Effect of TMCP Parameters on the Microstructure and Properties of an Nb–Ti Microalloyed Steel

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Thermal mechanical control processing (TMCP), which includes combination of controlled rolling and controlled cooling, provides a powerful means of developing high-strength low alloy (HSLA) steels by intensive microstructural control. In the present investigation, the effects of TMCP parameters, consisting of the finish cooling temperature and the start rolling temperature in non-recrystallization region, on the final microstructure and mechanical properties of an Nb–Ti microalloyed steel has been studied by tensile, Charpy impact tests, optical microscopy and scanning electron microscopy. The TMCP parameters for Q460 grade steel have been optimized by laboratory experiments. The microstructure and properties of industrial product were coincident with the results of laboratory experiments.

KEY WORDS: TMCP; HSLA; mechanical properties; Nb–Ti microalloyed; Q460.

1. Introduction

High strength low alloy (HSLA) steels have demonstrated superior mechanical properties since the 1980s through controlled rolling and controlled cooling, which is often called as Thermo-Mechanical Controlled Processes (TMCP). The improvement in properties of HSLA steels is associated with different strengthening mechanism, the most important of which is grain refinement whereby both strength and toughness are improved at the same time. To refine ferrite grain, it is necessary to maximize the area of austenite grain boundary per unit volume at the on-set of phase transformation, which may be achieved by controlled rolling process. Accelerating cooling after hot rolling has currently been regarded as a further advanced thermomechanical treatment in hot rolling process, as affect the transition and the precipitation behavior of micro alloy elements. The final microstructure and mechanical properties depend strongly on the chemical composition, controlled rolling parameters and cooling conditions of the plate.

The present work is a part of the development of a new Q460 grade plate steel, whose yield strength is more than 460 MPa, and it focused on the investigation of the effects of TMCP parameters on the microstructure and properties.

The chemical composition of the new steels had been determined in earlier work, and the effect of it on microstructure and properties will not be given intensively in the present work. At last, the TMCP parameters of the new Q460 grade plate steel would be optimized for industrial manufacturing.

2. Experimental Procedure

Table 1 shows the required composition range and the actual chemical composition of the new Q460 grade Nb–Ti microalloyed plate steels, which were supplied by Jiuquan Iron & Steel Corp., China. The steel was melted in a 50 ton oxygen blown converter. The 220 mm thick slab was rolled into plate, with the thickness of 100 mm for pilot hot rolling experiments.

Historically, carbon is known as the most important chemical element for strengthening steel; however, it has detrimental effects on many technological properties such as weldability and formability. Therefore, physical metallurgists now recommend the substitution of carbon by other strengthening mechanisms. Micro alloyed and TMCP are main technologies for realizing the substitution. The loss of strength for reducing carbon will be compensated by precipitation strengthening and grain refined strengthening.

| C       | Mn     | Si     | S     | P    | Nb   | Ti   | Al<sub>eq</sub> |
|---------|--------|--------|-------|------|------|------|----------------|
| 0.12–0.16 | 1.10–1.50 | 0.20–0.50 | <0.02 | <0.02 | 0.015–0.022 | 0.005–0.01 | >0.01 |
| Actual composition | 0.15 | 1.47 | 0.30 | 0.014 | 0.013 | 0.019 | 0.008 | 0.015 |

Al<sub>eq</sub>: acid solution aluminum
The low carbon steel (often less than 0.10 wt%) possesses the lower ductility-brittleness transition temperature (DBTT) usually, for example, the steels for making pipeline. However, if the required properties of the steels are not high, considering saving the micro alloyed elements, the content of carbon should be limited an appropriate range. Nb and Ti are micro alloyed elements in the steel, carbides and/or carbonitrides are expected to be formed in the course of hot working. These second phase particles, especially Nb(C, N) and TiN, prevent grain growth of the austenite during reheating. In addition, Nb or Nb(C, N) will enhance recrystallization stop temperature, *i.e.* expand the unrecrystallization temperature range.

A 4000 KN pilot hot rolling mill with 450 mm diameter rollers and a mist cooling facility for simulating industrial process specially were employed in the study. The plate, with the size of 100 mm thick $\times$ 90 mm width $\times$ 140 mm length, was rolled to 16 mm thick sample steels by using CCR + IAC, *i.e.* conventional controlled rolling and interrupted accelerated cooling. IAC was characterized by allowing accelerated cooling in a given temperature region after finish rolling, followed by air-cooling.

**Figure 1** shows that the start rolling temperature of 1100°C for recrystallization austenite region was used, and 60% reduction was performed in the recrystallization region and 60% reduction was performed in the non-recrystallization region for the two schedules. In schedule I the start rolling temperature of non-recrystallization austenite region was fixed at 890°C, and then the finish rolling temperature was about 800°C. The finish cooling temperature was changed from 690 to 570°C by controlling the mistcooling section and air-cooling to room temperature. In schedule II, the start rolling temperature of non-recrystallization region was changed from 890 to 830°C, and the finish rolling temperature was controlled in a range of 780–800°C by adjusting the reduction and inter-pass time. The finish cooling temperature was fixed at $\sim$650°C and air-cooling to room temperature.

Tensile, impact testing, and optical microscopy were used in this study. The tensile direction of the tensile specimens was parallel to the rolling direction and the lengthwise of Charpy impact specimens was parallel to the rolling direction of the plates. Standard Charpy impact test was carried out at various temperatures on a standard Charpy impact-testing machine. Metallographic specimens were received in the middle of rolled samples with the rolled surface. The Leica imagine analysis and linear intercept technique after etching in 2% nital were used for grain size measurements.

3. **Experimental Results**

3.1. Effect of Finish Cooling Temperature on Structures and Properties

The effect of finish cooling temperature, shortened as $T_{fc}$, on structures and properties was studied by schedule I process, as in Fig. 1 shown.

3.1.1. Effect of $T_{fc}$ on the Microstructures

**Figure 2** shows microstructures of samples at four typical $T_{fc}$. When $T_{fc}$ was in 620–690°C, the microstructure of the samples were ferrite and pearlite, and the difference of ferrite grain size and the difference of the ferrite volume percentage were slight. When $T_{fc}$ was 620°C, acicular ferrite or called as widmänstäten ferrite could be found, as the Fig. 2(b) shows. When $T_{fc}$ was in 610–570°C, the microstructure was granular bainite with a small mount of ferrite and pearlite. With $T_{fc}$ decreasing, the granular bainite increased and ferrite and pearlite decreased.

### 3.1.2. Effect of $T_{fc}$ on Yield Strength and Tensile Strength

**Figure 3** shows the effect of $T_{fc}$ on yield strength and tensile strength. The yield strength and tensile strength increased greatly with $T_{fc}$ decreasing from 690 to 620°C, and increased greatly with $T_{fc}$ decreasing from 610 to 570°C. The yield and tensile strength values of all samples were more than 460 MPa and 560 MPa, respectively.

### 3.1.3. Effect of Finish Cooling Temperature on Elongation

**Figure 4** shows the effect of $T_{fc}$ on the elongation. The elongation decreased from 28 to 15% with $T_{fc}$ decreasing from 690 to 570°C.

### 3.1.4. Effect of Finish Cooling Temperature on Impact Toughness

**Figure 5** shows the impact toughness of the four samples at various test temperatures. It can be seen that the impact toughness decreased and the ductility-brittleness transition temperature (DBTT) significantly increased with the $T_{fc}$ decreasing.

3.2. **Effect of Controlled Rolling Parameters on Properties and Structures**

The effect of controlled rolling parameters on properties and structures was studied by schedule II process. In schedule II, although the start rolling temperatures were not same, the finish rolling temperatures were controlled in a narrow range of 780–800°C by adjusting inter-pass time and the reduction.

3.2.1. Effect of $T_{nr}$ on Microstructure

**Figure 6** shows the final microstructures at different $T_{nr}$, which all are polygonal ferrite and pearlite. The ferrite grain was refined from 10 to 6 μm as the $T_{nr}$ decreasing from 890 to 830°C, as shown in **Fig. 7**.

3.2.2. Effect of $T_{nr}$ on Yield Strength and Tensile Strength

**Figure 8** shows the effect of $T_{nr}$ on yield strength and tensile strength. The yield strength and tensile strength...
increased with the start rolling temperature decreasing. The tensile strength increased significantly, about 100 MPa. The yield strength increased slightly, only about 50 MPa.

Fig. 9 shows the effect of $T_{nr}$ on the yield ratio ($R_e/R_m$), here, $R_e$ means yield strength and $R_m$ means tensile strength. When $T_{nr}$ is lower than 860°C, the yield ratio decreased greatly.

3.2.3. Effect of $T_{nr}$ on the Elongation

Figure 10 shows the effect of $T_{nr}$ on the elongation. With $T_{nr}$ decreasing, elongation decreased significantly.

Furthermore, the impact toughness of the samples had been measured, but no remarkable regular relation between $T_{nr}$ and impact toughness, and the DBTT of all samples was lower than $-60^\circ$C.
Fig. 6. Microstructures of sample steels at different start rolling temperature, (a) $T_{nr} \sim 894^\circ C$, average grain size $\sim 10 \mu m$; (b) $T_{nr} \sim 885^\circ C$, average grain size $\sim 8 \mu m$; (c) $T_{nr} \sim 854^\circ C$, average grain size $\sim 6 \mu m$; (d) $T_{nr} \sim 830^\circ C$, average grain size $\sim 6 \mu m$.

Fig. 7. Effect of $T_{nr}$ on the ferrite grain size.

Fig. 8. Effect of $T_{nr}$ on the yield strength and tensile strength.

Fig. 9. Effect of $T_{nr}$ on the yield ratio.

Fig. 10. Effect of $T_{nr}$ on the elongation.
4. Discussion

4.1. Effect of IAC on Microstructures and Properties

The course of IAC process is a course of controlled overcooling austenite transition in fact. The finish cooling temperature is the most critical parameter of IAC process. This could be interpreted by a CCT scheme, as shown in Fig. 11. The transition zones in CCT scheme crossed by the cooling curve represent the transition product. With the temperature decreasing at a cooling rate of ~7°C/s, the transition zone will be of ferrite, pearlite and bainite orderly. When $T_{fc}$ is in the ferrite transition zone, as curve (a) and (b) in Fig. 11 shows, the product will be ferrite and pearlite, which microstructures are shown as Figs. 2(a) and 2(b). When $T_{fc}$ is in the bainite transition zone, as curve (c) and (d) in Fig. 11 shows, the product will be ferrite, pearlite and bainite, which microstructures are shown as Figs. 2(c) and 2(d).

With $T_{fc}$ decreasing from 690 to 620°C, the difference of transition product is slight, consisting of the ferrite grain size and pearlite volume percentage. However, the acicular ferrite or called as widmanstätten ferrite, whose strength properties are better than polygonal ferrite and elongation worse than polygonal ferrite, increased in the steels, as is shown in Figs. 2(a) and 2(b). Therefore, the yield strength and tensile strength increased slightly with $T_{fc}$ decreasing and the elongation decreased.

With $T_{fc}$ decreasing from 610 to 570°C, the granular bainite volume percentage and the M-A island increased greatly, as is shown in Figs. 2(c) and 2(d). The bainite possess the higher strength and the lower ductility. Therefore, the yield strength and tensile strength increased and the elongation decreased greatly.

The mechanical properties depend on the microstructures and the properties of the steel are the natures of the microstructures. The effect of $T_{fc}$ on properties is the effect on microstructures essentially. As Fig. 5 shows, the microstructures of sample (a) were polygonal ferrite and pearlite, so it achieved the higher impact toughness and the lower ductile–brittle transition temperature than the other samples with widmanstätten ferrite or granular bainite.

The microstructures are determined by the combination of all process parameters. The parameter of $T_{fc}$ affects the transition product or the final microstructure chiefly. However, when the final microstructures were polygonal ferrite and pearlite, the effect of $T_{fc}$ on grain size could be neglected in the present work.

From the experimental results, the parameter of $T_{fc}$ should be limited in a range of 640–680°C for the good combination of strength, ductility and elongating property and being up to standards of Q460 steel grade.

4.2. Effect of CCR on Microstructures and Properties

The CCR process contains two deformation stages, in recrystallization region and in non-recrystallization region, as Fig. 1 shows. In recrystallization region, coarse austenite grain is refined by repeated deformation and recrystallization, which produce the recrystallized grains. In non-recrystallization region, deformation bands are formed in elongated, non-recrystallized austenite. The lower the temperature of deformation is and the more the total reduction in non-recrystallization region is, the more the deformation bands are. During cooling, ferrite would nucleate on the deformation bands as well as α-grain boundaries, giving fine α-grain, as shown in Figs. 6 and 7.

Generally, the yield strength will enhance as lowering the finish rolling temperature and the yield ratio ($R_m/R_y$) will enhance. However, Figs. 8 and 9 had shown reverse trends. The yield ratio represent the potentiality of strain hardening, the lower the yield ratio is, the higher the strain-hardening potentiality is. The austenite, accumulating deformation in lower finish-rolling temperature, is apt to restore, but not to recrystallize. While the density of dislocation in restored austenite is higher than that in recrystallized austenite, the same with the transformed ferrite inherited from the austenite. Therefore, the strain-hardening potentiality of the ferrite from restored austenite is weaker than that from recrystallized austenite. In the present experiment of Schedule II, for keeping the finish-rolling temperature, the reduction and inter-pass time had been adjusted. Though the $T_{sr}$ is lowered to less than 860°C, the austenite grain was apt to recrystallize during the non-recrystallization region for the adjusted reduction. In addition, the austenite grain was refined by recrystallization, the same with the transformed ferrite. Therefore, the yield strength and tensile strength increased with the ferrite grain refining, when the $T_{sr}$ was lower than 860°C, as shown in Fig. 8. The yield ratio decreased for the low dislocation density of the ferrite from recrystallized austenite, as shown in Fig. 9.

As an industrial technology, TMCP requires selecting the proper process parameters to control. The finish rolling temperature is a parameter that easy to record, but not proper to control perfectly. In some cases, it can reflect the result of accumulated deformation, so it is related to the mechanical properties. However, when it cannot reflect the accumulated deformation result, it is not related to the properties. The start rolling temperature is a parameter that easy to control in industrial producing, and it can determine the temperature range of deformation in non-recrystallization region and affect the mechanical properties of the steel.

From the experimental results, the start rolling temperature in non-recrystallization had effect on the properties. For the good combination of properties and being up to standards of Q460 steel grade, the start rolling temperature should be controlled at 850°C. The deformation of non-recrystallization region should be limited in a temperature range of 780–850°C.
Industrial producing results

Industrial producing was carried out in Plate Mill of JISCO. The steel was melted in a 50 ton oxygen blown converter. The exact chemical composition of the new steels in industrial producing is shown as Table 2. The 220 mm thick ingots were rolled to 12 mm, 14 mm, 16 mm, 20 mm and 30 mm thick product respectively. CCR+IAC process was employed. Referencing the results of laboratory experiments, the start rolling temperature of 1100°C for recrystallization region and 850°C for non-recrystallization region were used. The total reduction of non-recrystallization was above 60%. Finish cooling temperature was controlled at a range of 640–680°C. Furthermore, the cooling rate of accelerated cooling was controlled at \( \sim 7°C/s \).

Tensile and impact testing, as well as optical and scanning electron microscopies were used in this study. The tensile direction of the tensile specimens was perpendicular to the rolling direction and the lengthwise of Charpy impact specimens were parallel to the rolling direction of the plates. Standard Charpy impact test was carried out at various temperatures on a standard Charpy impact-testing machine. Metallographic specimens were received in the middle of rolled samples. The Leica imagine analysis and linear intercept technique after etching in 2% nital were used for grain size measurements.

Table 2 gives the mechanical properties of new steels in the industrial experiment and the standard of GB/T1591-94(CHN). The mechanical properties of new steels were up to standard.

Table 3 shows the scan electronic microscopy (SEM) photos of the new 460 MPa grade plate steel, whose thickness was 20 mm. The microstructures are ferrite and pearlite. The average diameter of ferrite was about 6 \( \mu m \).

Figures 13(a)–13(d) show the microstructures from surface to center in new 460 MPa grade steel (20 mm thick; Z-direction). The microstructures were ferrite and pearlite, except for acicular ferrite in the surface, which thickness was less than 0.2 mm. For ensure the finish cooling temperature \( (T_{fc}) \) of the whole plate to get a low temperature quickly, the surface would be cooled to a lower temperature, which was fit for acicular ferrite transformation. The average grain of 12–20 mm thick steels was of 6 \( \mu m \) in size, all with good uniformity from surface to center.

The properties and the microstructures of the plate steels in industrial producing were coincident with the results in

### Table 2. The exact composition of the Nb–Ti microalloyed steel for industrial producing in wt%.

| C   | Mn | Si | S  | P  | Nb | Ti | Alc |
|-----|----|----|----|----|----|----|-----|
| 0.15| 1.47| 0.30| 0.014| 0.013| 0.019| 0.008| 0.015 |

**Alc**: acid solution aluminum

### Table 3. The mechanical properties of new steels and the standard of GB/T1591-94.

| Thickness[mm] | Re[MPa] | Rm[MPa] | A[%] | 180° cold bend (a= thickness; d= thickness; \( 40°C \)) | akv at the temperature of \( 40°C \) | Ductility-brittle transition temperature/°C |
|---------------|---------|---------|------|-------------------------------------------------|-----------------------------------|---------------------------------|
| New steels     |         |         |      |                                                 |                                   |                                 |
| 12            | 470–515 | 565–645 | 22.5–27 | \( d=2a; \) good                               | Ave.\(=75 \)                     | ≤60                             |
| 14            | 485–510 | 570–635 | 21–25 |                                                 | Ave.\(=68 \)                     |                                 |
| 16            | 470–500 | 585–625 | 20.5–24 |                                                 | Ave.\(=73 \)                     |                                 |
| 20            | 475–490 | 590–635 | 18.5–21 | \( d=3a; \) good                               | Ave.\(=67 \)                     |                                 |
| 30            | 465–500 | 610–640 | 18–22 |                                                 | Ave.\(=67 \)                     |                                 |
| GB/T1591-94   | ≥16     | ≥460    | ≥17 | \( d=2a; \)                                     | ≥27                              |                                 |
| Q460E(CHN)     | >16–35  | ≥440    | ≥17 | \( d=3a; \)                                     | ≥27                              |                                 |

Re: Yield strength;  
Rm: Tensile strength;  
A: Elongation;  
akv: the Charpy-V type notch impact absorption work.

**Fig. 12.** SEM photos of new 460 MPa grade plate steel (20 mm thick).
laboratory. It is certified that the optimized process parameters in laboratory were proper for industrial producing.

6 Conclusion

(1) The TMCP parameters affect the microstructure and properties of the Nb–Ti microalloyed steel greatly. The yield strength and tensile strength increase and the elongation decrease with the finish cooling temperature or the start rolling temperature of non-recrystallization decreasing; the final microstructures was affected by the finish cooling temperature mainly and the ferrite grain size was affected by the start rolling temperature of non-recrystallization chiefly.

(2) The TMCP parameters for industrial producing were optimized by laboratory experiments for the new Q460 grade steel. The deformation in non-recrystallization should be controlled in 780–850°C and the finish cooling temperature of accelerated cooling should be controlled in 640–680°C for the good combination of mechanical properties.

(3) The microstructures of the new Q460 grade steels were polygonal ferrite and pearlite. The average grain of 12–20 mm thick steels was of 6 μm in size, all with good uniformity from surface to center.

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