



## Relation among rolling parameters, microstructures and mechanical properties in an acicular ferrite pipeline steel

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### ABSTRACT

The correlation among thermo-mechanical controlled processing (TMCP) parameters, microstructures and mechanical properties of an acicular ferrite (AF) pipeline steel was investigated in this study. The steel was hot rolled by four different kinds of TMCP to obtain different AF microstructures, and the corresponding mechanical properties were analyzed. Electron backscatter diffraction (EBSD) analysis was conducted to determine the effective grain size (EGS) in the steel. It was found that the EGS in the steel reduced obviously with decrease of the finish rolling temperature (FRT), but little changed with the cooling rate (CR) and the simulated coiling temperature (SCT). Additionally, the fraction of low angle grain boundaries (LAGBs) increased with increasing CR in the experimental range. It was shown that yield strength of the steel was enhanced by the increased CR and SCT, and reduced FRT, which were corresponding with the increases of LAGB fraction and precipitated carbonitrides as well as the decrease of EGS, respectively. Charpy impact results showed that the low temperature toughness of the steel with FRT about 40 °C above  $A_{r3}$  tended to be the best, which was in good accordance with the highest fraction of high angle grain boundaries (HAGBs), but seemed not to be related with the EGS.

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## 1. Introduction

In modern industry, pipeline steels are becoming higher in strength and toughness in order to increase the transportation efficiency, and it is a target to obtain excellent combination properties through optimizing thermo-mechanical controlled processing (TMCP) parameters and corresponding microstructures. Many researches have been done on the acicular ferrite (AF) microstructure pipeline steels [1–5], since it was first developed in the early 1970s [6,7]. With decrease of transformation temperature and increase of cooling rate for pipeline steels produced by TMCP, ferrite microstructures can be mainly divided into polygonal ferrite (PF), quasi-polygonal ferrite (QF) or massive ferrite, granular bainitic ferrite (GF) and bainitic ferrite (BF) [3–5]. Currently, it has been well accepted [2,6] that AF results from a mixture of diffusion and shear transformation mode beginning at a temperature slightly above the upper bainite temperature transformation range during continuous cooling, presenting an assemblage of interwoven non-parallel ferrite laths with high density tangled dislocations. However, there are still controversies and uncertainties on the metallographical identification and classification of the phases in AF. Sometimes, the microstructure of AF is also considered as bainite [8] or QF [9]. But anyway, from our previous work, the

microstructure of AF is complexly consisting of QF, GF and BF with dispersed islands of second phases in the matrix [10].

Pipeline steels with AF microstructure behave better combination of mechanical properties such as higher strength [11], better toughness [11], superior  $H_2S$  resistance [12], and better fatigue behavior [13] than those with PF – Pearlite (P) microstructure, which has led to the application of AF pipeline steels in manufacturing of large dimension pipes for gas and oil transportation in the low temperature area [14–16].

However, different components in AF microstructure which come from change of TMCP parameters bring forth different mechanical properties for AF pipeline steels. In this study, based on the previous work [17,18], high strength and toughness AF pipeline steels with different components in microstructure were fabricated by change of TMCP parameters. Through determination of the microstructural factors by electron backscatter diffraction (EBSD) analysis, the relation among TMCP parameters, microstructures and mechanical properties was investigated.

## 2. Experimental procedure

The steel used in this study, a laboratory smelting microalloyed pipeline steel designed by the authors, was prepared in a 100-kg vacuum induction melting furnace. Chemical composition of the steel is listed in Table 1. The materials, 70 × 78 × 80 mm in size, for hot rolling were cut from the forged slabs.

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**Table 1**  
Chemical composition of the experimental steel (weight percent).

C	Si	Mn	Mo	Nb	V	P (ppm)	S (ppm)	O (ppm)	N (ppm)	Fe
0.025	0.24	1.56	0.32	0.039	0.019	20	6	43	62	Bal.

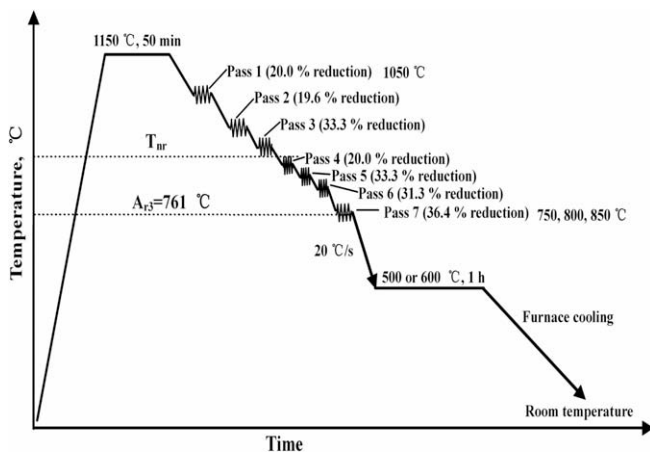
The hot rolling was carried out on a pilot rolling mill with 370 mm diameter twin rolls. According to the previous work, the continuous cooling transformation (CCT) diagrams of the experimental steel measured by Formastor-F and Gleeble-3500 [17,18], the parameters for TMCP were designed as designated in Fig. 1 and measured as listed in Table 2. The materials were heated at 1150 °C for 50 min and hot rolled from 70 mm to 7 mm thick plates by seven steps. The finish rolling temperature (FRT) was chosen as 850 °C, 800 °C, above  $A_{r3}$ , and 750 °C, just below  $A_{r3}$ , to study the effect of FRT on microstructures and mechanical properties of the steel. In order to obtain AF microstructure, the cooling rate (CR) was chosen to be about 20 °C/s. The simulated coiling temperature (SCT) was 500 °C or 600 °C.

The transverse cross sections of the rolled steels were mechanically polished and etched by a 3 pct nital solution, and microstructures were observed on an MEF-4 optical microscopy.

Specimens for tensile tests were cut from the middle of the rolled plates in the longitudinal direction and conducted at room temperature with a crosshead speed of 5 mm/min on an SCHENCK-100KN servo-hydraulic machine, according to the standard ASTM E 8M-04. Charpy impact tests were performed at temperatures of 0 °C, –40 °C, –100 °C, –120 °C, –150 °C, –180 °C, –196 °C and –269 °C on sub-size Charpy V-notch (CVN) specimens with size of 5 × 10 × 55 mm, which were machined from the middle of the rolled plates in the transverse direction, in accordance with the standard method in ASTM E 23-02. In order to reduce errors in data interpretation, a regression analysis for CVN energy vs. test temperature was done by a hyperbolic tangent curve fitting method [19]. Based on the data from the regression analysis, the energy transition temperature (ETT), which corresponds to the average value of upper shelf energy (USE) and lower shelf energy, was determined.

In order to examine the cleavage fracture unit and crack propagation path, the fracture surface and cross-sectional area beneath the fracture surface of the CVN specimens fractured at –196 °C after nickel plating were observed on an S-3400 N scanning electron microscope (SEM).

To investigate microstructural factors determining mechanical properties, the electron backscatter diffraction (EBSD) analysis was conducted on the transverse cross sections of specimens on



**Fig. 1.** Schematic diagram of the experimental program for TMCP.

the S-3400 N SEM, equipped with an HKL Channel 5 orientation imaging microscopy (OIM) system with a step size of 0.2 μm.

### 3. Results

#### 3.1. Microstructure and crystallographic orientation

Fig. 2 shows optical micrographs of Steels A–D subjected to different rolling processes. Microstructures of the steels with different TMCP parameters were acicular ferrite (AF), which could be further divided into quasi-polygonal ferrite (QF), granular bainitic ferrite (GF) and bainitic ferrite (BF), as marked in Fig. 2, according to different microstructural characteristics. The QF has irregular and jagged boundaries, containing high density of dislocations, subboundaries, and even M/A (martensite/austenite) components. The GF includes granular and equiaxed retained austenite or M/A islands dispersed in the ferritic matrix, also containing high density of dislocations. Whereas the BF consists of many elongated ferritic lath bundles, with elongated and dispersed M/A components.

Generally, high angle grain boundaries (HAGBs) with misorientation of 15 degree or higher obtained from EBSD can be used as a crystallographic domain parameter showing the effective grain size (EGS) [20,21]. Grains with misorientation angle larger than 15 degree for steels with different microstructures are shown in Fig. 3. Using the linear intercept method of HKL Channel 5 OIM analysis software, the EGS of the Steels A–D was estimated to be 3.04 μm, 2.26 μm, 1.63 μm and 1.63 μm, respectively.

Distributions of grain boundary misorientation of the Steels A–D are shown in Fig. 4. Calculated from Fig. 4, the fractions of low angle grain boundaries (LAGBs), misorientation angles less than 15 degree, of the Steels A–D are 45.5%, 28.7%, 30.2% and 35.1%, respectively; while those of HAGBs of them are 51.2%, 68.1%, 66.7% and 61.9%, respectively.

#### 3.2. Tensile properties

Room-temperature tensile test results are listed in Table 3. The yield strength of Steel A is over 483 MPa (70 ksi), satisfying the requirement of API X70 grade pipeline steels. Steels B and C have yield strengths above 552 MPa (80 ksi), satisfying the requirement of API X80 grade pipeline steels. The yield strength of Steel D is over 625 MPa (90 ksi), satisfying the requirement of ISO X90 grade pipeline steels. Both yield strength and tensile strength of Steels A–D increased with decreasing FRT and increasing CR during TMCP.

However, the elongation had not much change for Steels A–D. Steel C showed the highest elongation, then Steels A, B and D followed.

#### 3.3. CVN impact toughness

The curves of CVN energy vs. temperature for Steels A–D are shown in Fig. 5. Table 4 presents the CVN energy data in detail, and the results of USE and ETT obtained from Fig. 5. Steels A–D all showed excellent CVN properties, with high USE above 130 J and low ETT below –145 °C. It can be seen that Steel A had higher USE and absorbed energy at the tested temperatures from 0 °C to –120 °C than those of the other steels, but its CVN energy dramatically decreased below –150 °C. Steels B, C and D showed better low temperature toughness than Steel A, with lower ETT and

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