Effect of tempering time on microstructure and mechanical properties of high-Ti microalloyed quenched martensitic steel (Postprint)

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Abstract

The effects of tempering time on the microstructure and mechanical properties of high Ti microalloyed martensitic steel were investigated using transmission electron microscopy (TEM), X-ray diffraction (XRD), and Vickers hardness testing, elucidating the interplay between precipitation strengthening and microstructural softening in high Ti microalloyed martensitic steel during tempering. The results indicate that when high Ti steel is tempered at 600 ℃ for various durations, the hardness exhibits distinct trends. For tempering times of 10~300 s, the hardness continuously increases, as the precipitation strengthening effect of TiC far exceeds the softening effect resulting from matrix recovery. For tempering times of 300 s~10 h, the hardness maintains an extended plateau, which is attributed to the continuous precipitation of fine TiC particles with an increasing proportion of particles smaller than 5 nm; the strengthening contribution from the growing number of fine TiC particles counterbalances the hardness decrease caused by matrix softening. For tempering times of 10~20 h, the hardness decreases rapidly at a rate higher than that of Ti-free steel, with the average size of TiC particles coarsening from 2.76 nm at 10 h to 3.15 nm at 20 h. Calculations reveal that TiC particle coarsening accounts for a hardness reduction of 11.94 HV, while matrix softening accounts for a hardness reduction of 24.56 HV, indicating that matrix softening is the dominant factor for hardness decrease, whereas TiC particle coarsening accelerates the hardness reduction in high Ti steel and represents another significant contributing factor.

Full Text

Effect of Tempering Time on Microstructure and Mechanical Properties of High Ti Microalloyed Quenched Martensitic Steel

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Abstract

The influence of tempering time on the microstructure and mechanical properties of high Ti microalloyed martensitic steel was investigated using transmission electron microscopy (TEM), X-ray diffraction (XRD), and Vickers hardness testing. The interaction between precipitation strengthening and microstructural softening in high Ti microalloyed martensitic steel during tempering was elucidated. The results demonstrate that the hardness of high Ti steel exhibits distinct trends when tempered at 600 °C for varying durations. During tempering from 10 to 300 s, hardness continuously increases because the precipitation strengthening effect of TiC far exceeds the softening caused by matrix recovery. From 300 s to 10 h, hardness maintains a prolonged plateau due to the continuous precipitation of fine TiC particles, with an increasing proportion of particles smaller than 5 nm. The strengthening contributed by these growing fine TiC particles offsets the hardness decrease resulting from matrix softening. Between 10 and 20 h of tempering, hardness drops rapidly at a rate exceeding that of Ti-free steel. The average TiC particle size coarsens from 2.76 nm at 10 h to 3.15 nm at 20 h. Calculations indicate that TiC coarsening causes a hardness reduction of 11.94 HV, while matrix softening accounts for a 24.56 HV decrease. This reveals that matrix softening is the primary factor responsible for hardness reduction, while TiC particle coarsening accelerates the hardness decrease in high Ti steel, representing another important contributing factor.

Keywords: tempering time, hardness, TiC, coarsening, martensite lath

Introduction

Steel tempering involves complex processes including carbon atom segregation, carbide precipitation, decomposition of retained austenite, and recovery/recrystallization of the martensitic structure. These reactions occur simultaneously or intersect with one another, leading to complicated changes in steel properties during tempering. Carbide precipitation and growth are inevitably related to tempering time and temperature, while the tempered microstructure directly determines the mechanical properties of steel. Investigating the relationship between tempering parameters and mechanical properties is crucial for controlling steel performance and developing rational heat treatment processes, and has received extensive attention for quite some time.

For many years, numerous scholars have endeavored to explore functional relationships between tempering hardness and tempering temperature or time, providing theoretical foundations and experimental bases for predicting the tempering hardness of quenched steel and conveniently calculating optimal tempering parameters. However, with the development of transmission electron microscopy (TEM), application of three-dimensional atom probe (3DAP) technology, and continuous improvement of physicochemical phase analysis techniques, changes in martensitic matrix structure, carbide composition evolution, and particle growth kinetics during tempering have become research hotspots worldwide. These fundamental studies are essential for optimizing alloy composition and enhancing precipitation strengthening effects.

Caron and Krauss investigated the variation in the proportion of low-angle grain boundaries in lath martensite of C0.2Ni0.01 steel during tempering, finding that the area of low-angle grain boundaries per unit volume decreased sharply during high-temperature (600-700 °C) short-term tempering, then quickly reached a stable value before gradually decreasing with further tempering time extension. Takaki et al. demonstrated that for C0.21Ni1.59Cr0.50Mo0.16 steel subjected to long-term annealing at 700 °C, continuous carbide growth at martensite lath boundaries and subgrain formation were the main factors causing strength reduction. Zhong et al. studied the effects of tempering time (10 min to 400 h) at 510 °C on the microstructure and properties of C0.16Ni10Co14CrMo and C0.23Ni12Co14Cr3Mo steels, proposing that hardness reduction was primarily due to M2C carbide coarsening and loss of coherency with the matrix, with M2C coarsening in 23NiCo steel being slower than in 16NiCo steel.

Liu et al. used three-dimensional atom probe to investigate the effects of tempering time and temperature on the precipitation, growth, and coarsening of alloy carbides in C0.056Mn1.25V0.10Nb0.029 microalloyed steel, discussing in detail the influence of tempering time on carbide composition evolution and its underlying mechanisms. However, the reasons for hardness reduction after tempering at 550 °C for 100 h were not clarified. It is generally believed that during long-term tempering (>15 h) of low-carbon microalloyed steels, hardness reduction results from carbide coarsening, martensite recovery softening, or a combination of both. Nevertheless, which factor plays the dominant role has remained inconclusive, necessitating further in-depth research.

Meanwhile, with the advancement and application of Ti microalloying technology, theoretical and applied research on Ti in steels has become increasingly profound. However, to date, few reports have addressed the microstructural and mechanical property changes in high Ti quenched martensitic steel during long-term tempering and their influencing factors. Therefore, studying the evolution of microstructure and properties in high Ti quenched martensitic steel during tempering and investigating the causes of hardness reduction after prolonged tempering hold significant theoretical importance and practical value. This work examines the changes in TiC precipitation amount, particle size, and microstructure during tempering, and elucidates the intrinsic factors responsible for hardness curve variations, aiming to benefit the application and promotion of high Ti microalloying technology in tempered martensitic steels.

Experimental Methods

The experimental steels were melted in a 50 kg VITF-0.05 vacuum induction furnace, then hot-forged into bars with a diameter of 15 mm. The chemical compositions are listed in . Steel No. 1 is Ti-free steel, while Steel No. 2 is high Ti microalloyed steel. The steel bars were solution-treated at 1250 °C for 30 min, water-quenched, and then machined into standard specimens measuring 8 mm in diameter and 12 mm in length.

Using a Gleeble 1500D thermal simulator, standard specimens were held at 600 °C for 10, 60, 180, 300, and 600 s, respectively, followed by water quenching. Additional specimens were tempered in a heat treatment furnace for 0.5, 1, 3, 5, 7, 10, 15, and 20 h, respectively, with water quenching upon removal. All specimens were placed in the furnace at temperature, and timing began once temperature was reached.

The water-quenched specimens were ground and polished, and their hardness was measured using a VH5 Vickers hardness tester with a 5 kg load and 10 s dwell time. Five measurements were taken for each specimen and averaged. The hardness variation curves with tempering time were plotted using Origin software. TEM foil specimens were prepared by mechanical thinning followed by twin-jet electropolishing, and the morphology and distribution of precipitates were observed using an H800 transmission electron microscope. For selected specimens, after polishing and etching, carbon was deposited on the surface and extraction was performed using 4% nitric acid alcohol (volume fraction). The extracted residue was collected on a 75 m Cu mesh and air-dried. A JEM

2100F high-resolution field emission transmission electron microscope (HRTEM) with 0.23 nm resolution was used to analyze the composition, distribution, and size of precipitates. Particle sizes were measured from multiple HRTEM images using Nano-Measurer software, with approximately 1000 particles counted to determine the average size distribution after tempering.

The types and amounts of precipitates after different tempering times were determined by physicochemical phase analysis combined with X'Pert Pro MPD X-ray diffraction (XRD). The precipitate powders obtained from phase analysis extraction were prepared as small-angle scattering specimens, and the size distribution of precipitates after various tempering times was measured using Pro MPD (SAXS).

Results

2.1 Vickers Hardness

[Figure 1: see original paper] shows the hardness variation curves of the two experimental steels at 600 °C as a function of tempering time. Both steels exhibit significant hardness decreases after 10 s of tempering compared to the as-quenched state, though the reduction is more pronounced in Steel No. 1. With increasing tempering time, the hardness evolution of both steels can be divided into three stages: Stage I (10-300 s), where Steel No. 1 hardness decreases slowly while Steel No. 2 hardness increases steadily, reaching a peak of 360 HV at 300 s; Stage II (300 s-10 h), where Steel No. 1 hardness decreases gradually while Steel No. 2 high Ti steel exhibits a hardness plateau; and Stage III (10-20 h), where both steels show hardness reduction, but the decreasing rate is significantly higher for Steel No. 2.

Ti Precipitation Behavior

The Ti precipitation amount in Steel No. 2 after water quenching is 0.04% (mass fraction). After tempering for 300 s, 1 h, 3 h, 7 h, 10 h, and 20 h, the Ti precipitation amounts are 0.061%, 0.108%, 0.117%, 0.126%, 0.130%, and 0.132%, respectively. This indicates rapid Ti precipitation within 300 s, primarily due to high Ti supersaturation in the early tempering stage. Between 300 s and 10 h, the Ti precipitation rate gradually decreases while the precipitation amount continues to increase. From 10 to 20 h, the Ti precipitation amount increases by only 0.002%, indicating that precipitation essentially stagnates.

2.2 TiC Particle Size Distribution

[Figure 2: see original paper] presents the TiC particle size distributions in Steel No. 2 after different tempering times as determined by SAXS. After tempering for 1 h, the mass fraction of TiC particles smaller than 5 nm increases by 12.4% compared to 300 s tempering, corresponding to the rapid increase in TiC precipitation amount during this stage. After 3 h of tempering, the proportion of TiC particles under 10 nm rises to 27.8%. With further tempering time extension, the proportion of particles smaller than 5 nm continuously increases, reaching 33.8% after 20 h. Although the TiC precipitation amounts after 10 and 20 h are similar, the proportion of particles smaller than 5 nm increases by 13.5% compared to 7 h. However, hardness decreases significantly after 20 h compared to 10 h, suggesting that some TiC particles likely underwent coarsening during this stage.

[Figure 3: see original paper] shows the size distribution of TiC particles smaller than 5 nm in Steel No. 2 after tempering for 10 and 20 h, measured from HRTEM images. After 20 h of tempering, the proportion of 1-3 nm particles decreases while that of 3-5 nm particles increases. Calculations reveal that the average particle size is 2.76 nm after 10 h and increases to 3.15 nm after 20 h, confirming that TiC particles coarsen with extended tempering time, though the coarsening is limited. This limited coarsening is primarily attributed to the high diffusion activation energy of the controlling element Ti and the relatively low tempering temperature.

2.3 TEM Analysis

[Figure 4: see original paper] displays the morphology and EDS analysis of TiC particles in Steel No. 2 after various tempering times. After 300 s of tempering, TiC particles smaller than 10 nm are relatively few and unevenly distributed ([Figure 4a: see original paper]). After 1 h, the number of fine TiC particles increases significantly. Following 7 h of tempering, TiC particles remain very fine at approximately 2-3 nm. After 10 h, TiC particles under 10 nm become more numerous than after 7 h, with a finer average particle size ([Figure 4d: see original paper]). After 20 h, TiC particles smaller than 10 nm are even more abundant than after 10 h, consistent with the size distribution evolution shown in [Figure 2: see original paper].

[Figure 5: see original paper] shows TEM images of thin foil specimens from Steel No. 2 after different tempering times at 600 °C. After 300 s, the microstructure consists of lath martensite with numerous cementite particles precipitated between laths, measuring approximately 30-40 nm. After 1 h, cementite particles become coarser and less numerous. Following 7 h, cementite particles further coarsen to about 120-140 nm. After 20 h, the martensite laths show noticeable coarsening with blurred lath boundaries, and cementite particles within laths decrease in number ([Figure 5d: see original paper]), primarily residing at lath boundaries with sizes of 200-250 nm ([Figure 5e: see original paper]), while still maintaining the martensite packet morphology.

[Figure 6: see original paper] presents TEM images of thin foil specimens from Steel No. 1 after various tempering times. After 180 s, the microstructure is tempered lath martensite with short rod-shaped cementite particles distributed between and within laths, approximately 60-70 nm in size. Sub-cells appear within laths, causing significant hardness reduction compared to the as-quenched state ([Figure 6a: see original paper]). After 10 h, martensite laths widen compared to 300 s tempering, some subgrain boundaries disappear, subgrain sizes further increase, and cementite particles coarsen, indicating more pronounced martensite matrix recovery and continued hardness reduction ([Figure 6b: see original paper]). After 20 h, dislocation density decreases significantly compared to 10 h, grains show obvious polygonization, and the martensite matrix softens considerably, resulting in a 20 HV hardness decrease from the 10 h tempered condition.

Discussion

3.1 Effect of Tempering 10 s-10 h on Hardness of Steel No. 2

During tempering from 10 to 300 s, Steel No. 1 hardness decreases slowly while Steel No. 2 hardness increases linearly, reaching a peak of 360 HV at 300 s—manifesting so-called secondary hardening. The high Ti content in Steel No. 2 leads to a Ti precipitation increase of 0.021% after 300 s tempering compared to the as-quenched state, indicating rapid TiC precipitation. Within this short tempering duration, the precipitation strengthening effect from numerous fine TiC particles far exceeds the softening caused by dislocation density reduction and matrix recovery. Additionally, smaller cementite particles pin dislocations and hinder grain boundary migration, retarding martensite recovery and thereby slowing hardness reduction due to matrix recovery.

During tempering from 300 s to 10 h, Steel No. 2 exhibits a long-term hardness plateau. Literature indicates that Nb-V steel also shows a similar hardness plateau after tempering at 500 °C for 4 h. This phenomenon demonstrates not only excellent resistance to tempering softening but also dynamic microstructural and precipitate evolution within the steel that maintains equilibrium. The reasons for this prolonged hardness plateau in Steel No. 2 are elaborated below based on microstructural changes.

Between 300 s and 1 h of tempering, Ti precipitation amount increases by 0.047%, and the proportion of TiC particles smaller than 5 nm rises by 12.4% compared to 300 s tempering ([Figure 2: see original paper]). Fine TiC particles become significantly more numerous and uniformly distributed ([Figure 4b: see original paper]). However, this massive TiC precipitation does not increase Steel No. 2 hardness but rather maintains it at the previous level. With extended tempering time, cementite particles within martensite laths decrease in number and increase in size after 1 h, while martensite matrix recovery becomes more evident ([Figure 5b: see original paper]), which should theoretically reduce hardness. Therefore, it can be inferred that the hardness increment from TiC precipitation is exactly offset by the hardness decrement from matrix softening, resulting in unchanged hardness.

When tempering time extends from 1 to 7 h, Ti precipitation amount increases by 0.018%, and the proportion of TiC particles under 5 nm increases by 2.6% compared to 1 h tempering, with even finer average particle sizes. This indicates that TiC precipitation rate slows with increasing tempering time, though the precipitation amount continues to increase gradually, and the proportion of fine TiC particles slowly rises, which is beneficial for hardness improvement. After 7 h tempering, lath boundaries become less distinct compared to 1 h tempering, some have disappeared, lath width increases, and cementite particles within laths decrease in number but increase in size ([Figure 5c: see original paper]), indicating further intensified matrix softening compared to 1 h tempering. Consequently, the slight beneficial effect of slow TiC precipitation on hardness exactly compensates for the hardness reduction caused by martensite matrix softening. With extended tempering time, small cementite particles gradually dissolve while large ones coarsen. The TiC formed by strong carbide-forming element Ti combining with C replaces some dissolved cementite, leading to continuously increasing TiC precipitation amount. The sustained strengthening from TiC precipitation maintains dynamic equilibrium with matrix recovery softening, which is the fundamental reason for the long-term hardness plateau in Steel No. 2 during tempering from 300 s to 10 h.

During tempering from 300 s to 10 h, Steel No. 1 hardness decreases by 32 HV, whereas Steel No. 2 hardness remains constant. This demonstrates that appropriate Ti addition not only significantly increases tempering hardness but also strongly pins dislocation recovery, markedly slowing martensite recovery and strongly retarding recrystallization. Moreover, the higher Ti content enables more complete and prolonged TiC precipitation, providing stronger inhibition of martensite matrix recovery and thus maintaining a long-term hardness plateau.

3.2 Effect of Tempering 10-20 h on Hardness of Steel No. 2

During tempering from 10 to 20 h, Steel No. 1 shows substantial hardness reduction, indicating that softening due to increased matrix recovery is the primary factor for hardness decrease ([FIGURE:6b-c]), though its decreasing rate is significantly lower than that of Steel No. 2 at the same stage. However, Steel No. 2 does not undergo recrystallization after 10-20 h tempering ([Figure 5d: see original paper]), and its matrix recovery is less pronounced than in Steel No. 1. Therefore, other factors must contribute to the rapid hardness reduction in Steel No. 2, with matrix recovery softening being only one of them.

This phenomenon of matrix recovery without recrystallization is common in low-carbon martensitic steels during tempering. After 20 h tempering, the mass fraction of TiC particles smaller than 5 nm in Steel No. 2 increases by 13.5% compared to after 7 h, yet hardness decreases significantly after 10 h tempering. Research has shown that when tempering temperature is sufficiently high and holding time is sufficiently long, alloy carbides continue to coarsen under the driving force of interfacial energy, resulting in actual carbide sizes far exceeding those at precipitation completion and thereby weakening or eliminating their dispersion strengthening effect at elevated temperatures.

Thus, TiC particle coarsening occurs in Steel No. 2 during tempering from 10 to 20 h, representing another factor causing hardness reduction and being the main reason for the accelerated hardness decreasing rate compared to Steel No. 1. Liu et al.'s three-dimensional atom probe studies on Nb-V steel tempered at 550 °C showed that when tempering time increased from 10 to 20 h, carbide number density decreased while average precipitate size increased, further confirming carbide coarsening with extended tempering time and its contribution to hardness reduction, consistent with the trend observed in Steel No. 2 where TiC coarsening accelerates hardness decrease.

Based on the size distribution of TiC particles smaller than 5 nm in Steel No. 2 after tempering for 10 and 20 h ([Figure 3: see original paper]), the average sizes are 2.76 nm and 3.15 nm, respectively, confirming TiC particle coarsening. This stage primarily involves dissolution of small particles and growth of large particles—the so-called Ostwald ripening process. For the experimental steel, the main factors affecting strength changes during tempering include dislocation strengthening, solid solution strengthening, and precipitation strengthening. The Ti precipitation amount changes minimally in Steel No. 2 during tempering from 10 to 20 h, so the effect of solid solution strengthening on hardness can be neglected. Hardness reduction is determined by two aspects: (1) hardness decrease caused by reduced dislocation density and matrix recovery softening ([Figure 5: see original paper]), as confirmed by the hardness reduction in Steel No. 1 due to martensite matrix recovery softening; and (2) decreased precipitation strengthening capacity resulting from TiC particle coarsening.

The Ti precipitation amounts in Steel No. 2 after 10 and 20 h tempering are 0.130% and 0.132%, respectively. Considering measurement precision limitations, it is reasonable to assume that Ti precipitation amount remains constant at 0.132% during 10-20 h tempering, while ignoring Ti consumption by N combination. For second-phase strengthening, the volume fraction change of precipitates smaller than 5 nm is crucial for precipitation strengthening effects. Based on the mass fractions of TiC particles under 5 nm after 10 and 20 h tempering from [Figure 2: see original paper], the corresponding volume fractions can be estimated as 0.000532% and 0.000886%, respectively. When slip dislocations bypass non-deformable particles via the Orowan mechanism, the precipitation strengthening increment can be estimated, and the relationship between yield strength and hardness can be characterized by the following equations:

$$\sigma_p = 8.995 \times 10^3 \cdot \frac{f^{1/2}}{d} \ln(2.417d)$$

$$f = [Ti] \cdot \frac{\rho_{Fe}}{\rho_{TiC}} \cdot \frac{A_C}{A_{Ti}}$$

$$\sigma_y = -110.9 + 2.507H_v$$

where σ_p is the strengthening increment from TiC (MPa), f is the volume fraction of TiC (%), d is the average TiC size (nm), $[Ti]$ is the precipitated Ti mass fraction (%), ρ_{Fe} is the density of α-Fe matrix (7.875 g/cm^3), ρ_{TiC} is the density of TiC (4.944 g/cm^3), A_C is the atomic weight of C (12.011), A_{Ti} is the atomic weight of Ti (47.867), σ_y is the yield strength of the experimental steel (MPa), and H_v is the Vickers hardness (kg/mm^2).

Calculations using equations (1) and (2) yield precipitation strengthening values of 172.56 MPa and 142.63 MPa after 10 and 20 h tempering, respectively, with a difference of $\Delta\sigma_p = 29.93$ MPa. Based on the superposition principle of various strengthening mechanisms on yield strength in steel, the yield strength change due to precipitation strengthening is $\Delta\sigma_y = 29.93$ MPa. Combining with equation (3), the hardness change caused by precipitation strengthening is $\Delta H_v = 11.94$ HV.

The hardness difference in Steel No. 2 between 10 and 20 h tempering is 36.50 HV. Therefore, the hardness reduction caused by matrix recovery softening is approximately $\Delta H_v = 24.56$ HV. This demonstrates that martensite matrix recovery softening is the dominant factor for hardness reduction after 10 h tempering in high Ti steel, while TiC particle coarsening is an important factor that directly accelerates hardness decrease during this stage, explaining why Steel No. 2 exhibits a higher hardness reduction rate than Steel No. 1 after 10 h tempering. However, TiC particles exhibit excellent tempering resistance stability at 600 °C, resulting in a very low coarsening rate, so the effect of TiC coarsening on hardness is not prominent. The hardness reduction of 24.56 HV in Steel No. 2 is close to the 20.26 HV reduction in Steel No. 1 due to matrix softening, confirming that the calculated hardness reduction caused by TiC coarsening in Steel No. 2 is reasonably accurate.

Conclusions

1. High Ti steel tempered at 600 °C for different times exhibits distinct hardness trends: hardness increases steadily during 10-300 s tempering, reaching a peak of 360 HV at 300 s; maintains a long-term plateau during 300 s-10 h tempering; and decreases substantially after 10-20 h tempering.

2. With extended tempering time at 600 °C, high Ti steel shows reduced cementite particles within laths but with increased size, increased lath width, and progressively enhanced martensite matrix recovery. However, no recrystallization occurs, primarily due to the pinning effect of nanometer-sized TiC particles on grain boundaries that hinders matrix recovery and suppresses recrystallization. Additionally, fine cementite particles in the

early tempering stage (10-300 s) also contribute to grain boundary pinning and retard martensite matrix recovery.

3. The hardness increase during 10-300 s tempering in high Ti steel is mainly attributed to the strengthening effect from massive precipitation of fine TiC particles exceeding the softening effect from martensite matrix recovery and dislocation density reduction. The long-term hardness plateau during 300 s-10 h tempering results from dynamic equilibrium between the continuous precipitation of ~2-3 nm TiC particles (with increasing proportion of particles under 5 nm) and matrix recovery softening.
4. The average TiC particle sizes after 10 and 20 h tempering in high Ti steel are 2.76 nm and 3.15 nm, respectively, confirming TiC coarsening after 10 h tempering. This coarsening causes a hardness reduction of 11.94 HV, while matrix recovery softening accounts for 24.56 HV of hardness decrease. Martensite matrix recovery softening is the dominant factor for hardness reduction after 10 h tempering, while TiC particle coarsening accelerates hardness decrease but is not the primary factor. The close agreement between the 20.26 HV hardness reduction in Steel No. 1 (due to matrix softening) and the 24.56 HV reduction in Steel No. 2 validates the reasonable estimation of hardness reduction caused by TiC coarsening.

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