The X80 Pipeline Steel produced by a Novel Ultra Fast Cooling Process

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Abstract: The relation between microstructure characteristics and mechanical properties of X80 pipeline steels produced by ultra fast cooling process was investigated using energy dispersive spectrometer, scanning and transmission electron microscopy. Results showed that ultra fast cooling kept hot rolled austenitic work hardened, induced a large number of carbonitride precipitation, limited the growth of cementite. Therefore ultra fast cooling was beneficial to increase the steel strength and improve the steel ductility and toughness.

1. Introduction
With the rapid development of China's economy, the rapid growth of energy demand, the rapid development of oil and gas pipelines, large demand for pipeline steel [1-3]. Owing to its high strength and good toughness the X80 pipeline steel has become the most used steel grade in long distance pipeline projects over the world [4]. Traditional X80 pipeline steel is mainly produced by TMCP process [5-6].

The ultra fast cooling process mainly adopts a faster cooling rate than the traditional cooling process, refines the grain structure after finishing rolling, reduces the alloy content and production cost, and realizes low-carbon green production [7-10]. The present work aims to utilize ultra fast cooling process to produce the X80 pipeline steel and investigate its microstructure characteristics and mechanical properties.

2. Experimental procedures
The investigated steels are 17.5 mm thick plates. The chemical compositions are listed in Table 1. The TMCP parameters for the production of pipeline steels are shown in Table 2.

Table 1 Chemical composition of the X80 pipeline steel (%)

| C  | Si  | Mn   | Nb   | Mo +Cr+ Ti | P   | N    |
|----|-----|------|------|-----------|-----|------|
| 0.04~0.06 | 0.2~0.3 | 1.7~1.9 | 0.06~0.10 | 0.60~0.70 | ≤0.02 | 0.0073 |

The investigated steels are 17.5 mm thick plates. The chemical compositions are listed in Table 1. The TMCP parameters for the production of pipeline steels are shown in Table 2.
Table 2 The rolling process parameters of the X80 pipeline steel

| code | Recrystallization zone rolling stage | Not recrystallization zone rolling stage | Ultra fast cooling phase | Laminar cooling phase |
|------|------------------------------------|-----------------------------------------|-------------------------|----------------------|
|      | start Blooming temperature         | start finishing rolling temperature     | exit temperature°C      | Cooling rate°C/ s    |
|      | Finishing exit temperature         |                                        | exit temperature°C      |                       |
| 1#   | 1050                               | 950                                     | 800~850°C              | ≤650                 | ≥30                  |
|      |                                    |                                        |                         | 450                  | 6~10                 |
| 2#   | 1050                               | 950                                     | 800~850°C              | ≤650                 | ≥30                  |
|      |                                    |                                        |                         | 400                  | 6~10                 |
| 3#   | 1050                               | 950                                     | 800~850°C              | ≤650                 | ≥30                  |
|      |                                    |                                        |                         | 350                  | 6~10                 |
| 4#   | 1050                               | 950                                     | 800~850°C              | ≤650                 | ≥30                  |
|      |                                    |                                        |                         | 320                  | 6~10                 |

3. Results
Specimens for tensile tests were cut from the rolled plates in the transverse direction. Tensile test were conducted at room temperature at a crosshead speed of 5 mm/min on an SHT4106-1000KN servo-hydraulic machine. Drop weight tear tests (DWTT) specimens were sized by 75 mm×22 mm×305 mm (T-L), and DWTT were performed at -15℃using a JL-40000 impact machine. Fullsize Charpy impact specimens were machined from the rolled plates in the transverse direction with their notch parallel to the rolling direction (T-L), and impact tests were done from 20℃ to -100℃using a ZBC-300C impact machine. Specimens for SEM observation were mechanically polished and etched by a 4 pct nital solution or a Lepera solution, and were observed by a Quanta 600 SEM. Specimens for Transmission electron microscope (TEM) analysis were Used sand paper to grind about 40 um to 100um, and pressing into small rounds Φ 3 mm. After mechanical thinning of small wafers with double DJ2000 electrolytic jet apparatus thinned specimens to perforation, With JEM – 2100F TEM testing the microstructure, accelerating voltage of 200 kv. Mechanical properties are shown in table 3, Drop weight tear tests (DWTT) are shown in table 4, Fullsize Charpy impact are shown in table 5.

Table 3 Tensile properties of the X80 pipeline steel

| code | R_0.5/MPa | R_m/MPa | A50/% | R_0.5/R_m |
|------|-----------|---------|-------|-----------|
| 1#   | 652       | 720     | 38    | 0.91      |
| 2#   | 645       | 735     | 38    | 0.88      |
| 3#   | 635       | 745     | 37    | 0.85      |
| 4#   | 660       | 760     | 37    | 0.87      |

Table 4 DWTT of the X80 pipeline steel

| Code | SA/% |
|------|------|
| 1#   | 95   |
| 2#   | 92   |
| 3#   | 90   |
| 4#   | 90   |

Table 5 CVN of the X80 pipeline steel

| code | Transverse temperature |
|------|------------------------|
| 1#   | 30°                    |
| 2#   | 30°                    |
| 3#   | 30°                    |
| 4#   | 30°                    |
Use the SEM observation and analysis of X80 pipeline steel microstructure morphology of the sample. As shown in fig. 1, It can be seen, 1 # sample microstructure is relatively bulky, There is a small amount of polygonal ferrite(PF), A small M/A islands in the microstructure, 2 # sample mainly acicular ferrite, Compared with 2 # sample tiny evenly, M/A island content more, Most of the small size of M/A islands, and evenly distributed, 3 #, 4 # sample microstructure is fine, By the morphology and distribution of M/A islands, favours granular bainite microstructure type, Acicular ferrite matrix grain boundary in the mutual crisscross, direction of different characteristic is not obvious

Use the TEM observation and analysis of X80 pipeline steel microstructure morphology of the 2#, 4#sample, As shown in fig. 2,
Fig. 2 Transmission electron micrographs of precipitates in sample

2 # sample dislocation is very developed, Can be observed that a large number of Dislocation plug product, Precipitation is less, the Size is relatively fine and uneven distributed. Types of precipitation mostly Ti, Nb carbonitride precipitation composite, Through the EDS analysis can know Ti content is higher in rectangular particle, Nb content is higher in round particles. Nb/Ti ratio of 3.3.

Fig. 3 4 # sample TEM observations
4 # sample on the grain boundary have plenty of dislocation product, and has a wide distribution in the range of observation. Most precipitation particle size smaller, most typical particles as shown in the picture on the right around 30 nm, A single round particles can be observed. Types of precipitation mostly Ti, Nb carbonitride precipitation composite, Through the EDS analysis can know Nb content is higher in circular particle Ti content is low. Nb/Ti ratio of 15.1.

4. Discussion
Compared with the traditional laminar cooling process, the cooling rate of ultra fast cooling process is greater than 35 /s. When the final rolling temperature and cooling termination temperature are the same, the ultra fast process shortens significantly through austenite zone, and the work hardening time during rolling is less than that of softening. There are more hardened austenite before transformation, simultaneous interpreting a lot of nucleation power for phase transformation. The higher the cooling rate is, the greater the transformation kinetic energy is, the more nucleation cores are, and the finer the transformation grains are. It can be seen that there are a large number of dislocation packing and dislocation clusters near the M/A island, and a large number of dislocations gather at the grain boundary, causing stress concentration. In addition, small precipitated particles tightly bind the dislocations, which has a good pinning effect. And with the increase of cooling temperature precipitation number increases gradually, and also become evenly distributed. But with the increase of cooling temperature, precipitation size increases gradually, the highest in the cooling temperature of sample precipitation particle concentration, reunion phenomenon occurring in some places, So you need to formulate reasonable coiling temperature.

5. Conclusions
The relation between microstructure characteristic and mechanical properties of X80 pipeline steels by Ultra fast Cooling Process was investigated, and the following conclusions could be reached:

a) the X80 pipeline steel with the increase of coiling temperature impact toughness increased, Yield strength decrease first and then increased, Around 350 °C when the lowest yield strength, yield ratio with first decreases and then increases with the rising of cooling temperature. Comprehensive analysis shows that cooling temperature about 400 °C when the better comprehensive mechanical properties.

b) the cooling speed is greater than 30 degrees per second to ensure the passing through in austenite region in a tiny period of time, resulting in work hardened austenite before transformation, which can lay a solid foundation for the control of phase transformation in work hardened state. The faster the cooling rate, the microstructure is fine.

c) Types of precipitation mostly Ti, Nb carbonitride precipitation composite, Through the EDS analysis can know Ti content is higher in rectangular particle, Nb content is higher in round particles.

d) with the increase of coiling temperature precipitation particle number increases gradually, and also gradually become evenly distributed. But with the increase of coiling temperature, precipitation particle size increases gradually.

Acknowledgements
The authors gratefully acknowledge the financial support of Natural Science Foundation of Guangdong Province [grant number: 2018A030313287, 2017A030313276]; and Characteristic innovation projects of Guangdong Provincial Department of Education [grant number: 2017GKTSCX086, 2016KZDXM046]. Guangzhou Science and Technology Plan Project [grant number: 201804010168]; National Natural Science Foundation of China [grant number: 51801066]; Guangzhou collaborative innovation project [grant number: 201604016082].

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