

Article

Effect of Tempering Time on Carbide Evolution and Mechanical Properties of a Nb-V-Ti Micro-Alloyed Steel

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Abstract: The evolution of the microstructure, the precipitation behavior, and the mechanical performances of Nb-V-Ti micro-alloyed steel prepared under different tempering time were studied using transmission electron microscopy (TEM), X-ray diffraction (XRD), and mechanical tests. It was found that the width of the martensite laths increases with the increasing tempering time. Several kinds of carbides, including M_3C , M_2C , $M_{23}C_6$, M_7C_3 , and MC particles, were identified after tempering. The MC carbides remain stable during tempering, but the transformation behavior of other carbides was identified. The transformation sequence can be summarized as: $M_3C \rightarrow M_2C \rightarrow M_7C_3 \rightarrow M_{23}C_6$. The strength decreases and the Charpy impact toughness increases gradually with the increase in the tempering time. The ultimate strength (UTS) decreases from 1231 to 896 MPa, and the yield strength (YS) decreases from 1138 to 835 MPa. The -40°C Charpy impact toughness increases from 20 to 61 J as the tempering time increases from 10 min to 100 h. The evolution of carbides plays an important role in their mechanical performances.

Keywords: carbide; tempering time; Nb-V-Ti micro-alloyed steel; mechanical performance



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1. Introduction

With the rapid development of modern industry and technology, the exploitation and utilization of crude oil and natural gas have been gradually increasing [1–3]. In the past several decades, oil and gas exploration has been extended to abyssal regions [4]. To enhance the transportation efficiency, a larger diameter and a higher operation pressure have been adopted. This requires an excellent combination of a high strength and a good toughness for pipeline steels [5–8]. Therefore, micro-alloyed components are added to pipeline steels to improve their mechanical performance through grain refinement and precipitation strengthening [9–12]. Nowadays, micro-alloyed ultra-high-strength pipeline steel is indispensable to the development of offshore oil and gas exploitation.

The conventional heat treatment of micro-alloyed steels consists of quenching and tempering. Quenching and tempering at different temperatures for different times can provide a beneficial combination of a microstructure and precipitates [13,14]. Multi-phase microstructures and different types of carbides can further meet the requirements for excellent mechanical properties [15–17].

The precipitation of fine particles during tempering plays an important role in improving the strength and toughness of micro-alloyed ultra-high-strength steels. Much research on the evolution of carbide precipitates under different tempering times has been reported. Li et al. [1] investigated the evolution of precipitates in a G18CrMo2-6 steel during tempering at 680°C for up to 100 h. It was found that M_3C carbides were transformed from M-A particles first, and then MC carbides precipitated. M_3C particles refined and spheroidized gradually, and $M_{23}C_6$ coarsened with increasing the tempering time. Moon et al. [18] clearly showed the transformation sequence of carbide precipitates in Cr-Mo API steels. They also found that the precipitation sequence of carbides during tempering at 650°C was: MC +

$M_3C \rightarrow MC \rightarrow MC + M_7C_3 + M_{23}C_6$. Tao et al. [19] investigated the evolution of different carbides in X12CrMoWVNbN10-1-1 steel after tempering and found that $M_{23}C_6$ possesses a higher thermal stability than M_7C_3 . The precipitation sequence of carbides can be summarized as: $M_3C \rightarrow M_7C_3 \rightarrow M_{23}C_6$. On the contrary, Asadabad et al. [20] reported that part of $M_{23}C_6$ transformed into M_7C_3 in 4.5Cr-2W-0.25V-0.1C steel. Janovec [21] reported that the Cr content determines the transformation sequence of precipitates in Cr-alloyed steels. With the addition of 1wt.% Cr, the precipitation sequence of Cr-Mo-V steels was proven to be $M_{23}C_6 \rightarrow M_7C_3$ [18]. In middle- or high-Cr steels, Jia et al. found that four kinds of precipitates, M_2X , M_3C , $M_{23}C_6$, and M_7C_3 , existed in 9CrMoCoB (CB2) steel [22], and the thermal stability of M_7C_3 was lower than $M_{23}C_6$ [23–25]. However, the transformation sequence of the carbide precipitates of Nb-V-Ti micro-alloyed ultra-high-strength steel during high-temperature tempering is still not clear, and it should have more attention paid to it.

This paper focused on the carbide evolution of Nb-V-Ti micro-alloyed steel during different tempering times. The microstructure and precipitates under different tempering times were analyzed. Further, the mechanical properties of the explored steel under different tempering times were evaluated, and the correlation between the evolution of the microstructure, the precipitation behavior, and the mechanical performances was also discussed in detail.

2. Experimental

The chemical composition of the investigated steel was 0.25C-0.29Si-0.4Mn-0.97Cr-0.88Mo-0.015Nb-0.089V-0.026Ti (wt.%). In this study, the investigated steels were austenitized at 1000 °C for 30 min, quenched with water, and then tempered at 600 °C for 10 min, 30 min, 1 h, 2 h, 3 h, 5 h, 10 h, 25 h, 50 h, and 100 h, respectively. All the specimens were cooled in water to room temperature after tempering.

The thin-foil specimens were used for TEM characterization, and they were electropolished in a double-jet electropolishing device with a solution of 7% perchloric acid and 93% ethanol at −20 °C. The morphologies, sizes, distributions, and chemical compositions of the precipitates were identified using TEM (JEM-2100F, JEOL, Tokyo, Japan). The experimental steels were immersed in hydrochloric acid for 15 days, and then the precipitated particles were extracted by centrifuge. Hydrochloric acid was removed by continuously adding alcohol to the solution during extraction. The precipitated particles were then dried in a drying cabinet at 50 °C for more than 5 h. The phase composition and the various carbides formed during tempering were determined by XRD (D8 Advanced, Bruker, Karlsruhe, Germany).

Tensile tests were performed on the tempered specimens at room temperature. The gauge length of the specimens was 15 mm. An Instron 5565 (Instron, Boston, MA, USA) tensile machine was used for tensile test with a strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$. The standard CVN samples were machined to 10 mm × 10 mm × 55 mm with a notch depth of 2 mm. The tensile and the Charpy impact toughness experiments were repeated three times to ensure the accuracy of the results. The Charpy impact toughness tests were carried out at −40 °C using an Instron MPX (Instron, Boston, MA, USA) impact tester. The samples were cooled in a cold box with air as the cooling medium. Micro-hardness testing was conducted using a MH-6 type (Shanghai Everone Precision Instruments, Shanghai, China) Vickers hardness tester with a maximum load of 100 g for 5 s.

3. Results and Discussion

3.1. Microstructure Evolution under Different Tempering Times

Figure 1 shows the TEM images of samples tempered at 600 °C at different times. The martensite lath with dislocations indicated by arrows can be seen in the microstructure. With a prolonged tempering time, the width of martensite lath increased, and the mean widths of martensite lath were determined to be 149, 199, 247, 282, 305, and 388 nm for the tempering times of 10 min, 30 min, 5 h, 10 h, 50 h, and 100 h, respectively. The lath width of the martensite was obtained by measuring about 200 martensites. The density of the

dislocation was reduced with the extension of the tempering time, which was due to the recovery of the martensite lath.

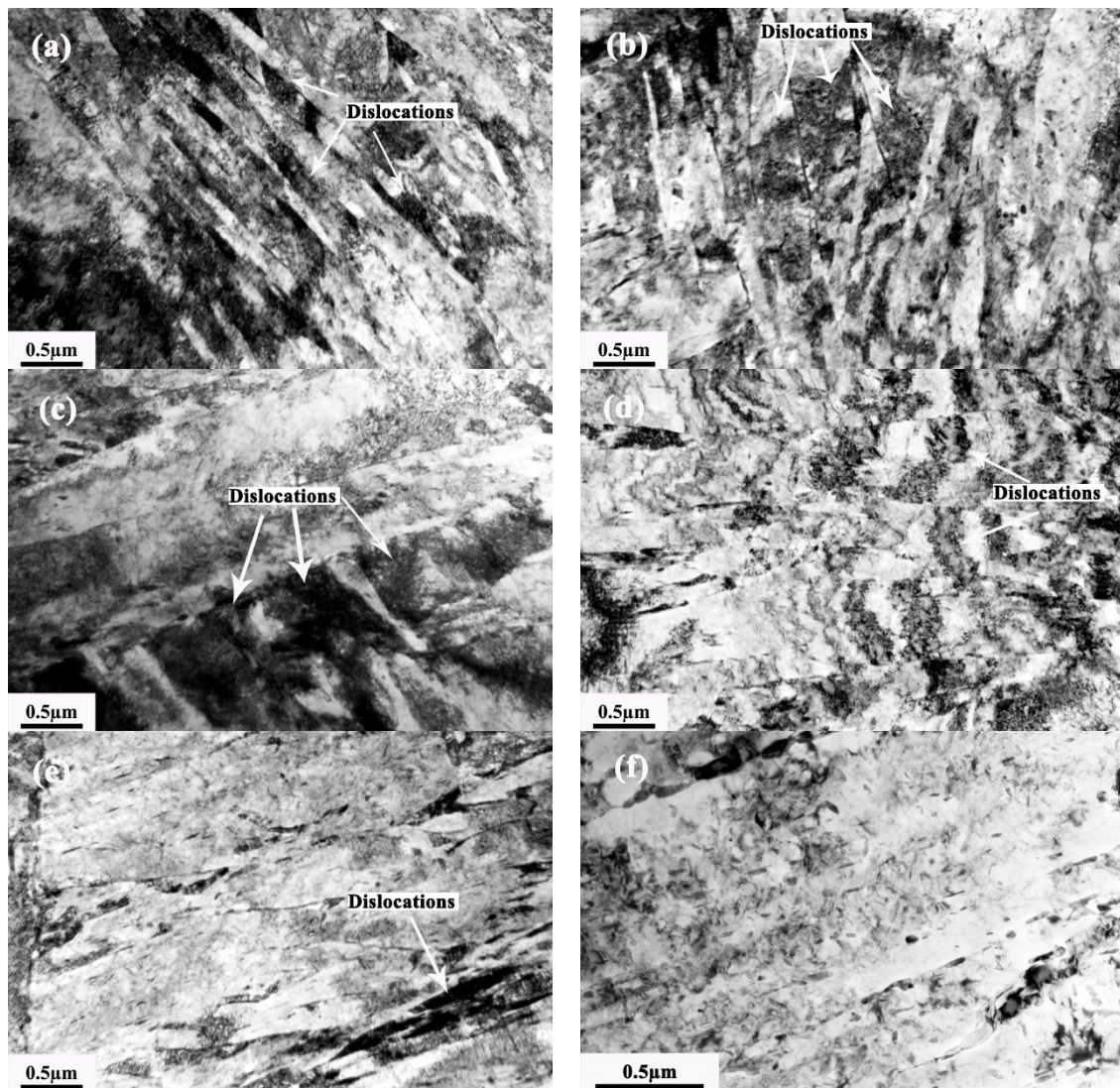


Figure 1. TEM micrographs of steel specimens after being tempered at 600 °C for different periods: (a) 10 min, (b) 30 min, (c) 5 h, (d) 10 h, (e) 50 h, and (f) 100 h.

The microstructure of the retained austenite in the samples tempered at 600 °C is shown in Figure 2. The retained austenites are marked by white arrows. The retained austenite films were continuously distributed between the martensite lathes when the tempering time was 10 min (Figure 2a), and then the films became discontinuous with a prolonged tempering time (Figure 2b,c). After tempering for 100 h, few films could be seen between the martensite laths (Figure 2d). The mean thickness of the retained austenite film decreased with the increase in tempering time, and the average thickness of the retained austenite films tempered for 10 min, 30 min, and 5 h was about 57, 35, and 27 nm, respectively.

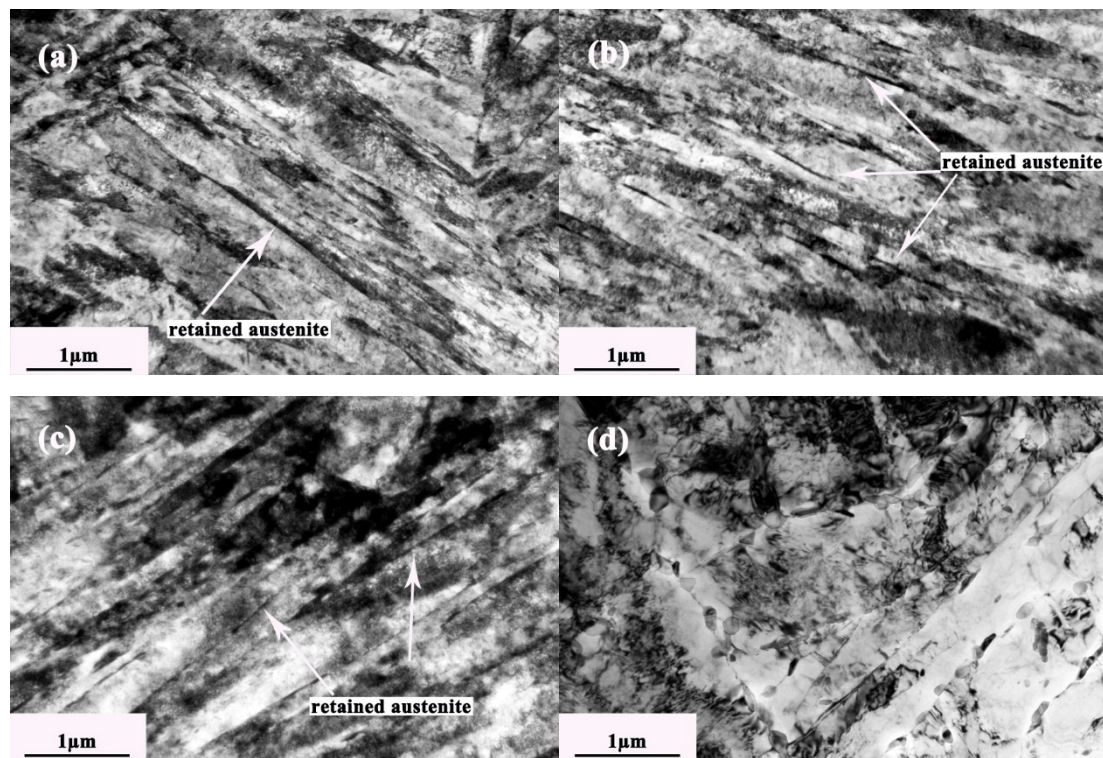


Figure 2. TEM micrographs of retained austenite in the steel specimens after tempering at 600 °C for different tempering periods: (a) 10 min, (b) 30 min, (c) 5 h, and (d) 100 h.

3.2. Precipitation Behavior of Carbide under Different Tempering Times

The TEM micrographs of samples under different tempering times were examined. After tempering for 10 min, needle-like particles could be observed in the interior of the martensite lath. As shown by the black oval in Figure 3a, the mean length of the needle-like precipitates was about 94.2 nm. The sizes of the carbides were obtained by measuring about 400 carbides. According to the corresponding electron diffraction pattern results shown in Figure 3b, the needle-like precipitates possessed an orthorhombic crystal structure. Based on the EDS results (Figure 3c), the carbides contained a high content of Fe and a small amount of Mn and Cr. Referring to the PDF No. 75-0910, the carbide was M_3C with $a = 4.518$ nm, $b = 5.069$ nm, $c = 6.736$ nm, and $\alpha = \beta = \gamma = 90^\circ$.

Spherical nano-sized precipitates with a mean diameter of about 4.89 nm and square particles with a mean dimension of 42.45 nm were also observed, as shown in Figure 4a,d. The EDS analysis results combined with the SAD identifications are shown in Figure 4b–e. These two kinds of particles both had an f.c.c. structure. The nano-sized spherical particles contained Nb and a small amount of Ti and V, and the corresponding PDF card was 47-1418. The square precipitates contained Ti with a small amount of Nb and V, and the corresponding PDF retrieval number was 65-7931. The Fe, Mn, and Cr elements were derived from the matrix. Therefore, these two types of carbides were identified as Nb-rich MC (I) carbide and Ti-rich MC (II) carbide.

When the tempering time was prolonged to 30 min, the needle-like M_3C carbides dissolved into the matrix during tempering and acted as nucleation sites for rod-like particles, as denoted by the arrow in Figure 5a. The EDS results (Figure 5d) indicated that the rod-like particles contained a high iron content and Mo with a tiny amount of Cr and Mn. The SAD pattern shown in Figure 5b matched well with the electron diffraction pattern of M_2C , and the PDF retrieval number was 35-0787. The mean length of the rod-like M_2C particles was about 59.04 nm. Irregular-shaped particles precipitated along the lath or grain boundaries, as marked by the white oval in Figure 5a. The EDS analysis results and the SAD identifications are shown in Figure 5c–e. These precipitates had a complex cubic

structure and contained Fe and a certain amount of Cr and Mn. The corresponding PDF retrieval number was 78-1499, and this type of precipitate was recognized as $M_{23}C_6$. The mean size of $M_{23}C_6$ was around 46.77 nm.

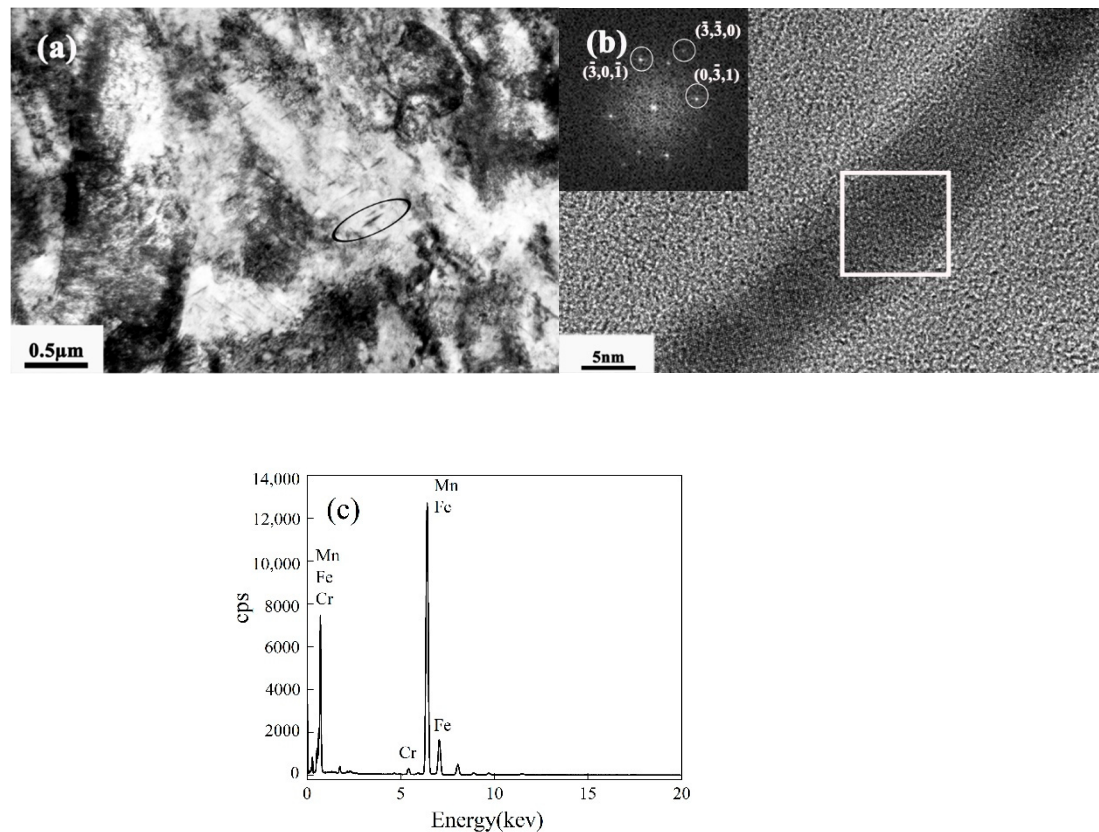


Figure 3. (a) TEM micrographs of M_3C after tempering at 600 °C for 10 min; (b,c) corresponding SAD pattern and EDS analysis of M_3C precipitates, respectively.

The TEM images of the specimens after being tempered for 5 h are shown in Figure 6. It was obvious that more M_2C particles precipitated, and the angle between the particles was about 60°. In addition, some of the M_2C particles connected and formed long-line precipitates, as demonstrated by the white oval in Figure 6a and the black arrow in Figure 6b. The EDS analysis results and the diffraction pattern of these precipitates are indicated in Figure 6c,d. The needle-like precipitates had a hexagonal structure and mainly contained Fe and certain amounts of Cr and Mo. The corresponding PDF retrieval number was 05-0720; thus, the needle-like precipitates were identified to be M_7C_3 carbide.

The TEM images of the samples tempered at 600 °C for 100 h are demonstrated in Figure 7. After tempering for 100 h, the density of the carbides obviously increased, compared with the specimen tempered for 5 h. Most of the needle-like M_7C_3 particles disappeared, and some of the needle-like M_7C_3 particles transformed into rod-like carbides, as shown in Figure 7a. The corresponding electron diffraction pattern is shown in Figure 7b. This type of carbide had a complex cubic structure, which was consistent with the previously identified $M_{23}C_6$. This result showed that the M_7C_3 -type precipitates transformed into $M_{23}C_6$ with a prolonged tempering time. Most of the coarse $M_{23}C_6$ carbides precipitated at the grain boundaries (Figure 7c) with an average size of 86.83 nm. Two kinds of MC carbide particles remained stable during the 100 h tempering, as shown in Figure 7c,d. The diameters of the MC particles presented almost no significant change when compared with the specimen tempered at 600 °C for 10 min. The average size of the MC(I) and MC(II) was about 5.02 and 54.34 nm after tempering for 100 h, respectively.

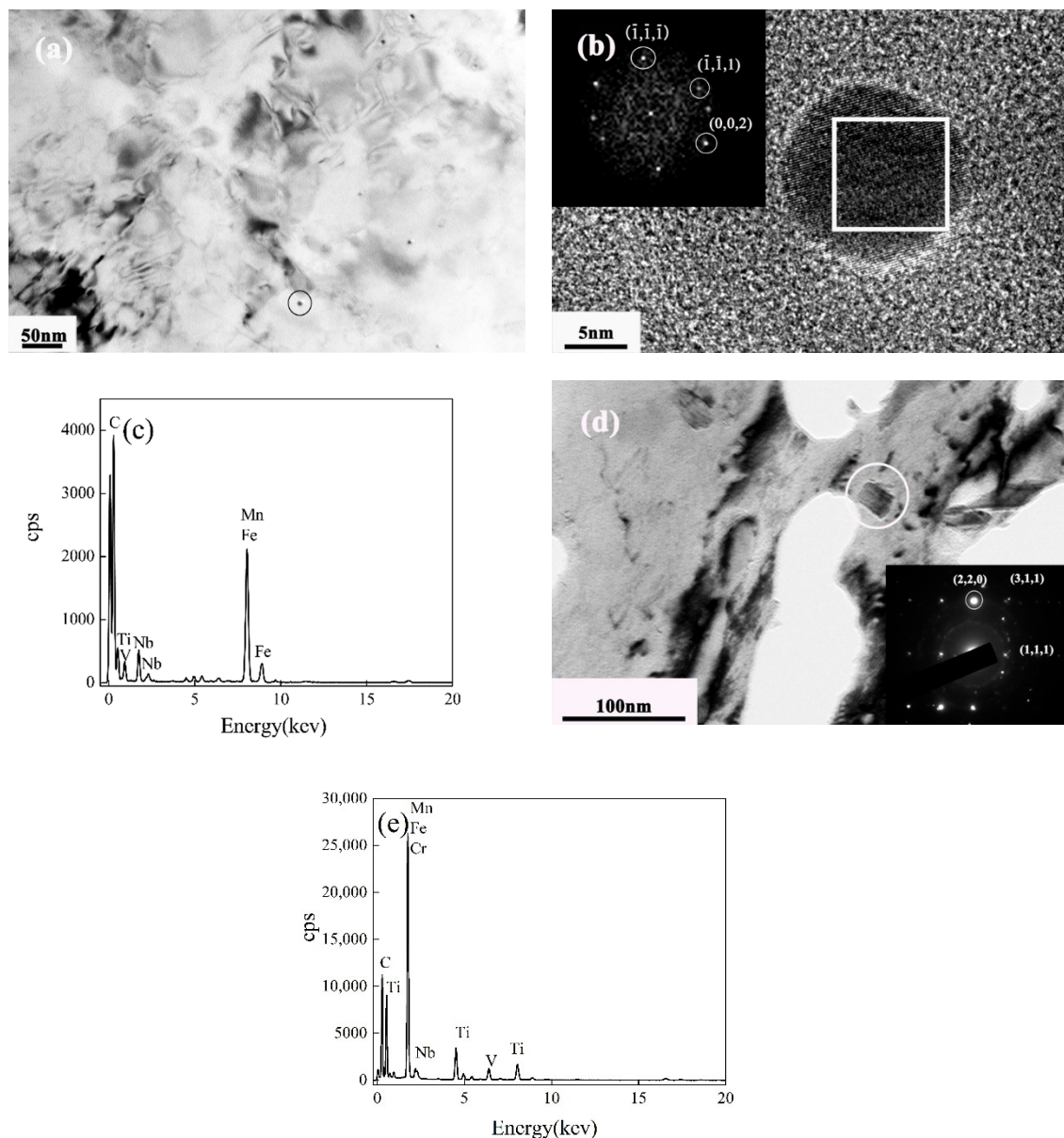


Figure 4. (a–c) TEM micrographs, corresponding SAD pattern, and EDS analysis of Nb-rich MC (I) carbide; (d,e) TEM micrographs, corresponding SAD pattern, and EDS analysis of Ti-rich MC (II) particles after tempering at 600 °C for 10 min.

Meta-stable carbides precipitated during tempering can be gradually replaced by more stable precipitates. The precipitation sequence of different types of carbides relates to the chemical composition of steel, the relative diffusion coefficient of the different alloying atoms, and the nucleation sites of the matrix [19]. Therefore, it is essential to comprehend the transformation sequence of carbides. Several kinds of carbides after tempering at 600 °C for different times were identified above. The transformation sequence of the carbides at different stages is summarized as follows.

Stage 1 (tempered for 10 min): three kinds of carbides (M_3C , MC(I), and MC(II)) were found after tempering at 600 °C for 10 min. The quick precipitation of the M_3C particles could be due to a carbon-diffusion-controlled reaction [26]. Even the meta-stable M_3C precipitated before the other carbides due to the relatively rapid diffusion of interstitial carbon, while the substitutionary atoms remained indiffusible [27]. Some researchers [28] have suggested that smaller spherical MC(I)-type carbide particles precipitate in the ferrite region following a K-S relationship with ferrite. MC(II)-type carbides precipitate in

austenite regions, exhibiting a cube–cube K-S relationship with the matrix. MC-type second-phase particles result in the strengthening of steel by forming fine and densely distributed carbides [29].

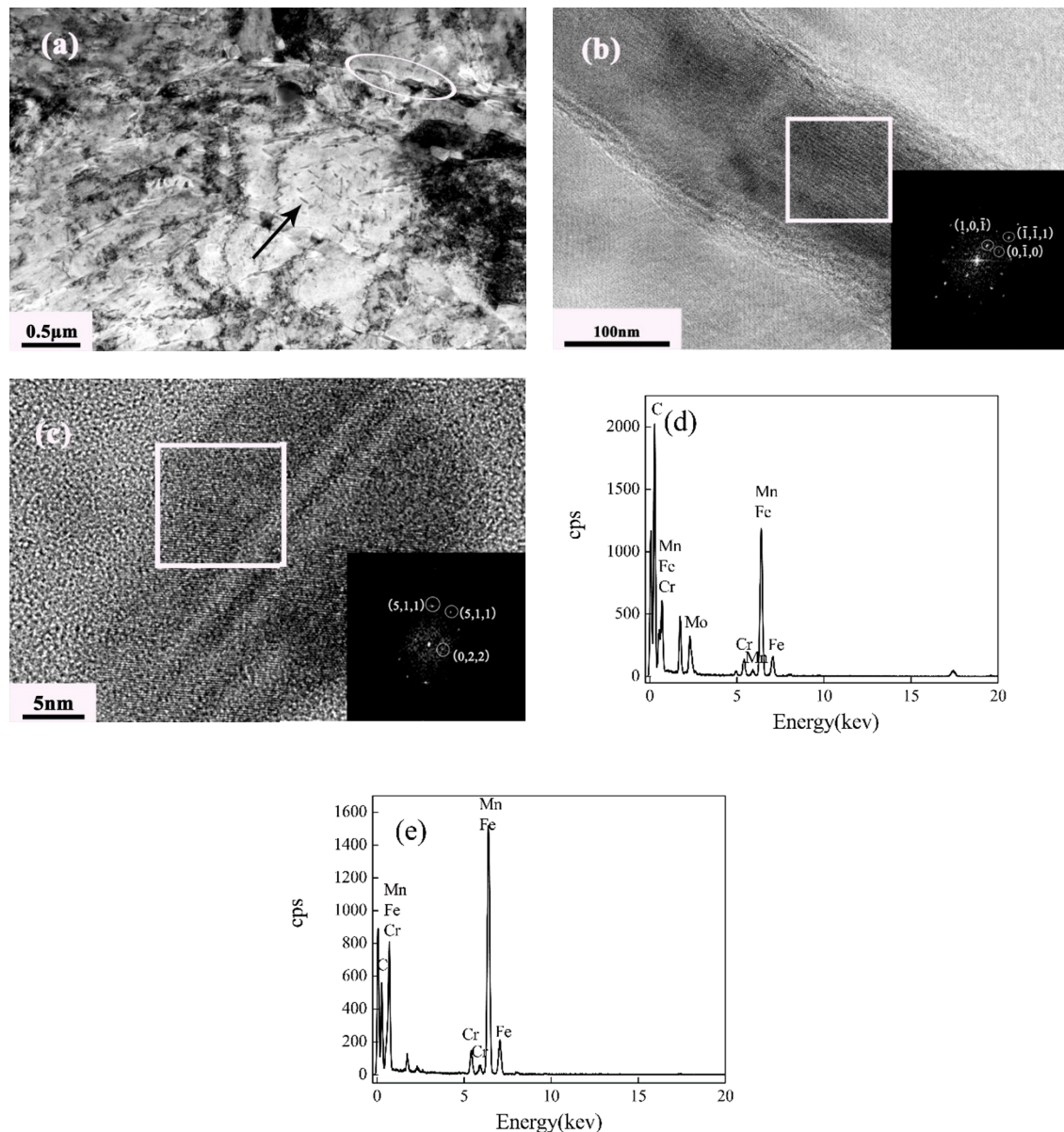


Figure 5. (a,b,d) TEM micrographs, corresponding SAD pattern, and EDS analysis of M_2C particles; (c,e) TEM micrographs, corresponding SAD pattern, and EDS analysis of $M_{23}C_6$ precipitates after tempering at 600 °C for 30 min.

Stage 2 (tempered for 30 min): the M_3C carbides disappeared, and the M_2C and $M_{23}C_6$ carbides precipitated. The formation of M_2C and $M_{23}C_6$ was controlled by substitutionary diffusion, and they hardly precipitated during the early tempering process since the substitutionary alloying components could not sufficiently diffuse during the relatively short tempering process [22]. As the tempering time was prolonged, the substitutionary alloying components could diffuse, which caused the formation of M_2C and $M_{23}C_6$ carbides. The precipitation temperature of the M_2C particles was approximately between 500 and 600 °C, which was mainly determined by the diffusion of Mo [22]. The formation of the M_2C carbides led to the dissolution of the M_3C carbides, which resulted in a secondary hardening effect in the tempered martensitic steel [30]. Fine and dispersive M_2C can improve the

toughness of steel, since the precipitation of M_2C results in the dissolution of brittle M_3C carbides [21].

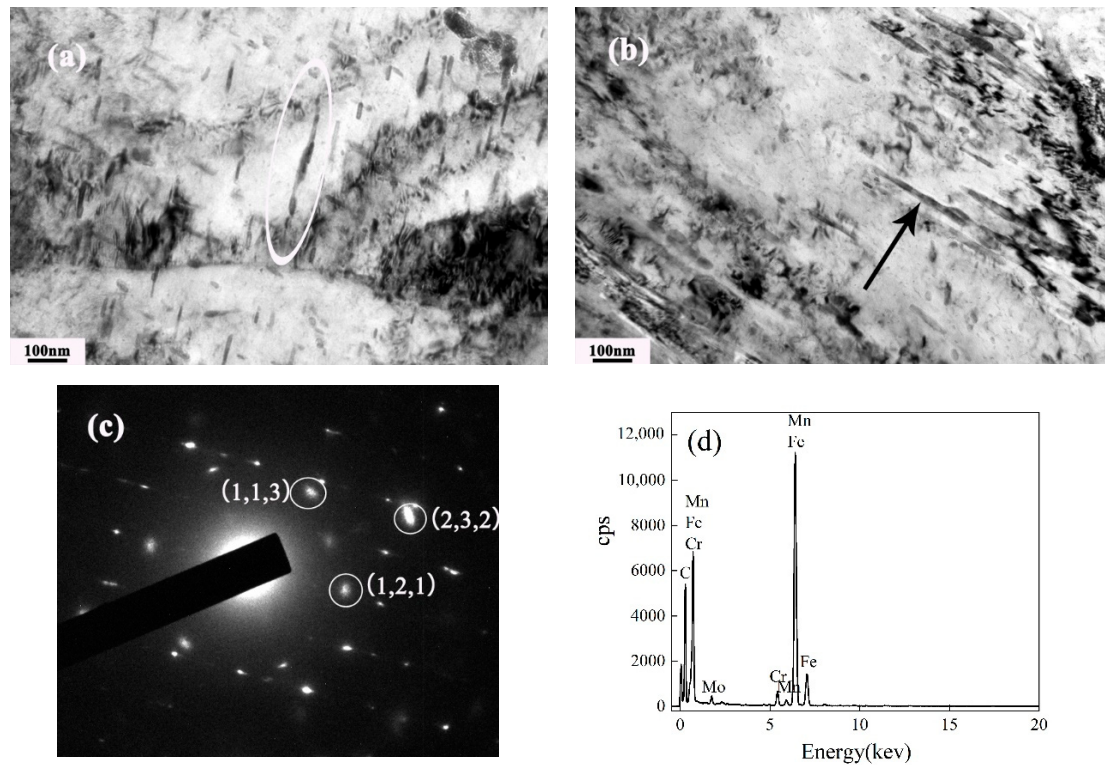


Figure 6. (a,b) TEM micrographs of steel specimen after tempering at 600 °C for 5 h; (c,d) corresponding SAD pattern and EDS analysis results of M_7C_3 particles.

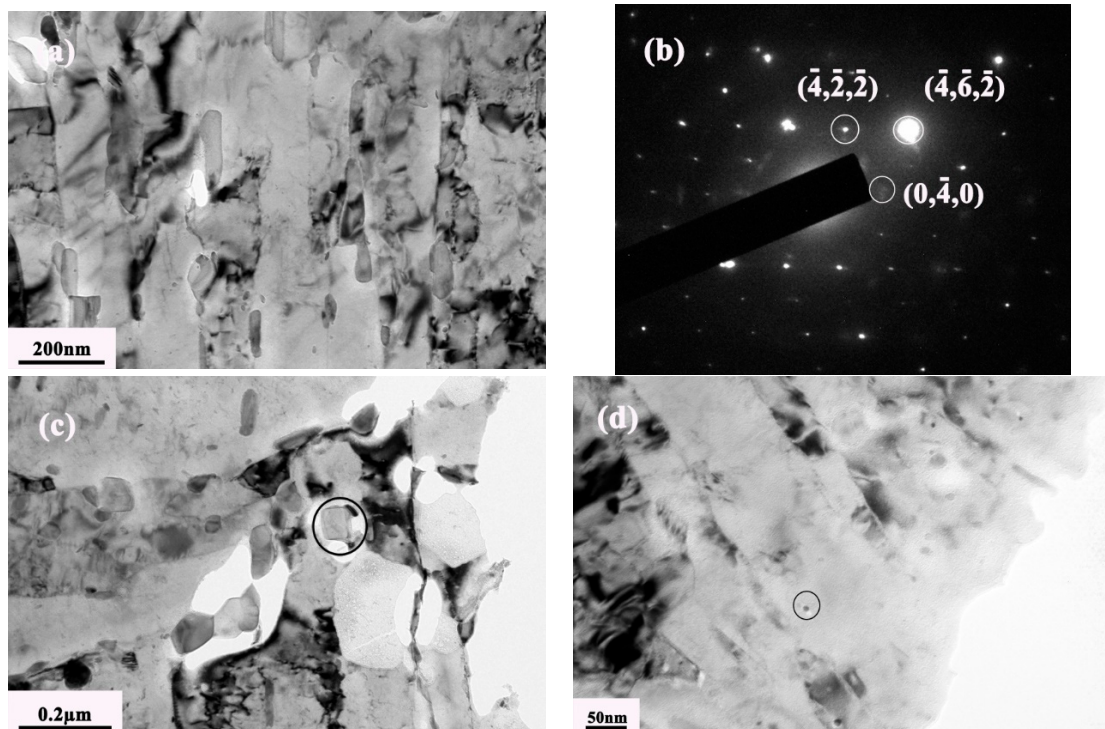


Figure 7. (a,c,d) TEM micrographs of the specimen after tempering at 600 °C for 100 h; (b) corresponding SAD pattern of $M_{23}C_6$ particles.

Stage 3 (tempered for 5 h): part of the M_2C dissolved into the matrix and then transformed into M_7C_3 particles. The M_2C in the experimental steel was a meta-stable transition phase. Previous studies have found that M_7C_3 nucleates on M_2C and grows inward into the M_2C through an in situ nucleation mechanism during tempering at 600 °C [31].

Stage 4 (tempered for 100 h): Hou et al. [32] found that the preferential nucleation of M_7C_3 occurred at dislocations and sub-grain boundaries, while $M_{23}C_6$ nucleated on grain and twin boundaries. As the tempering time was prolonged, the dislocation density decreased gradually. The recombination of the sub-grain boundaries caused a decrease in the sub-grain boundaries [33]. Thus, the number of M_7C_3 nuclei decreased and the $M_{23}C_6$ coarsened progressively with the elimination of the M_7C_3 particles. This indicates that the M_7C_3 carbides transformed into $M_{23}C_6$ with the increase in the tempering time [34,35]. The nucleation-free energy of $M_{23}C_6$ and M_7C_3 is -1.82 and -10.43 kJ/mol, respectively [36]. The higher the nucleation-free energy of precipitates, the more stable they are. The nucleation-free energy of $M_{23}C_6$ and M_7C_3 demonstrates that the M_7C_3 precipitated first and then transformed into $M_{23}C_6$. Two kinds of MC particles were also observed in the samples tempered for 100 h. This indicates that the MC(I) and MC(II) precipitates were stable when tempering at 600 °C for different times. During the prolonged tempering process, the precipitates in the specimen consisted of M_3C , M_2C , $M_{23}C_6$, M_7C_3 , MC(I), and MC(II) precipitates. The transformation sequence of the other four types of carbides tempered at 600 °C for a long time can be summarized as follows: $M_3C \rightarrow M_2C \rightarrow M_7C_3 \rightarrow M_{23}C_6$.

To further investigate the evolution of the different types of carbides, their extraction from hydrochloric acid was studied by XRD. The XRD patterns of the carbide particles of the specimens tempered at 600 °C under different times are illustrated in Figure 8. M_3C and two types of MC were identified in the sample tempered for 10 min. With increasing the tempering time to 30 min, M_2C , $M_{23}C_6$, and two types of MC were positively identified. After tempering for 100 h at 600 °C, M_2C , M_7C_3 , $M_{23}C_6$, and two types of MC carbide were identified. The XRD analysis showed that the peak intensity of the extracted particles was much greater than that of the specimens with shorter tempering times. This means that the number of precipitates obviously increased with increasing the tempering time, and the main carbide particles were $M_{23}C_6$.

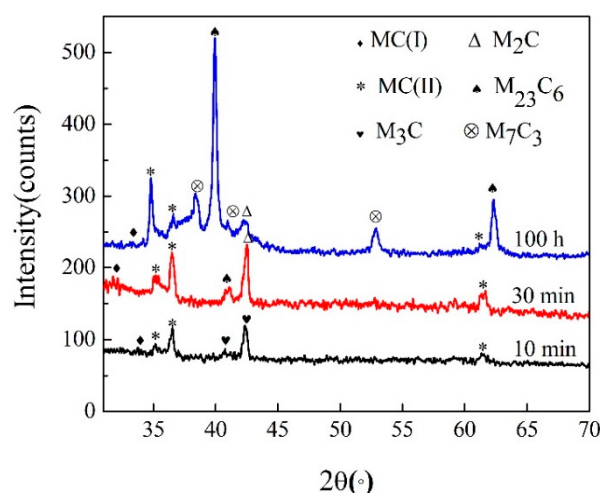


Figure 8. X-ray diffraction patterns of the carbides extracted from investigated steel after being tempered at 600 °C for different lengths of time.

The TEM images of the M_2C carbides after tempering at 600 °C are shown in Figure 9. It was confirmed that fine M_2C carbides were dispersedly distributed in the sample tempered for 30 min. The angle between the particles was about 60°, and the mean length of the M_2C carbides was about 59.04 nm. With increasing the tempering time to 1 h, the M_2C particles precipitated adequately, and the average length increased to 64.78 nm. The increase in the

average length of the M_2C carbides was attributed to the adequate diffusion of Mo atoms and their combination with carbon to form larger-scale M_2C . As the tempering time was prolonged to 10 h, part of the M_2C changed into M_7C_3 , and the average length reached a peak value of around 102.48 nm. For the experimental steel tempered for a long time (100 h) at 600 °C, the mean length decreased to 88.32 nm.

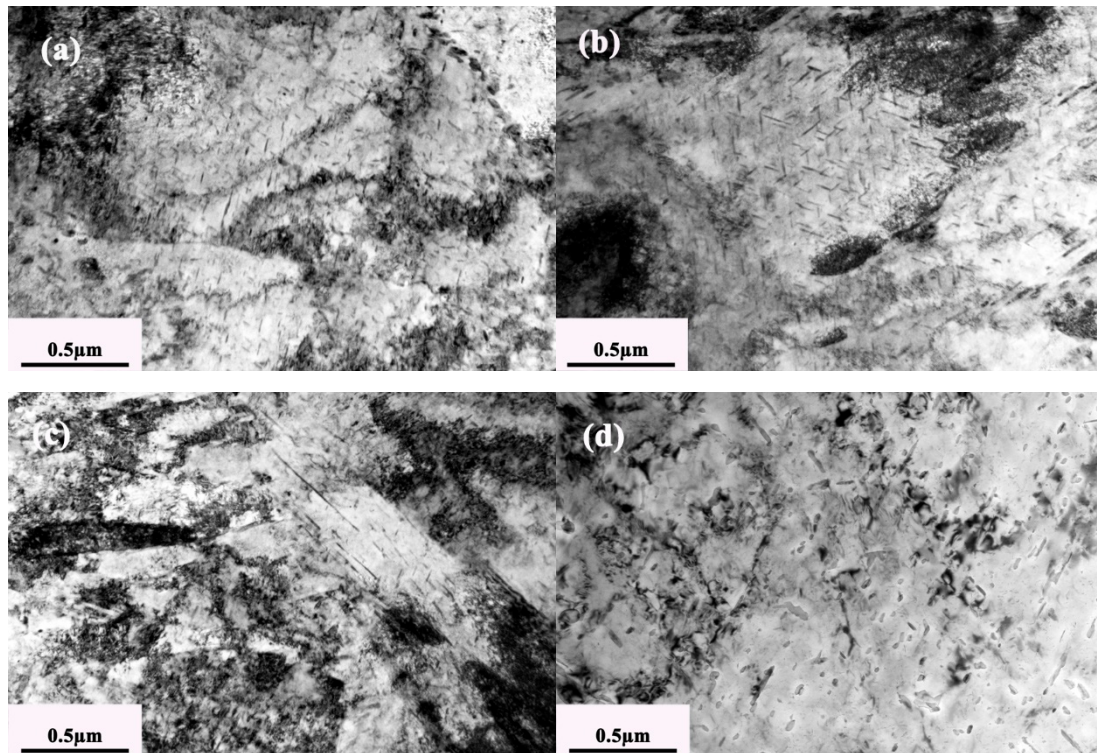


Figure 9. TEM micrographs of M_2C precipitates after tempering at 600 °C for different lengths of time: (a) 30 min; (b) 1 h; (c) 10 h; and (d) 100 h.

The mean length of the M_7C_3 particles and the average diameter of the $M_{23}C_6$ particles were also measured statistically. The variation in the mean size of the precipitates with the tempering time is given in Figure 10. It can be seen that the mean diameter of the $M_{23}C_6$ particles increased gradually with the tempering time. The average length of the M_2C and M_7C_3 particles first increased and then decreased. The variation in the size of carbides can be explained based on coarsening and evolution of the precipitates. The isothermal coarsening equation considering a constant volume fraction of carbides can be expressed as [37]:

$$d^3 - d_0^3 = kt \quad (1)$$

where d and d_0 are the mean diameter of the carbides at different tempering times, k is a constant, and t is the tempering time. Thus, the size of the $M_{23}C_6$ carbides increased with the tempering time. In addition, the decreased length of the M_2C and M_7C_3 particles at longer tempering times was correlated with the evolution of the carbide. Since relatively longer M_2C and M_7C_3 particles are apt to transform into M_7C_3 and $M_{23}C_6$, the sizes of the untransformed M_2C and M_7C_3 carbides were relatively small. With a prolonged tempering time, the amount of transformed carbides increased gradually. Therefore, the mean length of the M_2C and M_7C_3 particles decreased when the tempering time was extended to 10 h and 50 h, respectively.

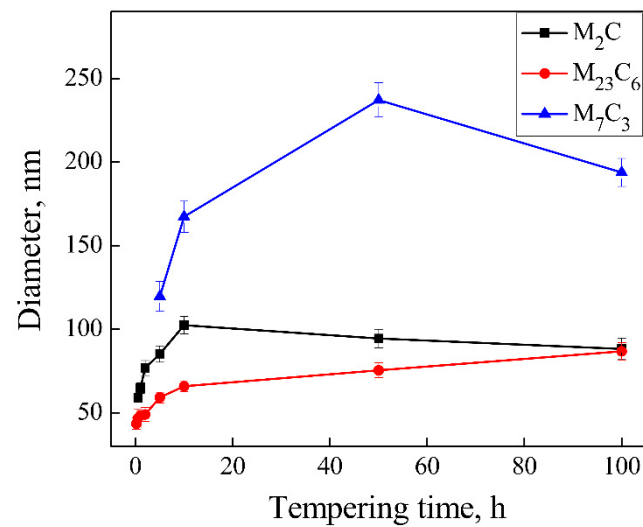


Figure 10. Mean size of precipitates as a function of time during tempering at 600 °C.

3.3. Mechanical Properties under Different Tempering Times

The stress–strain curve of the explored steel under different tempering times is demonstrated in Figure 11. The tensile property data can be seen in Table 1, including the YS, UTS, and total elongation (TE). With the increase in the tempering time, the YS and UTS decreased gradually. The UTS decreased from 1231 to 896 MPa, and the YS decreased from 1138 to 835 MPa.

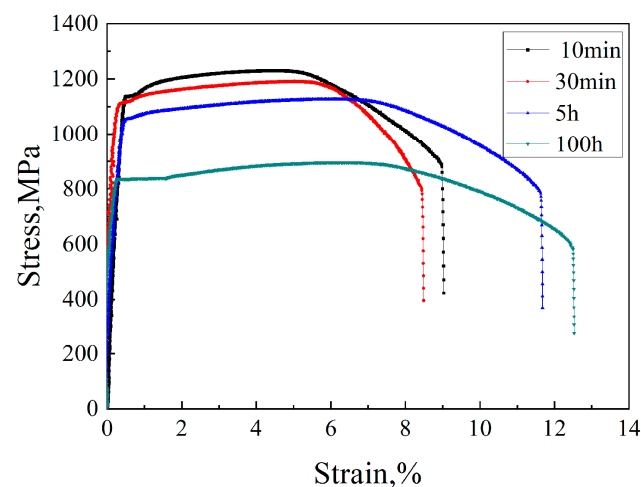


Figure 11. The stress–strain curves of the investigated steel during tempering at 600 °C.

Table 1. Tensile properties of the investigated steel during tempering at 600 °C.

Tempering Time	YS, MPa	UTS, MPa	TE, %
10 min	1138 ± 16	1231 ± 20	8.8 ± 0.3
30 min	1112 ± 12	1193 ± 18	8.4 ± 0.2
5 h	1055 ± 14	1132 ± 21	11.5 ± 0.5
100 h	835 ± 11	896 ± 15	12.3 ± 0.6

Zhang et al. [38] showed that the minimum structural unit that could effectively control the strength of 25CrMo48V micro-alloyed steel was the width of the martensitic

lath. The relationship between the width of the martensitic lath and the strength conforms to the Langford–Cohen formula:

$$\sigma_y = \sigma_0 + k_y d^{-1} \quad (2)$$

where k_y is the structural constant and d is the average lath width. In the Langford–Cohen model, the strength of the material is directly proportional to the reciprocal of the lath width.

With the prolongation of the tempering time, the width of the martensite lath increased and the dislocation decreased gradually. The recovery phenomenon was obviously enhanced, which resulted in matrix softening. Matrix softening was the main reason for the strength reduction. It is generally accepted that σ_0 is mainly composed of Peierls stress, precipitation strengthening, and solution strengthening [39,40]. That is to say, the strength of the sample was determined not only by the martensitic lath width and dislocation density but also by precipitation strengthening and solution strengthening. After tempering for 100 h, the tensile strength was about 900 MPa. The high strength was attributed to the precipitation strengthening from the nano-sized Nb-rich MC (I) particles. According to the Abshy–Oroman mechanism, nano-size precipitates with a size of less than 10 nm can provide about 200 MPa [41].

The $-40\text{ }^{\circ}\text{C}$ Charpy impact property of the specimens tempered under different times is shown in Figure 12. It was concluded that the Charpy impact toughness increased gradually with the increase in the tempering time. When the tempering time was extended from 10 min to 100 h, the Charpy impact toughness increased from 20 to 61 J.

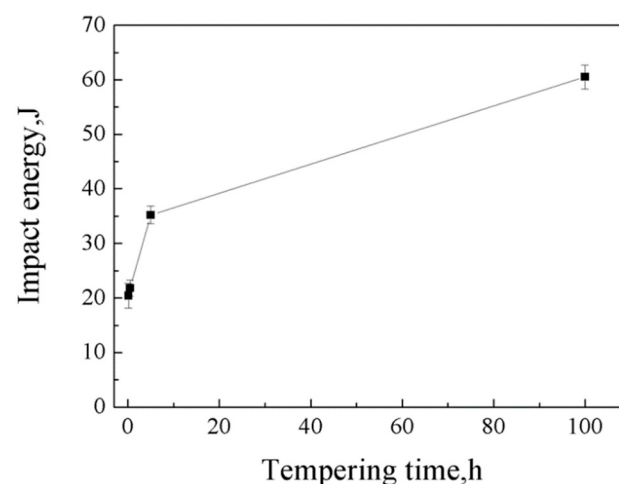


Figure 12. Change in the Charpy impact properties in investigated steel during tempering at 600 °C.

This phenomenon mainly depended on the following factors. One was matrix softening. With the increase in the tempering time, the recovery of the matrix was considerable, and the dislocation density decreased gradually. Furthermore, the increase in the number of precipitates meant that the interstitial atoms dissolved in the matrix precipitated gradually, and the supersaturation decreased, resulting in the softening of the metal matrix [42]. The other was the fact that some small-angle lath boundaries evolved gradually into large-angle boundaries. Several studies have indicated [43] that when cleavage cracks attempt to propagate through a large-angle boundary, the crystal orientation and crack direction may change, and then the growth of cracks may eventually be stopped. Consequently, an increasing amount of large-angle boundaries would lead to a larger deflection of cleavage cracks, and a higher toughness could be obtained.

The correlation between the micro-hardness and tempering time is depicted in Figure 13. The Vickers hardness first increased rapidly to the peak value when the tempering time was 1 h, and it then reduced remarkably after tempering for 5 h. Finally, the hardness decreased monotonically with increasing the tempering time to 100 h. This trend

was due to two competitive mechanisms that occurred during the tempering process. The strengthening was due to the precipitation of the carbides, and the softening was due to the recovery of the martensitic matrix. As described above, when the tempering time was between 10 min and 1 h, the hardness increased steadily due to the transformation from M_3C to M_2C . The precipitation of M_2C had a strong secondary hardening effect on the matrix, which results in the increasing hardness during tempering for 1 h. Then, the hardness decreased significantly when the tempering time was 5 h, since most of the M_2C transformed into long-line M_7C_3 . The secondary hardening effect was weakened, and precipitate particles were gradually coarsened, which had a detrimental impact on the Vickers hardness. With the increase in the tempering time, the coarsening of the carbides resulted in a reduction in the dislocation pile-up effect [44]. Martinez-de-Guerenu et al. [45] concluded that the recovery percentage of steels during tempering could be estimated by the following equation:

$$1 - R_y = b - a \ln t \quad (3)$$

where R_y is the recovery percentage, which is related to the dislocation density variation, t is the tempering time, and a and b are constants, which are related to the tempering temperature. During the continuous tempering, the recovery of dislocations in the matrix changed with the prolonging of the tempering time, which led to a gradual decrease in the Vickers hardness. It can be concluded that the variation in the Vickers hardness should be derived from the evolution and coarsening of the precipitate and the recovery of the lath martensite.

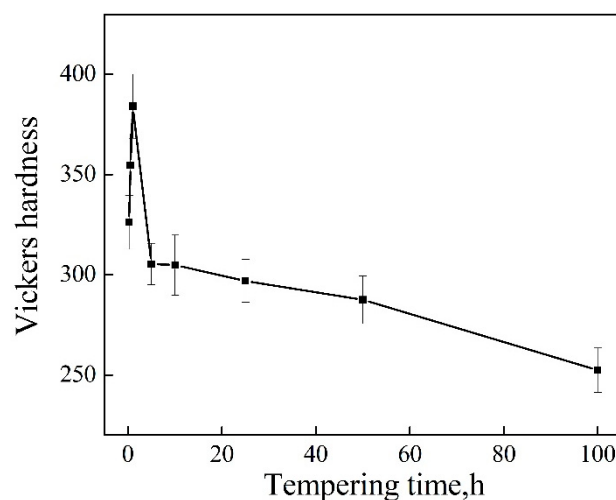


Figure 13. Changes in the Vickers hardness during tempering at 600 °C.

4. Conclusions

The evolution behaviors of the microstructure and precipitates and their effect on the mechanical properties of Nb-V-Ti micro-alloyed steel under different tempering times were explored, and the main conclusions obtained are shown as follows:

1. The width of martensite laths increased and the retained austenite and dislocation decreased gradually with the prolongation of the tempering time.
2. The evolution sequence of the carbides during tempering at 600 °C for the different times was identified as: $M_3C \rightarrow M_2C \rightarrow M_7C_3 \rightarrow M_{23}C_6$. The other two kinds of MC carbides remained stable during the tempering process.
3. The strength decreased and the Charpy impact toughness increased gradually with the prolongation of the tempering time. The Vickers hardness increased remarkably as the tempering time was extended to 1 h and then decreased sharply with a further increase in the tempering time up to 5 h. When the tempering time was between 5 and 100 h, the Vickers hardness values decreased gradually.

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