Effect of controlled rolling/controlled cooling parameters on microstructure and mechanical properties of the novel pipeline steel

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Abstract The study of controlled rolling/controlled cooling process parameters which affect the microstructure and mechanical properties of a novel pipeline steel have been optimized by the orthogonal experiment with four factors and three levels in this paper. However, the parameters of thermo-mechanical control process (TMCP) optimized by the Gleeble-3500 hot simulator could not satisfy performance requirements of the X100 pipeline steel. In order to improve the performance of this steel, the influence of finish cooling temperature (FCT) on the microstructure and property is studied in detail. It is found that, as this steel is thermo-mechanically treated by this set of parameters (the start heating temperature, finish rolling temperature (FRT), FCT and cooling rate of 1,180 °C, 810 °C, 350 °C and 35 °C/s, respectively), the microstructures are mainly composed of granular bainite (GB) and acicular ferrite (AF). The effective grain sizes are below 20 µm; the steel reached the optimal balance between the strength and the toughness; the yield strength

is 695 MPa; the tensile strength is 768 MPa; the elongation is 16.6 %; the impact energy is 262 J at room temperature. All indexes could meet the requirements of X100 pipeline steel.

Keywords Controlled rolling/controlled cooling · Finish cooling temperature (FCT) · Microstructure · Mechanical properties · X100 pipeline steel

1 Introduction

In the modern pipeline technology, much effort has been devoted on the strength, toughness and corrosion resistance of pipeline steel to increase its properties. Recently the transportation of crude oil or natural gas requires the steel with higher strength, toughness and good formability to get a reliable and safer access to energy [1]. Therefore, the development of novel pipeline steel attracted the attention of the engineers around the world [2]. Usually, the mechanical property of pipeline steel is determined by the proportion of multiple microstructures, which include polygonal ferrite (PF), acicular ferrite (AF), granular bainite (GB) and lathbainite (LB) with different technological processes [3]. Table 1 lists the general requirements of X100 pipeline steel in the engineering application [4]. The development and progress of the pipeline steel shows that the best combination of alloy composition design, metallurgical technology, controlled rolling and controlled cooling will determine the comprehensive performance of steel.

Currently, thermal-mechanical control process (TMCP) has been widely applied to control the grain structure in industrial production. For example, Gómez analyzed the evolution of microstructure and precipitation state of high-

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Table 1 The general requirements of X100 pipeline steel in engineering application

σ_b / MPa	σ_s / MPa	δ	Charpy V-notch impact toughness/J	P_{cm}
>760	>695	>15 %	>250	<0.20

level pipeline steel through TMCP process [5], in which the effects of processing parameters of TMCP [6], such as finish cooling temperature (FCT), finish rolling temperature (FRT) and coiling temperature on the microstructure and mechanical properties of low C-Mn steel were reported. Furthermore, Wang investigated that all strengthening types of fine grain precipitation and phase transformation could be obtained by TMCP. Therefore the alloy contents could be saved over 30 %; the strength of steel products could be increased by over 100–200 MPa; the energy could be reduced by 10 % [7].

In order to produce higher strength and toughness pipeline steel, the alloy design must be reasonably done. The pipeline steel is one of the most successful applications using the micro-alloying theory. Adding an appropriate amount of alloying elements Nb, V, Ti and Ni to general C-Mn steel makes the final grain of steel fine and improves the strength and toughness of the steel [8, 9]. The studies on the low carbon and high manganese of high-level pipeline steel attracted more and more attention. For example, Gao reviewed in details the development of pipeline alloy design—micro-alloying of low carbon or ultralow carbon and multi-alloying [10]. Wang and Lu [11] also systematically summarized the basic design idea aiming at developing the pipeline steel with better comprehensive properties.

In this work, one novel pipeline steel is suggested according to the current literature using the Gleeble-3500 hot simulator. The parameters of the TMCP including heating temperature, FRT, FCT and cooling rate are optimized by the orthogonal experiment with four factors and three levels. However the steel prepared by the optimum processing parameters of TMCP obtained by Gleeble-3500 hot simulator cannot meet the performance requirement of the X100 pipeline steel. Due to the key effect of the FCT on the microstructure and property of the pipeline steel, the influence of FCT will be further investigated. The final

goal is to squeeze out a set of TCMP parameters, by which the steel can reach the standard of the X100 pipeline steel.

2 Experimental

2.1 Chemical composition of pipeline steel

The steel was melted in 50 kg vacuum induction furnace in the laboratory, and then was forged into 80 mm × 80 mm square billet. Table 2 lists the chemical composition of novel pipeline steel.

2.2 Thermal-mechanical simulation test

Nine steel specimens with a size of $\Phi 10$ mm × 15 mm were cut from cast slabs and hot compressed to a cylinder with 4 mm in height through a two-stage controlled rolling process. It was well-known that, the factors such as the heating temperature, FCT, FRT and cooling temperature would mainly determine the microstructures and properties of pipeline steels [6]. Therefore, these four factors were used to build up the $L9(3^4)$ orthogonal table [12] for the Gleeble-3500 hot simulator in this study, as shown in Table 2.

2.3 Pilot rolling experiment

The hot rolling specimens with a size of 27 mm in thickness, 80 mm in width and 100 mm in length were cut from cast slabs. The optimum processing parameters of TMCP obtained from the Gleeble-3500 hot simulator were applied to the rolling experiment conducted in the laboratory.

2.4 Mechanical properties

Specimens for the tensile and impact tests were cut from the middle of the rolled plates in the transversal direction. The sample for tensile tests was prepared according to GB/T 228-2002, and the tensile tests were conducted at room temperature in a CMT5305 servo-hydraulic machine. To evaluate the charpy impact energy, the sub-size specimens (10 mm × 10 mm × 55 mm) with a V type notch were tested at room temperature with the standard method in a JB-6(80A00277) type impact testing machine. The

Table 2 Chemical composition of the novel pipeline steel

Element	C	Mn	Si	S	P	Nb	Mo	Ni	Ti	Al
Content/wt%	0.060	1.28	0.25	0.0017	0.016	0.053	0.31	1.30	0.012	0.0075

hardness of the samples was measured by the Rockwell hardness tester HRB69-1.

2.5 Microstructure of the hot simulation and the hot rolled specimens

Microstructures of the hot simulation and the hot rolled specimens were examined by the optical microscope (OM, Leca), the scanning electron microscopy (SEM, JSM6700F) and the energy dispersive spectrometer.

3 Results and discussion

3.1 Thermal-mechanical simulation of TMCP process

Nine steel specimens with the size of $\Phi 10 \text{ mm} \times 15 \text{ mm}$ are cut from cast slabs and final hot rolled to a cylinder with a height of 4 mm through a two-stage controlled rolling process, as shown in Fig. 1.

The results of the experiments and the analysis of orthogonal test are shown in Table 3.

Figure 2 shows the optical micrographs (a–i) of the specimens of hot simulation.

The hardness values of nine hot simulation specimens, HRB, were measured and listed in Table 2. The hardness of group e reached 101.5 HRB, which was significantly higher. For the simplicity, we measured the hardness instead of the strength according to GB/T 1172-1999 [13]. Generally, the strength is directly proportional to the hardness and the strength increases as the hardness increases. It may be reasonable to consider that the strength of group e may be higher than other groups.

Usually, the microstructure of pipeline steel is a multi-phase structure, including AF, LB, PF, GB, among which the AF can guarantee that the steel will have a higher toughness at low temperature and low ductile to brittle transition temperature without a falling in strength. Finally it greatly improves the service life of pipeline steel. Therefore, the main developing trend of modern high performance pipeline steel is how to get the microstructure with AF.

The nucleation of AF is attached to the existing interface, which is mainly provided by non-metallic inclusions [14] (such as titanium oxides). Meanwhile the residual distortion energy produced by the rolling process also promotes the nucleation of AF in our experiment. The microstructures of the hot simulation specimens at different process parameters are shown in Fig. 2. The optical micrographs (see Figs. 2a–i) are obtained under the processing parameters, as shown in Table 3. Then the effects of the start heating temperature, FRT, FCT and the cooling rate on the microstructure will be discussed in detail.

Fig. 1 The schematic of the thermo-mechanical treatments

Table 3 The analysis of orthogonal test

Level	Heating temperature/°C	FRT/°C	Cooling rate/(°C·s ⁻¹)	FCT/°C	HRB
a	1,150	860	15	400	84.1
b	1,150	810	25	450	90.0
c	1,150	760	35	500	88.4
d	1,180	860	25	500	90.5
e	1,180	810	35	400	101.5
f	1,180	760	15	450	85.2
g	1,200	860	35	450	82.8
h	1,200	810	15	500	80.8
i	1,200	760	25	400	92.1

These microstructures at cooling rate of 15 °C/s are similar to Duan's results [15], which are largely composed of the AF together with the LB, as shown in Figs. 2a, f, h. The AF is an acicular microstructure formed inside austenite grains and contains martensite austenite (MA) constituents at irregularly shaped grain boundaries. Small amounts of austenite grain boundaries are found around LB. LB grains are relatively large and its grain boundaries cannot be identified clearly. The microstructures of the specimens are heterogeneously dispersed, and the distance of AF is quite large. For specimens that are composed of PF together with LB at a cooling rate of 25 °C/s (see Figs. 2b, d, i), the amount of LB decreases. PF, which is transformed at the highest temperature, is an equiaxed microstructure and plate-like in shape. Compared with the specimen at a cooling rate of 15 °C/s, the size of grain is smaller. The specimens that are composed of the GB together with LB at cooling rate of 35 °C/s are shown in Figs. 2c, e, g. The GB contains the equiaxed MA constituents, and has well-developed substructures inside. The GB grains which are smaller than 10 μm in size are homogeneously dispersed. Its grains are relatively small and its grain boundaries are clearly identified.

Fig. 2 The optical micrographs (a–i) of the specimens of hot simulation

At the FCT of 400 °C (see Figs. 2a, e, i), PF is rarely observed with rapid cooling rate because a few high-toughness AFs are formed. A hard phase, such as MA, is formed coarsely. When FCT increases to about 450 °C (see Figs. 2b, f, g), specimens are largely composed of GB. High-toughness ferrite colonies increase in volume. When the FCT increases to higher than 500 °C (see Figs. 2c, d, h), AF and GB are primarily observed. With increasing FCT, high-toughness phases are formed coarsely and hard phases tend to be reduced in their sizes and are distributed homogeneously. This leads to low strength with good toughness [16].

When the FRT is 810 °C (see Figs. 2b, e, h), the microstructure is composed of LB, GB and AF. When FRT increases to 860 °C (see Figs. 2a, d, g), the microstructure is largely composed of GB and AF. Much high-toughness AF is formed. With increasing FRT, more AF observed leads to low strength [17]. The role of FRT on mechanical properties of the steel may be attributed to the acceleration of transformation and the refinement of grains by the reduction of FRT [18]. Therefore, the reduction of FRT will be beneficial to both strength and ductility. It has been reported [19] that when FRT is over a certain temperature (about 840 °C), both yield strength and toughness of the

Table 4 The inter-pass reduction distribution for the hot rolling experiment

Parameters	Value				
Rolling passes/No.	0	1	2	3	4
Thickness/mm	27.0	18.9	11.3	9.0	7.2
Reduction/mm		8.1	7.6	2.3	1.8
Pass reduction rate/%		30	40	20	20
Repeated reduction rate/%		30	58	67	74
Rolling temperature/°C		1,050	1,000	930	810

steel increase as the FRT decreases. If FRT is under this critical temperature, the banded microstructure appears which greatly reduces toughness.

In view of the results mentioned above, the hot simulated rolled specimens, which are treated by optimizing TMCP parameters (the start heating temperature, FRT, FCT and cooling rate are 1,180 °C, 810 °C, 400 °C and 35 °C/s, respectively), possess the microstructures that are mainly composed of GB, AF, LB and MA. The HRB value is maximized and may possess the desired mechanical properties. This set of parameters is proposed in the pilot rolling experiment for the development of an X100 pipeline steel.

3.2 Pilot rolling experiment

The initial and final thicknesses of the steel plates were 27 mm and 7.2 mm, respectively, and the total rolling reduction ratio was 73.3 %. Then the optimum processing

Fig. 3 The microstructure of pilot hot rolled samples (a–d): a, b OM micrographs, c the SEM micrograph, d EDS analysis

parameters of TMCP obtained from orthogonal experiment were used in the rolling experiment conducted in the laboratory. The specimen was heated at 1,180 °C for 30 min. It was finally rolled at 810 °C through a two-stage controlled rolling process. Then the hot rolled plate was immediately cooled at a controlled cooling rate of 35 °C/s and about 400 °C. Table 4 lists the inter-pass reduction distribution for the hot rolling experiment.

Figure 3 shows the microstructure of pilot hot rolled samples.

Table 5 lists the mechanical properties of pilot hot rolled samples.

Microstructures of the hot rolled specimens, with TMCP parameters of the start heating temperature, FRT, FCT and the cooling rate of 1,180 °C, 810 °C, 400 °C and 35 °C/s, respectively, were observed by OM, as shown in Fig. 3. The microstructures of all specimens are mainly composed of GB, LB and AF colonies. Polygonal ferrite was not found and the grain size in the microstructure was fine, as shown in Fig. 3a. At this condition, the amount of AF decreases and GB increases in volume (GB can be clearly seen). The AF colonies appear in isolation, and the relatively small amounts that cannot be easily identified [20], as seen in Fig. 3b. The microstructures were also observed by SEM and were composed of ferrite and bainite colonies, which were homogeneously dispersed. The ferrite is plate-like in shape. The width of the plate was 3–5 µm, and its length is 6 times of its width (see Fig. 3c). Moreover, the dislocation density of the samples is much high in the plate. The precipitates are mainly Ni, Mn carbonitrides (see Fig. 3d), which may be formed in the non-recrystallization zone as a result of rolling strain induced precipitation.

The performance of the steel treated by this set of TMCP parameters is shown in Table 4. The average tensile strength is up to 557 MPa; the ratio of tensile strength to yield strength is 0.89; the Charpy impact work at 20 °C is 371 J; the elongation is 21.3 %. Although the microstructure consists of GB, LB and AF, it does not meet the requirement of the X100 pipeline steel mechanical property [21]. As we know, the cooling system (cooling rate, FCT, FRT) is one of the key factors that decide the property and the constitution of microstructure of the X100 pipeline steel [22, 23]. In our previous work,

we designed three other factors and three levels orthogonal experiment with the cooling system factors to discuss its effect on comprehensive properties of X100 pipeline. We found that the FCT played a major role [24]. The FCT is easily controlled and will be investigated in details in order to improve the property of this steel and finally produce the steel which can reach the standard of X100 pipeline.

3.3 Optimization of FCT

The plates with sizes as mentioned above are divided into three groups and are rolled using the same controlled rolling process, as shown in Table 3. The only difference is the final temperature, as shown in Table 6.

The performance of steel with the different FCTs is listed in Table 7, and its effect on the mechanical property of specimens is shown in Figs. 4a–e.

Microstructures of specimens with the different FCTs are shown in Figs. 5, 6 and 7.

Figure 4 shows the relationship between the mechanical property of the steel and the FCT. From Figs. 4a, b, it can be seen that the tensile strength and yield strength of the steel decrease when the FCT increases, and the elongation and impact energy also increase (see Figs. 4c, d).

When the temperature is at 250 °C, the tensile strength and yield strength are 817 MPa and 719 MPa, respectively. The impact energy is 232 J and the elongation rate was more than 16 %. It cannot satisfy the requirements of plasticity and toughness of the X100 pipeline steel. The impact energy at the room temperature can be improved by increasing the FCT. When the temperature increases from 250 °C to 350 °C, the impact energy increases from 232 J to 262 J. Meanwhile the yield strength was 695 MPa; the

Table 5 Results of mechanical properties of pipeline steel

No. of samples	σ_s /MPa	σ_b /MPa	Elongation/%	Charpy impact energy/J	σ_s/σ_b
1	512	563	20	360	0.91
2	489	550	21	375	0.89
3	487	560	23	380	0.87
Average	496	557	21.3	371	0.89

Table 6 The three TMCP samples with different FCTs

Samples	FRT/°C	The finish rolling elongation/%	Cooling rate/(°C·s ⁻¹)	FCT/°C
1				450
2	810	20	35	350
3				250

Table 7 Mechanical properties of specimens with different FCTs

FCT/°C	σ_s /MPa	σ_b /MPa	Elongation/%	Charpy impact energy/J	σ_s/σ_b
250	743	817	14.3	232	0.91
350	695	768	16.6	262	0.89
450	662	719	23.4	274	0.92

Fig. 4 The mechanical property of the steel with the FCTs (250 °C, 350 °C, 450 °C)

tensile strength was 768 MPa; the elongation was 16.6 %. All indices can satisfy performance requirements of X100 pipeline steel. With the further increase of the FCT (450 °C), the elongation and impact energy are obviously increased, and the toughness is improved. However, its strength becomes lower and cannot meet the requirements of X100 pipeline steel.

Figures 5, 6 and 7 show the optical micrographs of the specimens at FCT of 450 °C, 350 °C and 250 °C, respectively. From Fig. 5, it can be seen that, at the FCT of

450 °C, the GB transformation occurs completely, therefore there exist lots of GB. This microstructure may result to the obvious decrease of the strength and the increase of the toughness of samples.

Figure 6 shows the microstructure of the steel at the FCT of 350 °C, which is mainly composed of GB and AF. As a result, the prior austenite grains are effectively separated, and the growth of lath-like is restricted in the separated zones. Hence the excessive growth of lath-like is limited and the grain orientation is not obvious. The

Fig. 5 The optical micrographs of the specimens at the FCT of 450 °C

Fig. 6 The optical micrographs of the specimens at the FCT of 350 °C

effective grain sizes become smaller and fall below 20 μm , and the steel possesses the optimal refinement microstructure. Compared to the specimens at FCT of 450 °C and 250 °C, these specimens reach the best balance between the strength and the toughness due to the occurrence of the refinement microstructure.

At the FCT of 250 °C, the microstructures of the specimens are composed of the lathbainite and martensite (LB + M), as shown in Fig. 7. The orientation of lath-like in the prior austenite grain is relatively simple, which runs through the austenite grain at the length direction of lath, and the part of boundaries become unclear. However the boundaries between the laths are clear and the lath characteristics are more obvious. The characteristics of massive

transformation exist at the local zone. The effective grain sizes become larger and increase to above 30 μm in general. This fact may cause the high strength and poor plasticity and toughness of specimens.

4 Conclusions

- (i) The X100 pipe steel developed in this study is studied by the Gleeble-3500 hot simulator as well as the orthogonal experiment design. The microstructures mainly composed of GB, AF, LB and MA are observed by this set of TCMP. The start heating temperature, FRT, FCT and the

Fig. 7 The optical micrographs of the specimens at the FCT of 250 °C

cooling rate of 1,180 °C, 810 °C, 400 °C and 35 °C/s, respectively. Its HRB value is maximized.

- (ii) The optimized TCMP parameters mentioned above are used in the pilot rolling experiment. The results show that the average tensile strength can be up to 557 MPa, and the ratio of tensile strength to yield strength is 0.89. The Charpy impact work at 20 °C can be up to 371 J, and the elongation can reach 21.3 %. Observed by using OM and SEM, its microstructure consists of GB, LB and AF.
- (iii) The FCT is investigated in details meanwhile other factors are kept unchanged. With the increase of FCT, the strength of the experiment steel decreases and both the elongation and Charpy impact energy increase. When the FCT is 350 °C, the steel reaches the optimal balance between the strength and the toughness.
- (iv) For the steel thermo-mechanically treated by this set of parameters (the start heating temperature, FRT, FCT and cooling rate of 1,180 °C, 810 °C, 350 °C and 35 °C/s, respectively), the microstructures are mainly composed of GB and AF, this is the optimal microstructure.

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