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Microstructure characterization and mechanical behavior analysis in a high strength steel with different proportions of constituent phases



Fei Peng^a, Yunbo Xu^{a,*}, Xingli Gu^a, Yuan Wang^a, Jianping Li^a, Hua Zhan^b

^a State Key Laboratory of Rolling and Automation, Northeastern University, Shenyang 110819, China
^b Maanshan Iron & Steel Company Limited. Maanshan 243003. China

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ABSTRACT

A high strength steel with Nb-V-Ti addition was heat treated by a series of isothermal bainite holding (IBH) processes and quenching and partitioning (Q&P) processes with different intercritical annealing temperatures. The microstructure observation showed that some martensite was formed during quenching in IBH condition with annealing temperature above 850 °C, mainly ascribed to heterogeneous carbon content distribution in parent austenite with relatively large size. Meanwhile, film-like RA in martensite, relatively coarser lath-like RA in bainite and blocky RA located in grain boundaries or phase boundaries were observed in both IBH and Q&P treatments. Moreover, Q&P process was proved to be more beneficial to retain austenite than IBH process, while the ability of austenite retention was similar in IBH conditions. An excellent combination of strength and ductility was obtained in Q&P process annealed at 880 °C with tensile strength of 1126 MPa and total elongation of \sim 18%, attributing to TRIP effect mainly occurred in the latter part of strain and fine microstructure with homogeneous distribution.

1. Introduction

In order to meet the requirements of improving fuel efficiency and occupant security in automotive industry, variable microstructure strategies are applied to obtain good combination of strength and ductility [1]. As one of the most promising approach, the addition of retained austenite can effectively improve the strength/ductility combination due to the transformation induced plasticity (TRIP) effect [2,3]. For the sake of obtaining abundant volume fraction of retained austenite (RA), isothermal bainite holding (IBH) treatment is applied as a traditional method in TRIP steel and the final microstructure comprises an aggregate of ferrite, carbide-free bainite including carbonenriched austenite and a small fraction of martensite [4,5]. However, due to the limited strength grade of IBH treatment, a novel heat treatment called quenching and partitioning (Q&P) is proposed and shows attractive mechanical properties at high strength levels [6,7]. In Q&P process, austenite after annealing is partially transformed into initial martensite after quenching to a controlled temperature (referred to as QT) between the martensite-start (M_s) and martensite-finish (M_f) temperatures. Subsequently, partitioning process is applied at OT (onestep Q&P) or a higher temperature (two-step Q&P). Simultaneously, the initial martensite tempers during partitioning process and the carbon atoms in supersaturated martensite transfers into neighbored austenite

to achieve austenite retention [8]. Instead of bainite ferrite, tempered martensite is introduced into Q&P steel to act as main hard phase and Q &P steel generally shows a more excellent combination of high strength and good elongation as compared to that of TRIP steel with similar chemical compositions [9].

Moreover, as a critical constituent phase, intercritical ferrite mainly acts as a soft phase to change strength level. It is well known that the volume fraction of ferrite could be precisely regulated by intercritical annealing temperature to change the chemical composition and phase transformation behavior of austenite during subsequent process [10]. As a consequence, different phase constituents are obtained and hence the final mechanical properties are controlled by annealing temperature. Actually, the martensite and bainite transformation kinetics were also affected by prior ferrite formation [11]. Furthermore, as reported in some articles, heterogeneous carbon distribution existed in austenite after intercritical annealing, which showed a relatively higher carbon content in the exterior and carbon-depleted in the interior of austenite [12,13]. Hence, different phase transformations would happen depending on the corresponding position in parent austenite [12]. However, as for high strength steels, few articles were focused on the effect of intercritical annealing temperature on phase transformation, especially phase transformation types and final phase constituents, which influence the final mechanical behaviors.

E-mail address: yunbo_xu@126.com (Y. Xu).

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^{*} Corresponding author.

Table 1

Chemical	composition	of tested	steel	(wt%).	
					-

С	Mn	Si	Al	Ti	Nb	V
0.20	2.11	1.49	0.81	0.035	0.045	0.22

In this article, IBH process with different intercritical annealing temperatures and two typical Q&P treatments were applied in a high strength steel with micro-alloyed addition. This paper aims to provide an insight into the influence of phase constituents under different heat treatment conditions on final mechanical behavior. Meanwhile, the characteristics of retained austenite were also observed and compared in detail.

2. Experimental procedures

The chemical composition of tested steel is listed in Table 1, which is a low-carbon TRIP composition with Nb, V and Ti additions. The steel was melt in a vacuum induction furnace and then forged into a billet with transverse dimension of $60 \text{ mm} \times 60 \text{ mm}$. After homogenization at 1200 °C for 5 h, the billet was hot rolled to 4 mm with the finish rolling temperature of 830 °C. Then, the sheet was ultra-fast cooling to 650 °C and covered by asbestos to simulate coiling procedure. The final microstructure was composed of ferrite and pearlite. Subsequently, the sheet was cold rolled to 1 mm after pickling in 20 vol% hydrochloric acid. The critical temperatures of Ac1 and Ac3 were measured by dilatometry as 676 °C and 938 °C, respectively. Two kinds of heat treatments were applied in this article: isothermal bainite holding at 350 °C for 200 s with different intercritical annealing temperatures ranged from 750 °C to 900 °C (hereafter referred to as IBH-750-900) and typical two-step Q&P process with annealing temperatures of 850 °C and 880 °C (hereafter referred to as Q&P-850/880). The Q&P process consists of quenching to 250 °C for 10s after intercritical annealing, followed by partitioning at 380 °C for 100 s and finally water quenched to ambient temperature. The schematic diagrams of IBH and Q&P heat treatments are presented in Fig. 1. The tensile specimens were machined along the rolling direction with gauge length of $50\,\mathrm{mm}$ and width of 12.5 mm.

The secondary electron (SE) images were obtained using JEOL JXA-8530F electron probe micro-analyzer (EPMA) and corresponding samples were etched by 4% nital solution after mechanical polishing. Electron backscatter diffraction (EBSD) technique (step size: $0.05 \,\mu\text{m}$ or $0.1 \,\mu\text{m}$, tilt angle: 70°) was conducted in a Zeiss Ultra-55 field emission scanning electron microscope (FE-SEM) at 20 kV and the result data was post-processed by Channel 5 software. The specimens of EBSD were electro-polished using an $875 \text{ ml CH}_3\text{CH}_2\text{OH} + 125 \text{ ml HClO}_4$ solution at 20 °C and 22 V.

Characterization of microstructure was obtained by a FEI G2 F20 transmission electron microscope (TEM) with an operating voltage of 200 kV. In addition, the precession electron diffraction (PED) technique was performed using digital precession unit from NanoMEGAS (Digistar/ASTAR) equipped in TEM to obtain the orientation and phase distribution maps with nanometer-scale spatial resolution. The precession angle of 0.5° was applied and camera length was selected to be 71 mm [14]. The scanning step size was 10 nm. A ϕ 3 thin foil with 40 µm thickness was twin-jet polished using a solution of 12.5 vol% perchloric acid alcohol at - 25 °C and 32 V.

In order to investigate the retained austenite fraction and corresponding carbon content in austenite, a D/max 2400 X-ray diffractometer (56 kV, 182 mA) was applied with Cu K α radiation and a 20 range of 40–100° (step size: 2°/min). The measured samples were prepared the same as EBSD samples. The amount of retained austenite was calculated based on the measured peaks of (200)_{α}, (211)_{α}, and (200)_{γ}, (220)_{γ}, (311)_{γ}. Austenite carbon concentration can be obtained based on the peak of (220)_{γ} using an empirical equation. The detailed calculation method can be obtained in the authors' prior [15].

3. Results and discussion

3.1. Characterization and comparison of microstructure in different annealing treatments

The microstructures of different heat treatments were characterized by secondary electron images, as presented in Fig. 2. Meanwhile, metallographic method was used to obtain ferrite fraction and mean diameter of secondary phase region (referred to as SPR), which indicates the retaining austenite before IBH or partitioning process [16]. In particular, the martensite packet with high-angle grain boundary, which represents the actual dimension of martensite, was also regarded as a special SPR during statistical process. The volume fraction of martensite formed during first quenching process was calculated using the Koistinen-Marburger equation [17] and the corresponding Ms temperature was obtained by the following empirical equation [18]:

$$Ms(^{\circ}C) = 539 - 423C - 30.4Mn - 7.5Si + 30Al (in wt. \%)$$
(1)

Where the austenite composition was calculated by Thermal-calc based on practical ferrite fraction. The volume fraction of retained austenite was measured by XRD technique. All of the statistical results are summarized in Fig. 3.

In the cases of IBH-750 and IBH-780 (Fig. 2a), the deformed ferrite (DF) band is observed along the rolling direction and abundant amount



Fig. 1. Schematic diagrams of different heat treatments (a) IBH condition; (b) Q&P condition.

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