Study on Direct Quenching and Tempering Process of 1000MPa Grade Low Alloy High Strength Steel

Peng Gao1,2, *, Ye Gao2, Jun Chen2, Zhenyu Liu2, Yue Zhuo2, Guodong Sun1

1School of Mechanical and Materials Engineering, Jiujiang University, Jiujiang 332005, China
2The State Key Laboratory of Rolling and Automation, Northeastern University, Shenyang 110819, China
3Library, Jiujiang University, Jiujiang 332005, China

*Corresponding author e-mail: 15781807@qq.com

Abstract. The direct quenching and tempering process of 1000MPa low alloy high strength steel had been studied in this paper. When the tempering temperatures were between 550℃ and 610℃ after direct quenching, carbides began to precipitate in the test steel, and the main microstructure were tempered martensite. When tempering temperatures were between 640℃ and 670℃, the recovery stage of martensite lath basically ended and recrystallization began. The precipitated carbides grew up and the microstructure were mainly tempered sorbite. The results showed that the optimum tempering temperature range of the test steel was 610℃~640 ℃. The yield strength and tensile strength of the test steel were greater than 1000MPa, and the comprehensive properties of the test steel were better. It provided guidance for the production of 1000MPa high strength steel by direct quenching and tempering process in industry.

1. Introduction
Because of its high strength, good toughness and weldability, low alloy high strength steel is widely used in equipment manufacturing, construction and other fields [1,2]. In order to produce steel sheets with excellent comprehensive properties, controlled rolling and cooling were adopted by more and more steel mills, and quenching and tempering processes were combined to produce high strength steel [3]. In the production of high strength steel, micro alloy composition design was generally used, and a new generation of ultra-fast cooling technology was used to refine the metallographic structure and improve the strength and toughness of steel plate. Direct quenching technology is to use a new generation of ultra-fast cooling technology to control the cooling speed and lower final cooling temperature after rolling, ferrite and pearlite transformation zone are avoided, and bainite or martensite transformation zone are directly reached. Final microstructures are refined bainite or martensite to improve the strength and toughness of steel plate [4,5].

Compared with off-line quenching process, direct quenching process does not need reheating, but uses the residual heat after rolling to quench on-line. The process flow is effectively shortened and the cost is reduced. In view of the advantages of direct quenching process, the production of high strength steel by direct quenching and tempering process were studied by more and more scholars at home and
abroad, and more research results were obtained [6-10]. Therefore, it is necessary to study the direct quenching process and combine tempering process to produce high strength steel with good performance and low cost.

The rapid cooling speed of direct quenching may cause thermal stress and structural stress in the steel plate, and ultimately lead to residual stress in the steel plate. In order to reduce the residual stress, the steel plate directly quenched needs to be tempered. Tempering temperature has a great influence on the structure and mechanical properties of steel plate in tempering process. Therefore, it is important to study the effect of different tempering temperatures on the microstructure and mechanical properties [11-13]. In this paper, a two-stage controlled rolling process for 1000MPa high strength steel was adopted in the laboratory. Combining on-line quenching and off-line tempering, the microstructure and mechanical properties of high strength steel at different tempering temperatures were studied. It provides theoretical guidance for industrialized production of on-line quenching and off-line tempering of high strength steel.

2. Experimental Methods

The experimental slab was taken from a continuous casting slab produced by a medium and heavy plate factory in China. The chemical composition of continuous casting billet was shown in Table 1. In the experiment, the billet was heated to 1200 ℃, and was preserved heat for 2 hours, then controlled rolling was carried out in two stages. The final thickness of the product was 12 mm. The first stage was rolling in recrystallization zone. The rolling temperature was 1150 ℃-1000 ℃. After one-stage rolling, the intermediate billet temperature was reduced to 950 ℃, and then two-stage rolling was carried out. The final rolling temperature was 850 ℃, and the rolling reduction of final rolling passes was more than 60%. After rolling, on-line quenching was carried out by using ultra-fast cooling, and then off-line tempering were carried out at 550, 580, 610, 640 and 670 ℃. The tempering holding time was 30 minutes. The process of direct quenching and tempering was shown in Fig. 1.

| Table 1. Chemical composition of billet (mass fraction, %) |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| C | Si | Mn | Ni | Ti | Cr | Mo | V | B | Nb |
| 0.17 | 0.22 | 1.24 | 0.296 | 0.015 | 0.393 | 0.436 | 0.038 | 0.0015 | 0.022 |

Figure 1. Direct quenching and tempering process

After grinding and polishing, the quenched and tempered specimens were corroded by 4% nitric acid and alcohol solution, and then the microstructures of the specimens were observed by optical microscopy.
Because of the size limitation of the experimental billet, the size of the finished steel plate was smaller. In order to facilitate processing, the tempered specimens were processed into standard tensile specimens with a growth of 80 mm and an effective diameter of 5 mm. The V-notched impact specimens were processed into 10*7.5*55mm. Tensile and impact tests were carried out respectively. The impact test temperature was -20 °C. Microstructure morphology and impact fracture after tempering were observed by scanning electron microscopy. The TEM specimens were cut from tempered specimens by wire cutting. The thickness of the specimens was 0.5 mm. Then the specimens were thinned to less than 45 microns with abrasive paper. After that, the circular thin sheets with a diameter of 3 mm were made by a punch. Finally, the specimens were thinned by double jet electrolysis. After the transmission samples were made, the morphology and precipitates were observed by transmission electron microscopy.

3. Experimental Results and Analysis

3.1. Microstructure and Precipitate Analysis

The metallographic structure of the samples before tempering and after tempering at 550, 580, 610, 640 and 670 °C were shown in Fig. 2. It could be seen from Fig. 2 (a) that the microstructure of steel plate before tempering was mainly composed of lath martensite. After two-stage rolling, the original austenite grains were flattened and distributed in bands. Each group of slab bundles had different orientations, showing a large angle grain boundary distribution. Within the same slab bundle, the distribution of grain boundaries showed a small angle. After tempering at 550 °C, the martensite lath began to recover, and carbides precipitated along grain boundaries and lath bundles, which made the grain boundaries more easily corroded and difficult to identify. At this time, the main microstructure was tempered martensite. After tempering at 580~610 °C, the recovery continued, martensite lathes merged gradually and the number of lathes decreased. Carbides precipitated at grain boundaries and in grains, and the amount of precipitation increased. At this time, tempered martensite was still the main structure. After tempering at 640 °C, the recovery stage basically ended and began to enter the recrystallization stage. At this time, a small amount of tempered sorbite appeared in the structure. With the tempering temperature rising to 670 °C, the diffusion of carbon was further accelerated, most of the slabs had recrystallized, the martensite slabs and the original austenite grain boundaries disappeared, and new grain boundaries were formed. At this time, the structure was mainly tempered sorbite.

![Figure 2. Microstructure of test steel](image-url)
Microstructure and appearance of the test steel after tempering were shown in Fig. 3. It could be seen from Fig.3(a) that the original austenite grain boundaries were clearly visible after tempering at 550°C. A large number of fine carbides were precipitated on the austenite grain boundaries and martensite lath bundles. The structure of the tested steel was tempered martensite. It could be seen from Fig.3(b) that after tempering at 610°C, martensite laths began to recover, some martensite lathes began to merge and widen, carbides began to precipitate and grow in the crystals, and the structure was still mainly tempered martensite. It could be seen from Fig.3(c), after tempering at 670°C, most martensite laths ended their recovery stage and completed recrystallization. A large number of carbides precipitated and grew at grain boundaries and in grains, and the structure was mainly tempered sorbite.

![Figure 3. Microstructure of experimental steel observed under scanning electron microscope](image)

The scanning morphology of impact fracture of tempered steel were shown in Fig.4. Fig.4(a) showed that after tempering at 550°C, dimples of different sizes were distributed in the impact fracture. The dimples were shallow, there were more small dimples, fewer large dimples and surrounded by small dimples. There were cleavage steps and cleavage surfaces on the fracture surface, and there were a few river patterns on the cleavage surface. From Fig.4(b), it could be seen that after tempering at 610°C, the large dimples of impact fracture decreased, the small dimples increased, and there were small dimples in the large dimples. There were river patterns on the fracture surface, and the cleavage step rised. On the cleavage surface, there were steps formed by river patterns crossing. After tempering at 670°C, the small dimples of impact fracture increased, and the dimples were deeper and evenly distributed. There were still cleavage steps at the fracture surface. The cleavage surface was uneven and had a white tearing edge. It showed that plasticity was good and had a certain ability to resist crack propagation [14,15].

![Figure 4. Impact fracture morphology observed by scanning electron microscope](image)
The TEM structure and precipitate morphology of the tempered steel were shown in Fig.5. It could be seen from Fig.5(a), after tempering at 550°C, martensite lath began to widen, and the width of lath was about 300nm. Dislocations began to migrate and gradually decreased. A small amount of carbide precipitated on the martensite lath, and the structure of the test steel was tempered martensite. From Fig.5(b), it could be seen that after tempering at 610°C, the lath began to recover, some martensite lathes merged and widen, and the lath interface began to disappear. The dislocations were greatly reduced, fine carbides were precipitated on the martensite lath, and the structure of the test steel was tempered martensite.

It could be seen from Fig.5(c) that the martensite lath began to recrystallize after tempering at 670°C. After decomposition of martensite lath, there was no supersaturated carbon, and the structure changed from unsteady state to steady state, forming tempered sorbite structure. The dislocation density further decreased and the carbides gradually grew up.

3.2. Mechanical Properties Analysis

After direct quenching and tempering at different temperatures, the yield strength, tensile strength and elongation of the tested steel were shown in Fig.6. From Fig.6, it could be seen that the yield strength and tensile strength of the tested steel increased first and then decreased with the increase of tempering temperature.
Direct quenching could make alloy elements in solid solution state, increased hardenability of steel, and increased precipitation strengthening effect with the increase of carbide precipitation amount [16]. When tempering temperature increased from 550°C to 580°C, the yield strength and tensile strength gradually increased. This was because there were two effects in the tempering process. Firstly, with the increase of tempering temperature, dislocation interaction occurred, and some dislocations merged and disappeared, leading to the gradual decrease of dislocation density. The strength of matrix decreased as the martensite lath began to recover. Secondly, after tempering, the alloying elements redistributed, and the precipitation and growth of alloy carbides could be achieved by nucleation and growth alone. A large number of dispersed carbides played a role in precipitation strengthening. As the precipitation of alloy carbides such as Mo, V, Cr and Nb occurred in the test steel, and the effect of precipitation strengthening on the strength increase was greater than that of the strength decreased caused by the decrease of dislocation density, the yield strength and tensile strength gradually increased. In addition, the precipitation of carbides played a role in fixing dislocations, making dislocation migration difficult, and also played a role in increasing strength.

When tempering temperature increased from 580°C to 670°C, the martensite lath further recovered and recrystallized, and the lath gradually softened and formed tempered sorbite. Dislocation density decreased sharply, carbide growth weakened precipitation strengthening effect, precipitation strengthening effect was not enough to compensate for the effect of strength reduction caused by dislocation reduction, and ultimately the yield strength and tensile strength of test steel began to decline. Because the test steel had Mo, V, Cr, Nb and other strong carbide micro-alloying elements, these alloying elements maintained solid solution state after on-line quenching, which made the test steel have higher tempering stability, some of the structures still maintained lath shape, and still had higher yield strength and tensile strength.

The elongation of test steel was consistent with the softening trend of martensite lath, that was, softening was slower at the initial tempering stage and faster at the later tempering stage. With the increase of tempering temperature, the elongation of test steel increased first, then decreased, and then increased. The elongation value ranged from 17% to 19%.

The plastic impact work and section shrinkage of the test steel at -20°C were shown in Fig.7. The -20°C plastic impact energy increased slowly with the increase of tempering temperature, and increased rapidly until the tempering temperature reached 640°C. The recovery and recrystallization of martensite lath began to occur, the matrix began to soften gradually, and the plastic impact energy began to increase. When tempering temperature was higher than 640°C, most martensite slabs recrystallized to form tempered sorbite, which increased the softening degree of test steel matrix. Most martensite slabs had been decomposed and the plastic impact energy increased rapidly. Due to the size limitation of steel plate, the sample processed in this experiment was an auxiliary impact sample, and the standard value of impact energy after conversion was 25.5J at -20°C. The range of plastic impact energy in this experiment was 36~45J. Although it met the requirements, the surplus was small, which was due to the large residual stress caused by the uneven cooling of direct quenching.
Figure 7. -20°C impact work and section shrinkage at different tempering temperatures

Because martensite slab softened slowly at the initial stage of tempering and quickly at the later stage of tempering, the change trend of section shrinkage of test steel was basically consistent with the softening law of martensite slab, and its value was between 33% and 45%.

4. Conclusion
(1) When tempered between 550°C and 610°C, martensite lathes began to recover and carbides precipitated, and tempered martensite was the main structure. When tempered between 640°C and 670°C, the martensite lath began to recrystallize and the carbides grew up gradually. The structure obtained was mainly tempered sorbite.

(2) In the early stage of tempering, the precipitation strengthening effect was greater than the softening effect of martensite matrix, so the yield strength and tensile strength first increased and then decreased. The plastic impact energy of test steel ranged from 36 to 45J, which met the requirements of corresponding standards. The elongation of test steel ranged from 17% to 19%. The value of section shrinkage was between 33% and 45%.

(3) The optimum tempering temperature of direct quenching and tempering process was 610°C –640°C. It provided theoretical guidance for the production of 1000MPa high strength steel by direct quenching and tempering process in industry.

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