Effect of Nb on Continuous Cooling Transformation Behavior and Microstructure-Properties of Ti-Mo Microalloyed Steel Postprint

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Abstract

Abstract: The continuous cooling transformation behavior of Ti-Mo and Ti-Mo-Nb low-carbon microalloyed steels was investigated using a thermal simulator, scanning electron microscope (SEM), high-resolution transmission electron microscope (HRTEM), and energy dispersive spectrometer (EDS), and the effect of Nb on the microstructure and properties of Ti-Mo microalloyed steel was discussed. The results show that Nb element can increase the Ac1 and Ac3 temperatures of steel, decrease the decomposition temperature of austenite during cooling, shrink the ferrite-pearlite phase region, and shift the bainite phase region to the lower left. In addition, the addition of Nb can refine the microstructure of Ti-Mo-Nb microalloyed steel and increase its hardness. HRTEM analysis of samples at a cooling rate of 50 °C/s revealed that both Ti-Mo and Ti-Mo-Nb microalloyed steels contain a small amount of strain-induced precipitated carbides, namely (Ti, Mo)C and (Ti, Nb, Mo)C particles, which are randomly distributed. Both precipitates have a NaCl-type structure, with lattice constants of 0.432 and 0.436 nm, and average particle sizes of 12.11 nm and 8.69 nm, respectively. The higher volume fraction and smaller size of precipitates in Ti-Mo-Nb microalloyed steel are the main reasons for its microstructure refinement and hardness improvement.

Full Text

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Abstract

In recent years, with the rapid development of the automotive industry, increasing attention has been focused on developing high-strength automobile steels with excellent formability. Microalloying elements such as Nb, Ti, and Mo, which facilitate grain refinement and precipitation hardening, have been added to steels to achieve high strength and good formability, leading to the development of Ti-Mo and Ti-Mo-Nb microalloyed high-strength ferritic steels. In this work, the continuous cooling transformation (CCT) curves of Ti-Mo and Ti-Mo-Nb steels were obtained, and the effect of Nb on the microstructure and mechanical properties of Ti-Mo low-carbon microalloyed steel was investigated using SEM, HRTEM, and EDS. The results showed that Nb could raise the Ac_1 and Ac_3 temperatures and restrain ferrite-pearlite and bainite transformation. Moreover, Nb could refine the microstructure and harden the steel matrix, which was attributed to the strain-induced precipitation of nano-sized (Ti, Nb, Mo)C particles identified by HRTEM and EDS. It was also found that strain-induced precipitation of (Ti, Mo)C existed in the Ti-Mo steel. Both (Ti, Mo)C and (Ti, Nb, Mo)C particles exhibited a NaCl-type structure. The lattice constants and average particle sizes of (Ti, Mo)C and (Ti, Nb, Mo)C were 0.432 nm and 0.436 nm, and 12.11 nm and 8.69 nm, respectively.

KEY WORDS Ti microalloyed steel, CCT curve, Nb, nano-sized precipitation, hardness

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Introduction

The development of high-performance automotive steels to meet the demands of vehicle lightweighting and safety performance has become a research hotspot in the field of steel materials. Ultra-high strength ferritic steel offers advantages such as low cost, good ductility and toughness, excellent formability, and weldability, representing an important development direction for high-performance automotive steels. However, currently produced ferritic steels in China have relatively low strength, making it difficult to meet the development needs of

the automotive industry. Typically, one or more microalloying elements such as Ti, Nb, Mo, and V must be added to the steel to achieve strength enhancement through solid solution strengthening, grain refinement strengthening, and precipitation strengthening.

In recent years, Funakawa et al. developed an ultra-high strength ferritic automotive steel with tensile strength exceeding 780 MPa using Ti-Mo microalloying technology, achieving a precipitation strengthening increment of 300 MPa. Building on this work, Zhang et al. systematically studied low-carbon Nb-Mo steel and found that compared with Nb steel, Nb-Mo steel exhibited a finer microstructure with a higher content of nanometer-sized MC-type precipitates (Nb, Mo)C (less than 10 nm), which provided significant precipitation strengthening. Jang investigated interphase precipitation in Ti-Nb and Ti-Nb-Mo microalloyed steels at 700°C and carbide coarsening after aging treatments at different temperatures and times, finding that Mo could significantly reduce carbide size and strongly inhibit coarsening during heat treatment. Bu et al. studied the precipitation behavior of nanometer-sized carbides in Ti-Nb-Mo composite microalloyed steel during tempering, observing that the yield strength of the experimental steel significantly increased after tempering at 650°C for 0.5 h following hot rolling and air cooling. TEM observation revealed numerous fine and uniformly distributed nanometer-sized carbide particles formed in the ferrite matrix and on dislocations after tempering. These studies demonstrate that the combined addition of Ti, Nb, and Mo microalloying elements, through their interactions, can produce a large number of fine and stable second-phase particles in steel, which can substantially increase the strength of ferritic automotive steels through grain refinement and precipitation strengthening. However, current research has primarily focused on the precipitation behavior and strengthening mechanisms of microalloyed second phases.

Based on this background, the present work investigates Ti-Mo and Ti-Nb-Mo low-carbon microalloyed steels, systematically studying the effect of Nb on the phase transformation behavior and microstructure-properties of microalloyed steels using a thermal simulator, scanning electron microscopy (SEM), and high-resolution transmission electron microscopy (HRTEM), thereby establishing a theoretical foundation for the development of Ti-Mo-Nb ultra-high strength ferritic automotive steels.

Experimental Methods

The microalloyed steels used in this study were melted in a 50 kg vacuum induction furnace. After removing the shrinkage cavity, the ingots were forged into cast billets measuring 250 mm × 100 mm × 60 mm. The chemical compositions are shown in Table 1 . The billets were heated to 1250°C, held for 2 h, and then cooled before being machined into thermal simulation specimens with a diameter of 8 mm and length of 12 mm. The dynamic continuous cooling

transformation (CCT) curves were determined using a THERMECMASTER-Z thermal simulator, with the specific process route shown in Figure 1 [Figure 1: see original paper]. The specimens were first heated to 1250°C at a rate of 20°C/s and held for 3 min, then cooled to 1050°C at 10°C/s. After holding for 2 s, they were deformed 50% at a strain rate of 5 s^{-1}, cooled to 900°C at 10°C/s, held isothermally for 2 s, and then deformed 30% at a strain rate of 5 s^{-1}. After deformation, the samples were cooled to room temperature at cooling rates of 0.5, 1, 5, 10, 20, 30, and 50°C/s, with thermal expansion curves recorded under different process conditions.

The thermal simulation specimens under different processing conditions were sectioned along the axis using wire-cut electrical discharge machining to prepare metallographic samples. After grinding and polishing, the samples were etched in 4% (volume fraction) nitric acid alcohol solution for 15 s. Microstructural observation was performed using an Axiplan2 Imaging optical microscope (OM) and a Nova 400 Nano scanning electron microscope (SEM). The microhardness was measured using an HV-1000B Vickers hardness tester with a load of 200 g and loading time of 10 s.

To further observe the precipitates in the specimens, carbon extraction replicas were prepared for transmission electron microscopy. The precipitate morphologies were examined using a JEM-2100 field emission high-resolution transmission electron microscope (HRTEM), and the precipitate compositions were characterized using the attached energy dispersive spectrometer (EDS). The particle sizes were statistically analyzed using the intercept method. The carbon extraction replica preparation process was as follows: after deep etching of the polished sample, carbon was deposited on the surface. The carbon-coated surface was then scribed into 3 mm × 3 mm squares using a scalpel, and the carbon film was extracted using 4% (volume fraction) nitric acid alcohol solution. The extracted carbon film was cleaned in absolute alcohol, transferred to deionized water, and then collected on a copper grid and allowed to dry naturally on filter paper.

2.1 Effect of Nb on Continuous Cooling Transformation Behavior

Figure 2 [Figure 2: see original paper] shows the temperature-expansion curves for the Ti-Mo and Ti-Mo-Nb steels. Using the tangent method, the ferrite-to-austenite transformation start temperature (Ac_1) and finish temperature (Ac_3) were determined to be 804°C and 906°C for the Ti-Mo steel, and 845°C and 941°C for the Ti-Mo-Nb steel, respectively.

Nb is a strong carbide-forming element that exists in steel primarily in solid solution and precipitated forms. During heating, the strong affinity between solid-solution Nb and C atoms reduces the diffusion rate of C atoms in the steel, slowing the phase transformation process and thereby increasing the transformation temperatures. On the other hand, Zhang' s research indicates that when Nb exists in precipitated form, it combines with C to form carbides, reducing

the solid-solution Nb and C contents in austenite and resulting in increased Ac_1 and Ac_3 temperatures in Nb-microalloyed steels, which is consistent with the present results.

Figure 3 [Figure 3: see original paper] shows SEM images of the Ti-Mo steel after cooling at different rates. At a cooling rate of 0.5°C/s, the microstructure consisted of polygonal ferrite with a small amount of pearlite, with an average ferrite grain size of 5.0 m (Figure 3a). When the cooling rate increased to 1°C/s, a small amount of granular bainite appeared, and the matrix microstructure became a mixture of ferrite, pearlite, and granular bainite, with pearlite content significantly reduced (Figure 3b). With increasing cooling rate, the bainite content continuously increased while the ferrite content gradually decreased (Figures 3c–g). At a cooling rate of 10°C/s, the matrix microstructure was granular bainite with a small amount of polygonal ferrite (Figure 3d). When the cooling rate further increased to 20°C/s, the polygonal ferrite completely disappeared, and the matrix microstructure consisted of granular bainite and lath bainite (Figure 3e). As the cooling rate continued to increase to 50°C/s, the volume fraction of lath bainite in the steel continuously increased (Figures 3f and g).

Figure 4 [Figure 4: see original paper] shows SEM images of the Ti-Mo-Nb steel after cooling at different rates. Comparing the microstructures under the same cooling conditions reveals that at 0.5°C/s, the Ti-Mo-Nb steel also exhibited a polygonal ferrite + pearlite microstructure, but with a significantly smaller average ferrite grain size of only 3.7 m (Figure 4a). Related studies have shown that during soaking, cooling, and deformation of Ti-Mo-Nb steel, Nb combines with C and N to form Nb(C,N) particles, reducing the solid-solution C content in austenite, decreasing austenite stability, and increasing the driving force for ferrite transformation. Additionally, the precipitated Nb(C,N) particles can pin grain boundaries, inhibiting austenite grain growth and recrystallization, thereby refining the austenite grain size and increasing the ferrite nucleation rate to refine ferrite grains. At a cooling rate of 1°C/s, the Ti-Mo-Nb steel microstructure consisted of ferrite with significantly reduced pearlite, but no granular bainite was observed (Figure 4b). This is primarily because the reduced solid-solution C content and grain refinement favor high-temperature diffusional transformation, delaying the bainite transformation. At a cooling rate of 5°C/s, the Ti-Mo-Nb steel microstructure was ferrite + granular bainite, similar to that in the Ti-Mo steel. At 10°C/s, the Ti-Mo-Nb steel microstructure was entirely bainitic, while the Ti-Mo steel still contained a small amount of ferrite (Figure 4d). The main reason is that solid-solution Nb, especially that segregated at grain boundaries, interacts with C to inhibit nucleation of ferrite-pearlite, thereby delaying the ferrite-pearlite transformation. With further increase in cooling rate, the amount of lath bainite in the steel continuously increased with cooling rate (Figures 4e–g), consistent with the results for the Ti-Mo steel.

The dynamic CCT curves for the Ti-Mo and Ti-Mo-Nb steels were obtained using the thermal simulator by determining the phase transformation points from

the temperature-expansion curves using the tangent method, combined with metallographic observation and hardness measurements, as shown in Figure 5 [Figure 5: see original paper]. Within the cooling rate range of 0.5–50°C/s, the dynamic CCT curves of both steels consist of two parts: the ferrite-pearlite transformation region and the bainite transformation region. With increasing cooling rate, the ferrite-pearlite transformation region gradually shrinks while the bainite transformation region expands. The austenite transformation temperatures of both steels generally decrease with increasing cooling rate. Comparison of the two dynamic CCT curves reveals that the decomposition temperature of undercooled austenite in the Ti-Mo-Nb steel is lower, particularly the critical cooling rate for bainite formation is increased and the bainite transformation start temperature is decreased. At a cooling rate of 5°C/s, the austenite-to-ferrite transformation temperature in the Ti-Mo-Nb steel is approximately 677.9°C, about 50°C lower than that in the Ti-Mo steel. At a cooling rate of 30°C/s, the bainite transformation temperature in the Ti-Mo-Nb steel is 30°C lower than that in the Ti-Mo steel. The primary reasons are as follows: thermodynamically, Nb as a ferrite-forming element increases the austenite-ferrite equilibrium transformation temperature (Ae_3); however, kinetically, due to the large lattice mismatch between Nb and Fe, solid-solution Nb in steel tends to segregate to grain boundaries, reducing grain boundary energy, and Nb decreases the activity of C atoms in steel, inhibiting C diffusion and thereby suppressing ferrite nucleation. Additionally, Nb atoms segregated at phase interfaces exert a strong solute drag effect on phase boundary migration, delaying the austenite-to-ferrite transformation and lowering the ferrite transformation temperature. However, the solute drag theory is based on diffusional transformation and is not applicable to bainite transformation. Fossaert et al. pointed out that the inhibiting effect of Nb on bainite transformation is mainly manifested by the stabilization of austenite grain boundaries through Nb segregation, which suppresses nucleation of bainitic ferrite at grain boundaries. On the other hand, Yuan et al. demonstrated that when Nb exists as precipitates, smaller precipitates along grain boundaries replace high-energy austenite grain boundaries with coherent or semi-coherent precipitate/austenite interfaces, inhibiting nucleation of ferrite and bainite and thereby lowering their transformation temperatures.

2.2 Effect of Nb on Microstructure and Properties

Figure 6 [Figure 6: see original paper] shows the hardness variation curves for the two steels at different cooling rates. The hardness of both steels exhibits a trend of rapid initial increase, followed by a plateau, and then a slow decrease. At a cooling rate of 0.5°C/s, since both Ti-Mo and Ti-Mo-Nb steels consisted of ferrite and pearlite, their hardness values were similar at 189.1 HV and 188.0 HV, respectively. At 1°C/s, the microhardness of the Ti-Mo steel was significantly higher than that of the Ti-Mo-Nb steel, mainly because the increased cooling rate induced bainite transformation in the Ti-Mo steel, and bainite has higher

hardness than ferrite. At 5°C/s, both steels primarily consisted of ferrite + bainite, and the microhardness increased linearly with similar values. At 10°C/s, the hardness increase trend slowed, but the Ti-Mo-Nb steel showed significantly higher hardness than the Ti-Mo steel (271.4 HV vs. 265.0 HV). As discussed previously, this is primarily because the Ti-Mo steel still contained a small amount of ferrite, while the Ti-Mo-Nb steel was fully bainitic. At cooling rates of 20, 30, and 50°C/s, both steels were fully bainitic. The average microhardness values for the Ti-Mo steel were 268.0, 253.5, and 245.1 HV, respectively, while those for the Ti-Mo-Nb steel were 269.3, 268.6, and 254.1 HV, respectively. Comparison reveals that the microhardness of both steels decreased slightly at high cooling rates, but the Ti-Mo-Nb steel maintained higher hardness than the Ti-Mo steel at the same cooling rate.

Studies have shown that the hardness of steel materials is closely related to the volume fraction and size of second-phase particles precipitated in the matrix. Higher volume fraction and smaller size of microalloyed second-phase particles result in higher hardness. To determine the precipitation amount of microalloyed second-phase particles in different steels, this work calculated the solid-solution content of relevant elements in the steel using solubility product formulas and ideal stoichiometric ratios. The solubility product formulas for TiC, NbC, and MoC in austenite are as follows:

$$\lg\{[\mathrm{Ti}][\mathrm{C}]\}_{\gamma} = 2.75 - \frac{7000}{T} \tag{1}$$

$$\lg\{[\mathrm{Nb}][\mathrm{C}]\}_{\gamma} = 2.96 - \frac{7510}{T} \tag{2}$$

$$\lg\{[\mathrm{Mo}][\mathrm{C}]\}_{\gamma} = 1.29 - \frac{523}{T} \tag{3}$$

where [M] (M = Ti, Nb, Mo, C) represents the solid-solution content of element M in austenite; γ denotes austenite; and T is the solution temperature in K.

The volume fraction of precipitates in different steels was then calculated using the following formula:

$$v_f = \frac{([\mathrm{M}] - [\mathrm{M}]_{\gamma}) \times 100}{\rho_{\mathrm{Fe}}/\rho_{\mathrm{MC}}}$$

where M represents the added amount (mass fraction) of each element (M = Ti, Nb, Mo, C) in the steel; M - [M] is the precipitated amount of the second phase at equilibrium; _{Fe} is the density of the Fe matrix; and _{MC} is the density of the precipitated carbide, which can be obtained by linear interpolation:

$$\rho_{\mathrm{MC}} = k_1\rho_{\mathrm{M1C}} + k_2\rho_{\mathrm{M2C}}$$

where k_1 and k_2 are the proportions of M_1C and M_2C phases in the MC phase, determined from the solubility product formulas, with $k_1 + k_2 = 1$; _{TiC} = 4.944×10^3 kg/m^3, _{NbC} = 7.803×10^3 kg/m^3, and _{MoC} = 8.774×10^3 kg/m^3.

The calculated variation curves of precipitate volume fraction with temperature for the Ti-Mo and Ti-Mo-Nb steels are shown in Figure 7 [Figure 7: see original paper]. Under equilibrium conditions, the volume fraction of precipitates in both steels increases with decreasing temperature, and the volume fraction in the Ti-Mo-Nb steel is significantly larger than that in the Ti-Mo steel. For example, at 900°C, the volume fractions of microalloyed second-phase particles in the Ti-Mo-Nb and Ti-Mo steels are 0.2467% and 0.1601%, respectively.

Figure 8 [Figure 8: see original paper] shows the precipitate morphologies and corresponding EDS spectra for both steels cooled at 50°C/s. Figures 8a and 8c reveal that at high cooling rates, both steels contain a small amount of approximately spherical, dispersedly distributed second-phase particles, with the Ti-Mo steel containing significantly fewer particles than the Ti-Mo-Nb steel, consistent with the calculated results in Figure 7. EDS analysis indicates that the microalloyed second-phase particles in the Ti-Mo steel are primarily (Ti, Mo)C, while those in the Ti-Mo-Nb steel are mainly (Ti, Mo, Nb)C. Considering the cooling conditions for both steels, at a cooling rate of 50°C/s, austenite transforms to bainite. Bainite transformation is a semi-diffusional transformation where C atoms cannot undergo long-range diffusion; instead, C primarily forms carbides between ferrite laths, while the diffusion capacity of Nb and Ti decreases, preventing the formation of large amounts of (Nb, Ti)C precipitates. Therefore, the fine (Ti, Mo)C and (Ti, Mo, Nb)C particles in the steels were mainly strain-induced precipitates formed during deformation.

Further HRTEM analysis of the (Ti, Mo)C and (Ti, Mo, Nb)C particles in the two steels is shown in Figure 9 [Figure 9: see original paper]. Fourier transform analysis indicates that the precipitates in both steels have an fcc structure. Using the (200) interplanar spacing, the lattice constants of the (Ti, Mo)C and (Ti, Mo, Nb)C particles were calculated to be 0.432 nm and 0.436 nm, respectively, with the latter being slightly larger, primarily due to the larger atomic radius of Nb.

Multiple observation fields were randomly selected, and the size distribution of second-phase precipitates was statistically analyzed using the intercept method, with results shown in Figure 10 [Figure 10: see original paper]. The average particle sizes of (Ti, Mo)C in the Ti-Mo steel and (Ti, Mo, Nb)C in the Ti-Mo-Nb steel are 12.11 nm and 8.69 nm, respectively. The (Ti, Mo)C particle sizes are concentrated in the 5–10 nm range, with 60.5% of particles smaller than 10 nm. In contrast, the (Ti, Mo, Nb)C particle sizes in the Ti-Mo-Nb steel are concentrated in the 2–5 nm range, with 75.3% of particles smaller than 10 nm. Evidently, the larger volume fraction and smaller size of precipitates in the Ti-Mo-Nb steel are important reasons for its increased hardness.

On the other hand, the hardness of steel materials is also closely related to their microstructure. Figure 11 [Figure 11: see original paper] shows the austenite grain morphologies of the two steels after deformation at 900°C. The austenite grains in both steels were elongated to varying degrees, with the Ti-Mo-Nb steel showing more severe flattening of austenite grains than the Ti-Mo steel. The measured average austenite grain sizes were 9.69 μm for the Ti-Mo-Nb steel and 12.94 μm for the Ti-Mo steel, indicating that the second deformation pass for both steels occurred in the non-recrystallization region. Since solid-solution Nb precipitates at austenite grain boundaries, subgrain boundaries, and dislocation lines during deformation, it can effectively hinder the movement of grain boundaries, subgrain boundaries, and dislocations, strongly inhibiting recrystallization and grain growth, thereby refining the deformed original austenite and resulting in finer microstructures and higher hardness after cooling. These results are confirmed in Figures 7 and 8. Moreover, due to grain refinement and other factors, the bainite transformation start temperature in the Ti-Mo-Nb steel is lower than that in the Ti-Mo steel, favoring the formation of lath bainite in the matrix and further increasing the microhardness of the Ti-Mo-Nb steel, as shown in Figure 4.

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3 Conclusions

1. Nb can increase the Ac_1 and Ac_3 temperatures of steel, reduce the decomposition temperature of austenite during cooling, shrink the ferrite-pearlite phase region, shift the bainite phase region to lower temperatures and shorter times, and refine the transformed microstructure.
2. During deformation, both Ti-Mo and Ti-Mo-Nb microalloyed steels undergo strain-induced carbide precipitation as (Ti, Mo)C and (Ti, Nb, Mo)C particles, respectively, distributed randomly. Both precipitates have a NaCl-type structure with lattice constants of 0.432 nm and 0.436 nm, and average particle sizes of 12.11 nm and 8.69 nm, respectively.
3. In the cooling rate range of 20–50°C/s, the Ti-Mo-Nb steel exhibits finer microstructure and higher hardness compared with the Ti-Mo steel, which is mainly attributed to the presence of numerous nanometer-sized (Ti, Nb, Mo)C particles.

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Note: Figure translations are in progress. See original paper for figures.