s, numerous uniformly distributed nano-precipitates smaller than 10 nm existed in the ferrite matrix. At a cooling rate of 2 ℃/s, although nano-precipitates as small as 5 nm were present, their number density in the same area was significantly lower than at 0.1 ℃/s. With increasing cooling rate, the number of nano-precipitates in the matrix gradually decreased, and the volume fraction of precipitates decreased.

Generally, precipitates in micro-alloyed steel are mainly M(C, N) phases. In the experimental steel used in this work, 0.11% Ti was added while the N content was 0.0045%. According to the solubility product formula for TiN, the dissolved N content in the matrix at 1250 ℃ was below 0.0001%, indicating that virtually no N remained in the matrix. N combined with Ti at high temperatures to form TiN particles larger than 200 nm. Therefore, the nano-precipitates in the Nb-Ti experimental steel matrix were primarily (Nb, Ti)C.

Second-phase precipitates formed in ferrite are smaller and more uniformly distributed than those formed in austenite, providing higher precipitation strengthening. According to classical nucleation theory for micro-alloyed carbonitrides, the nucleation rate per unit volume is mainly affected by nucleation sites, diffusion coefficients, interfacial energy, and nucleation driving force. In the processing route of this work, the heating temperature, deformation temperature, and deformation amount before coiling were identical. Therefore, considering the precipitation kinetics of micro-alloyed carbonitrides, the nucleation and growth of (Nb, Ti)C were primarily determined by the diffusion of Nb, Ti, and C, requiring sufficient time for precipitation. The decisive factor for precipitation in these experiments was the variation in nucleation driving force caused by different cooling rates during coiling. The driving force for (Nb, Ti)C nucleation at phase boundaries, grain boundaries, dislocations, and vacancies is a function of temperature and the contents of Nb, Ti, and C. Higher cooling rates increase undercooling, increasing the supersaturation of micro-alloying elements Nb, Ti, and C, thereby increasing the nucleation driving force and nucleation rate for (Nb, Ti)C. However, precipitation of micro-alloyed carbonitrides is a typical diffusion-type solid-state transformation, and precipitate nucleation and growth are affected by the diffusion rates of Nb, Ti, and C. If the cooling rate is too fast, the diffusion rate of precipitate-forming elements in the matrix decreases significantly, potentially suppressing diffusion and maintaining them in solid solution. Under such conditions, precipitates cannot nucleate or fail to reach critical nucleus size before being suppressed.

As shown in

Figure 5

Figure 2: Figure 5

Figure 5

Figure 3: Figure 5

, at a cooling rate of 2 ℃/s, precipitate size and distribution in the matrix were non-uniform. Although small precipitates existed, their low number density and large inter-particle spacing resulted in weak strengthening effects, reflected by the decreasing micro-hardness with increasing cooling rate during coiling.

In actual production, billets undergo substantial compression deformation in the recrystallization and non-recrystallization zones (in this work, samples were deformed at 1050 ℃ and 840 ℃ with true strain of 0.3). The high deformation temperature creates numerous vacancies, deformation bands, subgrain boundaries, and dislocations in the austenite. These defect sites have high energy, reducing the nucleation work required and facilitating nucleation of micro-alloyed carbonitrides. Precipitation tends to occur along prior austenite grain boundaries and internal deformation bands, increasing intergranular strengthening but potentially becoming crack initiation sites and propagation paths, which is detrimental to comprehensive mechanical properties. Applying rapid cooling after rolling suppresses nucleation of micro-alloyed carbonitrides at austenite grain boundaries, maintaining more micro-alloying elements in solid solution in undercooled austenite while preserving numerous dislocations, deformation bands, and vacancies formed during deformation. These sites become the primary nucleation locations for (Nb, Ti)C during low-temperature transformation or after transformation, enabling more uniform and dispersive precipitation during coiling.

Furthermore, combining [FIGURE:2]-

, the cooling rate at which micro-hardness drops sharply can be considered the critical cooling rate for substantial (Nb, Ti)C precipitation during coiling. Metallographic observation also reveals that this cooling rate represents the critical cooling rate for ferrite transformation. At a cooling rate of 0.5 ℃/s, the matrix contained a small amount of ferrite, while at cooling rates greater than 1 ℃/s, no ferrite phase existed in the experimental steel, and the matrix consisted entirely of bainite. Therefore, for the experimental steel investigated in this work, bainite transformation is less favorable than ferrite transformation for nucleation and growth of (Nb, Ti)C nano-precipitates during coiling, which may be related to the bainite transformation mechanism. Studies suggest that bainite transformation occurs through a shear mechanism and is a diffusionless transformation, during which carbon atom diffusion capacity decreases and cannot proceed over long distances, making diffusion insufficient. Under identical rolling processes, cooling rates, and coiling temperatures, the cooling rate during

Figure 6

Figure 4: Figure 6

coiling determines the diffusion rates of C and micro-alloying elements. When the cooling rate exceeds 0.5 ℃/s, austenite transforms mainly to bainite, carbon forms carbides between ferrite laths, and the diffusion capacity of Nb and Ti decreases, preventing combination with C to form substantial (Nb, Ti)C.

2.4 Nanoindentation Experiments

Compared with micro-hardness and macro-hardness, nanoindentation uses a smaller indenter that can measure hardness within individual grains and continuously determine the variation of nanohardness with indentation depth. Therefore, nanoindentation can eliminate the influence of grain boundaries on hardness, directly reflecting the effect of intra-phase microstructure on hardness and other mechanical properties. To investigate the effects of Nb and Ti micro-alloying elements and (Nb, Ti)C precipitation on the micro-mechanical properties of ferrite, comparative nanoindentation experiments were conducted on Nb-Ti experimental steel and conventional C-Si-Mn steel without micro-alloying elements (Fe-0.07C-0.22Si-1.77Mn), with more than 20 indentations performed for each steel.

shows the nanoindentation morphology and load-depth and nanohardness-depth curves for both steels. The nanohardness of ferrite phase in Nb-Ti experimental steel was measured at a cooling rate of 0.1 ℃/s. The nanomechanical properties of ferrite phase, including nanohardness and Young's modulus, can be calculated using the following equations:

$$H = \frac{P}{A}$$

$$A = 24.5h_c^2 + 793h_c^{1/16} + 4238h_c^{1/32} + 332h_c^{1/64} + 0.059h_c^{1/128} + 36.9h_c + 8.68h_c^{1/2} + 35.4$$

$$E_r = \frac{\sqrt{\pi}}{2\beta}\frac{S}{\sqrt{A}}$$

$$\frac{1}{E_r} = \frac{1-v^2}{E} + \frac{1-v_i^2}{E_i}$$

where H is nanohardness, P is applied load, A is contact area, h_c is contact depth, h is indentation depth, β is a constant related to the indenter (0.75 for a Berkovich indenter), S is the slope at the top of the unloading curve, E_r is

Figure 6

Figure 5: Figure 6

Figure 6

Figure 6: Figure 6

reduced modulus, E and E_i are Young' s moduli of the sample and indenter respectively, and v and v_i are Poisson' s ratios of the sample and indenter respectively. For a diamond Berkovich indenter, E_i = 1114 GPa and v_i = 0.07.

As shown in

b, under the same loading force, the indentation depth in conventional C-Si-Mn steel (170 nm) was 61 nm deeper than that in micro-alloyed steel (109 nm), indicating that ferrite in C-Si-Mn steel is softer than ferrite in Nb-Ti experimental steel. The calculated nanohardness values of ferrite phase were 2.64 GPa and 4.13 GPa, respectively, representing a 1.49 GPa increase in nanohardness for the Nb-Ti experimental steel. TEM observation at 0.1 ℃/s cooling rate revealed numerous nano-precipitates in the ferrite matrix of Nb-Ti experimental steel, which were responsible for the increased nanohardness. The measured Young' s modulus of C-Si-Mn steel ranged from 220.5 to 261.1 GPa, with an average of 237.4 GPa, while that of Nb-Ti experimental steel ranged from 239.2 to 288.5 GPa, with an average of 249.3 GPa—a difference of 11.9 GPa.

From the load-depth curves (

b) and nanohardness-depth curves (
c) for both steels, the loading force corresponding to the same indentation depth was greater for Nb-Ti experimental steel. After indentation depth exceeded 40 nm, the nanohardness of ferrite in C-Si-Mn steel remained essentially constant, while that of ferrite containing nano-precipitates continued to increase, stabilizing after indentation depth exceeded 80 nm. This indicates that during indenter penetration, dislocation movement around the indenter was effectively inhibited by nano-precipitates in ferrite. As the indenter penetrated deeper, dislocation density around it increased, resulting in a higher strain hardening exponent for Nb-Ti experimental steel compared to C-Si-Mn steel.

schematically illustrates the interaction between dislocation movement and precipitate particles during nanoindentation. The interaction mechanism between

Figure 6

Figure 7: Figure 6

Figure 7

Figure 8: Figure 7

precipitates and dislocation movement in the experimental steel is primarily the Orowan bypassing mechanism. When dislocations move along slip planes, they are hindered by precipitate particles. When the stress on dislocations exceeds the critical stress required to bypass the precipitates, dislocations bypass the particles and leave behind a dislocation loop. The left-behind dislocation loops also hinder dislocation movement. This hindering effect of precipitates and the continuously generated dislocation loops on dislocation movement leads to increased nanohardness and work hardening rate of ferrite. Uniformly dispersed nano-precipitates in ferrite can not only significantly increase steel strength but also improve plastic uniformity.

Conclusions

1. Under continuous cooling conditions, increasing cooling rate suppresses ferrite transformation and promotes bainite transformation in Nb-Ti experimental steel, resulting in gradually increasing matrix micro-hardness with cooling rate.

2. After coiling at 640 °C, increasing cooling rate caused microstructural transition from ferrite + pearlite to bainite, and the matrix micro-hardness exhibited a decreasing trend. Using smaller cooling rates during coiling could still produce fine ferrite structures while promoting (Nb, Ti)C precipitation in ferrite.

3. (Nb, Ti)C nano-precipitates can significantly increase matrix strength. The nanohardness of ferrite in Nb-Ti micro-alloyed steel was 4.13 GPa with a Young’ s modulus of 249.3 GPa, compared to 2.64 GPa and 237.4 GPa for conventional C-Si-Mn steel. The contribution of nano-precipitates to ferrite nanohardness reached 1.49 GPa, Young’ s modulus increased by 11.9 GPa, and nano-precipitates enhanced the strain hardening exponent of ferrite.

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Figure 10

Figure 9: Figure 10

Figure 14

Figure 10: Figure 14

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Figures

Figure 15

Figure 11: Figure 15