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Effects of non-recrystallization zone reduction on microstructure and precipitation behavior of a ferrite-bainite dual phase steel

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A B S T R A C T

A dual-phase pipeline steel with high deformability was processed by a two-stage controlled rolling and followed by relaxation process before accelerated cooling. The mechanical properties of the samples were studied, and the effects of different reductions in the second rolling stage on the microstructure and precipitation behavior of the steel were analyzed using optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM), positron annihilation technique (PAT) and physicochemical phase analysis. The samples mainly consisted of ferrite, bainite, and a small amount of martensite/austenite (M/A) islands. When the reduction increased to 80% in the second rolling stage, lower yield ratio and better impact toughness were obtained, and the microstructure with 49.6% (volume fraction) ferrite and higher dislocation density became more homogenous. The nominal chemical formulas of M(C, N) for sample A and B were $(Nb_{0.735}Ti_{0.260}Cr_{0.005})(C_{0.420}N_{0.580})$ and $(Nb_{0.757}Ti_{0.237}Cr_{0.006})(C_{0.464}N_{0.536})$, respectively and the phase structure of precipitates of both samples were almost the same. The strain induced more precipitation of M(C, N) in the non-recrystallization zone of austenite than in the intercritical zone. Also the fraction of M(C, N) and the precipitation strengthening effectiveness were promoted by the increase of reduction.

1. Introduction

Low carbon microalloyed steel with ferrite-bainite dual phase has many excellent properties, such as continuous yielding behavior, high initial work-hardening rate, good strength–ductility combination and low yield strength/tensile strength ratio (Y/T). Ferrite–bainite dual phase pipeline steel has been widely used in seismic and/or permafrost region for its good formability, work hardening ability and weldability [1–3]. In ferrite–bainite dual phase pipeline steel, the polygonal ferrite is a key factor to ensure high deformability, which should guarantee appropriate volume fraction and grain size, less precipitates and dislocation tangles, and the movable dislocation density among the ferrite/bainite interface should be increased [4]. To obtain desired microstructure and mechanical properties, alloying additives such as Mn, Nb, V, Ti, Mo, Ni, Cr and Cu are commonly employed in pipeline steels [5,6]. The precipitation of these elements plays an important role in controlling the final microstructure and mechanical properties. For instance, the presence of precipitates of Nb and Ti can increase the austenite grain coarsening temperature and the non-recrystallization temperature, which plays an important role in determining the parameters of reheating process. Furthermore, the interaction between precipitates and dislocations increases the flow stress, which is an effective way to increase the strength of material [7–9]. Besides, the processing parameters such as reheating temperature, percentage reduction, deformation temperature, relaxation time, cooling rate and coiling temperature are also important in determining the ultimate microstructure. For example, the cumulative deformation during hot rolling refines the austenite grains and increases the dislocation and substructure in austenite, and these effectively promote the transformation of fine ferrite [10]. In controlled rolling process, the recrystallization of austenite and ferrite caused by deformation is the primary method of grain refinement. Following the controlled rolling, the accelerated cooling also refines bainitic microstructure [11]. It is known that the particle size, number density and distribution of precipitates strongly affected the mechanical properties of metals [12]. And the microstructure evolution and precipitation behavior based on the thermo-mechanical controlled rolling in dual-phase pipeline steel with different alloy elements have been analyzed in many articles [3,13–18]. However, systematic studies about the effects of parameters of the second rolling stage, especially the percentage reduction, on the mechanical properties, microstructure and precipitation behavior of ferrite–bainite dual phase steel are limited.

In this paper, a ferrite–bainite dual phase pipeline steel was analyzed. The steel contained Cr instead of Mo and was processed by two stage thermo-mechanically controlled rolling. Different percentage reduction was applied during the second stage of rolling process. Its influence on microstructure, mechanical properties and precipitation behavior was studied. The precipitation behavior was seriously studied in detail with SEM, TEM and physicochemical phase analysis. And the

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difference of dislocation density in samples was analyzed with PAT. This work may provide technical reference for the development and practical production of high deformability pipeline steel with ferrite–bainite dual phase.

2. Materials and methods

The experimental steel studied in this paper was a commercial pipeline steel (API X80 grade) with large deformability and the chemical composition was given in Table 1. The phase transition point Ac_3 and Ac_1, obtained by differential scanning calorimetry (DSC), were 818 °C and 672 °C, respectively. Non-recrystallization temperature (T_{nr}) was determined by the empirical equation [19]:

$$T_{nr} = 887 + 464C + 890Ti + 363Al - 357Si + \left(6445Nb - 644\sqrt{Nb}\right) + \left(732V - 230\sqrt{V}\right). \quad (1)$$

Where C, Ti, Al, Si, Nb, and V are the weight percentages of carbon, titanium, aluminum, silicon, niobium and vanadium, respectively. After continuous casting, the slab billets were reheated to 1200 °C, held for 1 h in a resistance heating furnace. A two stage thermo-mechanical processing was performed on a pilot rolling mill with twin rolls of 450 mm in diameter to roll the billets to a thickness of 25 mm, and the TMCP (Thermal Mechanical Control Processing) schedule is shown in Fig. 1. The first rolling stage was in the austenite recrystallization region at high temperature, and the second rolling stage happened in the austenite non-recrystallization and intercritical regions. Two different thermo-mechanical processes (marked as A and B) were designed by varying the rolling reduction in the second rolling stage as shown in Table 2, and the section size of slab billets for A and B was 100 × 100 mm (cross dimensions) and 200 × 200 mm, respectively. For sample A, the accumulated reduction in the second stage was 60%, while the parameter for sample B was 80%. After hot rolling, a relaxation process (free cooling) was followed and then the steel plates were cooled by water to 230 °C at a rate of 23 °C/s.

In microstructure study, all samples were derived from the position at the quarter thickness of the plate by wire cut electrical discharge machining. After mechanical grinding, polishing and etched with 4% nital, the final microstructures of the specimens A and B were observed by an Olympus GX51 optical microscope and S-4300 scanning electron microscope (SEM), respectively. The volume fraction of ferrite for different treatment processes was counted by Image Pro-Plus 6.0 software. For further microstructural analysis, transmission electron microscopy (TEM) observation was carried out on thin foils. The foils were prepared by cutting thin wafers from small coupons and grinded to 70 μm. Disks of 3 mm in diameter, punched from the wafers, were twin-jet electropolished by a solution of 4 vol.% perchloric acid in ethanol at −20 °C. These foils were examined by a JEM-2100 (HR) TEM operated at 200 kV and EDX analysis was also carried out. Besides, the positron annihilation technique (PAT) was used to analyze the change in dislocation density. A fast–slow positron annihilation lifetime spectrometer was applied to measure positron annihilation lifetime of the specimens [20]. In order to quantitatively analyze the precipitates, physicochemical phase analysis was employed. In this part, samples with dimensions of 100 mm × 22 mm × 7 mm were prepared. To get the precipitates in samples, the samples were electrolyzed in aqueous solution (0.5% KCl and 0.5–1.0% citric acid) under the conditions of ampere density i = 0.025–0.03 A/cm^2, temperature T = 5–10 °C and total current $I \leq 0.7$ A. After electrolysis, the precipitates were collected by suction

Table 1
Chemical composition of the experimental steel (wt.%).

C	Si	Mn	Cr	Ni	Nb	Ti	Cu	Al	N
0.06	0.3	1.77	0.25	0.21	0.07	0.014	0.24	0.034	0.0055

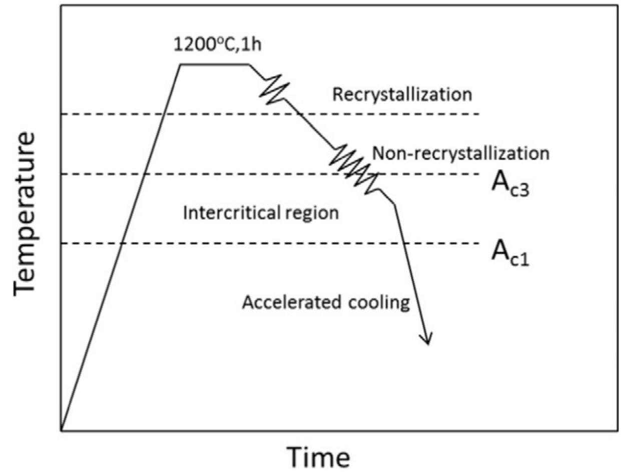

Fig. 1. Schematic diagram of thermo-mechanical processing schedule.

filtration using filter membrane with the porosity of 0.2 nm and washed them with deionized water. Then the retained precipitates were dissolved in a solution (50 mL C_2H_2OH + 5–10 mL H_2O_2, 20 °C) and let stand for 24 h to obtain M_3C. And in order to obtain MC and M(C, N), the retained precipitates were dissolved in another solution (10–20% hydrochloric acid + 1–5% citric acid ethanol solution, 20 °C), stirred and let stand for 4–6 h. A Philip APD-10 X-ray diffractometer (CPS: 100 or 200, 33 kV, Cokα 15–115° (2θ)) was used to analyze the obtained precipitates. And the content of each precipitated phase was determined by adiabatic method. To study the size distribution of the obtained precipitates, the precipitates were mixed with the collodion cotton acetone solution (70 g/L) to produce the collodion film, and then the particle size (diameter) distribution was determined with X-ray diffraction-spectrometer/Kratky small angle scattering goniometer with CoKα at 30 kV and 30 mA [21] according to GB/T 13221-2004 standard. The particles with size more than 300 nm were not included here.

The mechanical properties of the samples were measured by tensile test and drop weight tearing test (DWTT). For tensile tests, round specimens of 20 mm gauge length and 4 mm gauge diameter were prepared as per ASTM E8M standard from the rolled plate in longitudinal direction. Tensile tests were conducted at room temperature with a universal tensile testing machine of 200 kN capacity at a crosshead speed of 1.5 mm/min. In DWTT experiment, the specimens (305 mm × 75 mm × thickness) were machined from the rolled plate in transverse-longitudinal direction according to API RP 5L3 standard, and then a pressed notch was introduced into them. The specimens were tested at −15 °C by a DWTT testing machine (model: DWTT JL-50000) with a maximum energy capacity of 50 kJ to measure the fracture ductility of the steels.

3. Results and discussion

3.1. Mechanical properties of the samples

The mechanical properties of the samples were studied and Fig. 2 shows engineering stress–strain curves of the two samples. Both samples A and B showed continuous yielding phenomenon. Through the engineering stress–strain curves, the main mechanical properties of the samples are summarized in Table 3, and the results of DWTT are also included. The tensile test results indicate that yield strength ($R_{t0.5}$) of sample A was higher than that of sample B, but the ultimate tensile strength (UTS) of samples didn't show much difference with the variance of rolling reduction. The stress ratios of $R_{t1.5}/R_{t0.5}$ and $R_{t2.0}/R_{t1.0}$, which are verified to perfectly characterize the strain-hardening capacity of high strain pipeline steel in engineering, were a