Effect of direct quenched and tempering temperature on the mechanical properties and microstructure of high strength steel

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Abstract

The effect of direct quenched (DQ) and tempering temperature on the microstructure and mechanical properties of high strength steel were studied by means of SEM, EBSD, TEM and mechanical properties test. The results showed that in DQ state, the tensile strength could reach 1420 Mpa, the yield strength could be 1050 Mpa, and the elongation was about 9.0%, impact energy at −20 °C was 59 J. High density entangled dislocation was distributed inside the lath and at the boundary. A small amount of Nb and Ti carbide was precipitated at the dislocation and lath boundary, and a small amount (about 2.15%) of residual austenite distributed between the lath. With the tempering temperature rising from 500 °C to 720 °C, the tensile strength of the experimental steel decreased from 1220 MPa to 840 MPa, the yield strength decreased from 1190 MPa to 780 MPa, the elongation increased from 10% to 13%, and the impact energy at −20 °C increased from 84 J to 153 J. When the tempering temperature rised from 500 °C to 640 °C, the structure was mainly composed of lath martensite and a large number of dislocations were still distributed inside the lath. The size of carbides precipitated inside and on the boundary of the lath. With the tempering temperature rising from 500 °C to 720 °C, the structure was mainly composed of lath martensite and a small amount of polygonal ferrite. There were still a large number of entangled dislocations inside and on the boundary of martensite. The carbide precipitate at the matrix boundary and dislocation line was obviously coarsening (70–80 nm). When tempered at 720 °C, the microstructure was mainly polygonal ferrite and the carbide precipitated at the matrix boundary and dislocation line significantly coarsened (about 100 nm). With the tempering temperature rising from 500 °C to 720 °C, the proportion of small-angle grain boundary was gradually decreased from 88.64% to 70.50%.

1. Introduction

The growing demand for more energy efficient solutions during the 21st century has led to increasing interest in the development of structural steel with higher strength made by energy efficient production processes [1].

As a part of Thermo Mechanical Control Process (TMCP), DQ refers to directly quench high temperature plate to a lower temperature after rolling via on-line quenching equipment [2] and is a new technology that has been applied to produce high-strength steel. Compared with conventional off-line reheating quenching, DQ takes advantages of good strength-toughness balance, reduction of energy consumption, shorter process and lower cost for manufacturing steel [2–4]. Therefore, many researchers have studied the development of high-performance steel plate by DQ process [5–8]. Most of that are concentrated in the laboratory. The method is still far from being used for production line to produce qualified products.
Table 1. Chemical composition of billet (wt%).

| C  | Si  | Mn  | P   | S   | Al  | Cr  | Nb  | V   | Mo  | Ti  | Fe  |
|----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 0.12 | 0.23 | 1.16 | 0.011 | 0.002 | 0.03 | 0.493 | 0.027 | 0.091 | 0.534 | 0.02 | bal. |

Table 2. The mechanical properties in DQ state.

| Yield strength (MPa) | Tensile strength (MPa) | Total elongation (%) | Charpy impact energy at $-20 \degree C$ (J) |
|----------------------|------------------------|----------------------|------------------------------------------|
| 1050                 | 1420                   | 9.0                  | 59                                       |

With the development of engineering machinery and coal mining machinery, there is more and more demand for high strength steel and wear-resistant steel of 960 MPa or above. The steel mainly used for concrete pump truck boom or dump truck box. Traditional processes of high strength steel were reheating, quenching and tempering [9, 10]. In order to save the cost of high-strength steel and improve the efficiency, this paper tries to produce a new high-strength steel with strength above 960 MPa by DQ and tempering (no reheating) in a production line of HBIS (not in laboratory). The influence of DQ and tempering temperature on the microstructure and performance of the new high-strength steel is studied in this paper. This will promote the development of high strength steel in steel mill.

2. Experimental materials and procedures

The chemical composition of billet is shown in table 1. The billet was processed in 3500 mm production line of HBIS hansteel including rolling, DQ and tempering.

The thickness of the billet was 260 mm, and was homogenized at 1200 \degree C for 4.5 h. Then the billet was rolled to 70 mm thick slab with the start temperature of 1100 \degree C and the finish temperature of 1000 \degree C in the first stage; the 70 mm thick slab was rolled to 20 mm thick plate with the start temperature of 900 \degree C and the finish temperature of about 820 \degree C in the second stage, and the plate was direct quenched from 760 \degree C to 200 \degree C by the cooling equipment equipped behind the mill, and the average cooling rate was about 20 \degree C s$^{-1}$. To study the effect of tempering on microstructure and mechanical properties, tempering treatment was performed at 500 \degree C, 600 \degree C, 640 \degree C, 680 \degree C and 720 \degree C respectively, and the tempering time was 90 min. Heat treatment samples with size 400 mm × 100 mm × 20 mm (length, width and thickness) were cut along the rolling direction of the plate.

Mechanical properties in terms of strength and elongation were tested by SHT5605P electro-hydraulic servo tension tester at 1 mm min$^{-1}$ and 25 \degree C, according to GB/T 228.1-2010, the gauge length of tensile samples is 50 mm and the diameter is 10 mm; mechanical properties of impact toughness were tested on the CBD-300 impact tester, and the size of impact sample is 10 × 10 × 100 mm (impact temperature was $-20 \degree C$, and the average value was taken from 3 impact values).

Samples were mechanically polished and then etched with 4% nital using metallographic procedures for SEM (FEI Quanta 600) to observe the microstructure. Samples prepared for EBSD were sectioned longitudinally, mechanically polished and electro-polished for 30 s at 20 V and 2 A in an electrolyte solution of 12.5/87.5 (v/v) perchloric acid and ethanol. The phase proportion and boundary misorientation were measured with Link Opal electron backscatter diffraction (EBSD, Oxford Instrument HKL, Channel 5 software) at an acceleration voltage of 20 kV with step size of 0.15 \mu m. TEM was carried out using JSM-2100 transmission electron microscopy. TEM foils were prepared by electro polishing 3 mm discs in 5% perchloric acid.

3. Experimental results and analysis

3.1. Mechanical properties and microstructure in DQ state

The mechanical properties of DQ stage are presented in table 2.

Figure 1 shows that lath martensite was obtained by DQ. And it also can be seen from figure 1 that the primary austenite grains were deformed by controlled rolling which refined the effective grain size. The width of martensite lath in DQ state is about 100–200 nm, and a large number of entangled dislocations distribute inside and at the boundary of martensite, as shown in figures 2(a) and (b).

Figure 2 shows that dislocations are distributed inside and at the boundary of martensite lath. As the density of dislocations is high inside the lath, dislocation strengthening effect is produced. In addition, C and...
other alloying elements could be retained in the matrix, resulting in solid solution strengthening effect. The results of literature [13] showed that the lath width is an important factor that affects tensile strength. Therefore, the dislocation strengthening, solid solution strengthening and the strengthening effect of fine lath made the tensile strength reach 1420 MPa and yield strength up to 1050 MPa.

In DQ state after rolling, a small amount of austenite retained in the matrix (shown in figure 3), which could be helpful to relax the boundary stress and the stress concentration at crack tip. So the appearance and the propagation of the crack could be prevented and the Charpy impact energy of the martensite steel could be increased [14]. The charpy impact energy at $-20^\circ C$ of DQ state could be as high as 59 J.

3.2. Effects of tempering temperature on mechanical properties and microstructure

Figure 4 showed the mechanical properties results as a function of tempering temperature that raised from 500 °C to 720 °C. When the tempering temperature raised from 500 °C to 640 °C, the strength of the experimental steel decreased slightly. The tensile strength decreased from 1220 MPa to 1170 MPa, and the yield strength decreased from 1190 MPa to 1160 MPa. It showed that the strength of DQ steel is very stable when tempered below 640 °C. When the tempering temperature is above 680 °C, the strength decreased significantly. The tensile strength decreased from 1030 MPa to 840 MPa, and the yield decreased sharply from 1020 MPa to 780 MPa.

With the increase of tempering temperature from 500 °C to 720 °C, impact energy and plasticity showed a trend of gradual increase. The impact energy at $-20^\circ C$ increased from 84 J to 108 J, and the elongation increased from 10% to 12.5%.

SEM micrographs of the tempered steels were shown in figure 5. With the tempering temperature rising from 500 °C to 640 °C (figures 5(a)–(c)), the matrix was lath martensite, and the boundary of the lath was gradually blurred, showing a tendency of merging and growing. When tempered at 680 °C, there were both lath
martensite and polygonal ferrite in the matrix (figure 5(d)), while the matrix was mainly polygonal ferrite (figure 5(e)) when tempered at 720 °C.

TEM results of the tempered steels were shown in figure 6. When tempered at 600 °C and 640 °C, the width of the lath increased significantly compared with the DQ state, which is about 200–300 nm, and the martensite boundary began to blur (figures 6(b), (d)). There were still high-density entangled dislocations inside and at the boundaries of the lath; some tiny precipitates precipitate in the matrix and dislocation lines (figure 6(b), (d)), with the size of about 20–30 nm. The evolution of microstructure shows that the martensite lath of DQ steel process a very high tempering stability.

When tempered at 680 °C, the microstructure was composed of martensite (figure 6(e)) and polygonal ferrite (figure 6(f)) and the width of lath martensite was about 200–400 nm, which means that partial martensite lath began to recrystallize. The coarsened precipitates with size of 75–150 nm could be found inside and at the boundary of polygonal ferrite.

When tempered at 720 °C, the microstructure was mainly polygonal ferrite (figure 6(g)), and the size of precipitates increased significantly to 100–150 nm(figure 6(h)), which means that almost all the martensite lath has recrystallized.

Results of grain boundary misorientation of the different tempering temperature were shown in figure 7, the green line was the grain boundary with its grain misorientation angle less than 15°, and the black line was the grain boundary with its grain misorientation angle greater than 15°. The statistics of frequency of the low misorientation angle at different tempering temperatures were shown in table 3.
According to the data in table 3 and figure 7, with the tempering temperature rising from 500 °C to 720 °C, the proportion of small-angle grain boundary decreased from 88.64% to 70.50%. For martensite and tempered martensite, lath boundary and subblock boundary are the grain boundary with small misorientation angle [15, 16]. The proportion of lath boundary and subblock boundary decreases with the tempering temperature increasing from 500 °C to 680 °C and that will influence the creep strength [17, 18]. And when tempered at 720 °C, the microstructure was mainly ferrite with a typical large-angle grain boundary, and there was no substructure with a small misorientation angle boundary in the polygonal structure.

With the increase of tempering temperature from 500 °C to 680 °C, the boundary of lath martensite gradually blurred and fused, which increased the width of lath to 200–400 nm (tempered at 680 °C), the precipitates gradually polymerized and grew.

Studies in literature [13] showed that the width of martensite block was one of the main factors affecting the tensile strength of martensite steel. Literature [19] showed that the segregation of carbon atoms at the
martensitic grain boundary affected the tensile strength of the experimental steel. During the tempering process, martensite grain boundary was first affected [20], and precipitations were precipitated to reduce the content of partial poly (C) element at martensite grain boundary, thus reducing the tensile strength of the steel. Therefore,
with the tempering temperature gradually rising from 500 °C to 680 °C, the amount of carbides precipitated at the boundary increased, and therefore decreased the segregation of carbon at the boundary. Thus the tensile strength reduced from 1220 MPa to 1030 MPa. When tempered at 720 °C, the microstructure was mainly polygonal ferrite, which significantly reduced the tensile strength to 840 MPa.

Table 3. Distribution of low misorientation angle at different tempering temperatures.

| Temperature °C | 500  | 600  | 640  | 680  | 720  |
|----------------|------|------|------|------|------|
| Frequency %    | 88.64| 83.83| 83.29| 79.16| 70.50|

Figure 7. Boundary map of the steel plates at different tempering temperature. (a) 500 °C; (b) 600 °C; (c) 640 °C; (d) 680 °C; (e) 720 °C.
Studies in literature [21] showed that the main factor which affects the strength is the proportion of grain boundary with small angle, and the trend of yield strength is the same as that of grain boundary proportion with small angle. So with the increase of tempering temperature from 500 °C to 720 °C, the yield strength decreases from 1190 MPa to 780 MPa.

In addition to dislocation strengthening, the strengthening effect of precipitates in the tempering process would also affect the yield strength. According to Orowan mechanism, precipitation strengthening could be expressed by the following formula [22]:

\[
\alpha_p = 8.995 \times 10^{5} \frac{f}{d^{0.5}} \ln (2.417d)
\]

where \( f \) represents the proportion of precipitates and \( d \) represents the size of precipitates.

So the strengthening effect would decrease with the increasing of the grain size and the decreasing of proportion of the precipitated phase.

The secondary cleavage cracks could turn sharply at or near the block boundary with big misorientation which change the main propagation direction of the crack in the lath packet, inhibit the propagation of cleavage crack and increase the energy dissipation. The path of crack propagation influences the toughness of steel. materials with a smaller grain size and larger amount of grain boundaries tend to have a higher toughness and a higher impact energy [23, 24]. Therefore, with the increase of tempering temperature, while the proportion of large misorientation angle grain boundaries increased, and its impact energy also increased from 84 J to 108 J.

4. Conclusion

The effect of tempering temperature on the mechanical properties and microstructure of direct quenched Q960 steel has been studied, and the conclusions are as followed:

(1) In the DQ state, the microstructure is composed of fine lath martensite (lath width is 100–200 nm), entangled dislocations are distributed inside and at the boundary of the lath. The tensile strength of the experimental steel could reach 1420 Mpa, the yield strength could be 1050 Mpa, and the elongation is about 9.0%, impact energy at −20 °C is 59 J;

(2) With the increase of tempering temperature from 500 °C to 640 °C, the tensile strength decreased from 1220 MPa to 1170 MPa, and the yield strength decreased from 1190 MPa to 1160 MPa, indicating that the strength of the DQ steel is very stable when tempered below 640 °C. When the tempering temperature continues to rise from 680 °C to 720 °C, the tensile strength further decreases from 1030 MPa to 840 MPa, and the yield strength further decreases from 1020 MPa to 780 MPa. With the increase of tempering temperature from 500 °C to 720 °C, the elongation increases from 10% to 13%, and the impact work at −20 °C increases from 84 J to 153 J.

(3) With the increase of tempering temperature from 500 °C to 640 °C, the martensite lath gradually gets fused and wider, the width is about 200–300 nm when tempered at 640 °C, indicating that the martensite lath of DQ steel process very high tempering stability. When tempered at 680 °C, the width of the lath was further increased to 200–400 nm, some martensite lath began to recrystallize and some ferrite appeared accordingly; and all the martensite transformed into ferrite when tempered at 720 °C.

(4) The size of precipitation increases with the increase of tempering temperature, especially when tempered above 680 °C, the size of precipitation could reach about 75–150 nm.

(5) The proportion of grain boundary with small angle gradually decreases from 88.64% to 70.50% with the increase of tempering temperature from 500 °C to 720 °C.

(6) When tempered at 680 °C, the mechanical properties of the experimental could meet the requirements of GB/T 16270–2009.

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