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The relationships of microstructure-mechanical properties in quenching and partitioning (Q&P) steel accompanied with microalloyed carbide precipitation



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ABSTRACT

A low carbon steel microalloyed with Nb-V-Ti were heat treated by means of two-step quenching and partitioning (Q&P) process after partial austenitization. Combined use of SEM equipped with EBSD, XRD and TEM showed that the microstructure was composed of intercritical polygonal ferrite, tempered lath martensite, secondary twin martensite and carbon-enriched retained austenite, as well as microalloyed carbides (mainly VC) precipitated inside ferrite and martensite. When increasing partitioning time from 0 s to 3600 s, the volume fraction of retained austenite increased up to 6.5% with mean carbon content in austenite keeping a constant level. The retained austenite keep K-S orientation relationship with adjacent tempered lath martensite and neighboring secondary twin martensite in different martensite packet. As partitioning time increased up to 3600 s, the initial lath martensite tempered gradually to ferrite without any cementite formation.

1. Introduction

As a novel heat treatment for advanced high strength steel (AHSS), the Q&P process starts with a full or partial austenitization followed by a fast quenching to martensite transformation region to form a controlled fraction of martensite. Subsequently, an isothermal partitioning step is done at the same (one-step Q&P) or a higher temperature (two-step Q&P) [1,2]. During the partitioning procedure, the carbon atoms in supersaturated martensite partition to neighbor austenite, which leads to carbon enriched austenite with higher chemical stability [1,3]. The Q &P process is ended by quenching to room temperature, and some austenite with insufficient stability will transform into fresh martensite. Consequently, an excellent combination of strength and ductility is obtained with final microstructure consisted of depleted martensite, and/or ferrite, fresh martensite and a considerable retained austenite [4,5].

The "constrained carbon equilibrium" (CCE) model has been suggested as a well description for the thermodynamics of carbon partitioning process [1], where the competing reactions are suppressed, including bainite transformation and carbide formation, which act as a sink of carbon that deteriorates the stability of austenite. Therefore, some alloy elements such as silicon and aluminum are used to achieve the retardation of carbide precipitation [2]. However, the carbide precipitation could not be suppressed entirely even if containing a high

amount of Si in some cases [6]. Based on the situation that the influence of carbide precipitation during Q&P treatment is lack of relevant knowledge, some researchers have currently focused on the interaction of carbide precipitation and carbon partitioning process, where the transition carbide and cementite have been well investigated [7,8].

In addition, some strong carbide forming elements like niobium, vanadium and molybdenum have also been added into the Q&P steel composition [9,10]. Surprisingly, excellent mechanical properties have been obtained due to the significant effects of grain refinement and carbide precipitation strengthening. In particular, some researchers has proposed a modified Q&P termed as Q-P-T process, which contains Nb and/or Mo complex carbides precipitated inside martensite [11]. Nevertheless, few articles focused on VC precipitation in Q&P steel are reported [5,10] and the competition relationship between microalloyed carbides and cementite formation, as well as the tempering behavior of martensite, has not been given enough attention.

Therefore, as studied in this article, the influence of micro-alloyed carbides on the carbon partitioning behavior and martensite decomposition process were researched by experimental and simulative methods. And an accurate description of the relationship between the evolution of microstructures containing microalloyed carbides (mainly VC) and the final mechanical properties have also been discussed.

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Table 1
Chemical composition of tested steel (wt%).

| С | Mn | Si | Al | Ti | Nb | v | Fe |
|------|------|------|------|-------|-------|------|------|
| 0.20 | 2.11 | 1.49 | 0.81 | 0.035 | 0.045 | 0.22 | Bal. |

2. Experimental procedure

The chemical composition of the studied alloy is listed in Table 1. It was a typical low-carbon TRIP-assisted steel microalloyed with niobium, vanadium and titanium. Silicon and aluminum was used to avoid cementite formation during partitioning process [12]. The addition of niobium, vanadium and titanium was applied to refine grains and strengthen the ferrite matrix [9–11]. A 90 kg ingot was cast after melting in a vacuum induction furnace, and then reheated at $1200\,^{\circ}\mathrm{C}$ for 2 h, followed by hot forging to 60 mm thickness. The forged slab

was heated up to 1200 °C for 4 h and then hot rolled to sheets with thickness 4 mm after seven passes rolling from 1150 °C to 830 °C. Subsequently, the sheet was ultra-fast cooling to 570 °C followed by furnace cooling. Finally, the sheet was cold rolled to strips with 1 mm thickness after pickling in 20 vol% hydrochloric acid. The tensile specimens were machined with their axis orientation parallel to the rolling direction and cut into the gage length of 50 mm with the width of 12.5 mm, followed by a consequent heat treatment shown in Fig. 1. The intercritical annealing temperature was selected to be 850 °C with 50 vol% ferrite, as a prediction of Thermal-calc software. The corresponding critical temperatures of M_s and M_f were measured by dilatometer analyzer to be 292 °C and 172 °C, respectively. The quenching temperature was determined to be 245 °C based on the CCE model prediction in following section. During the heat treatment process, specimens were firstly partial austenitized at 850 °C for 300 s, followed by quenching to 245 °C for 10 s. Subsequently, some of these specimens were as-quenched to room temperature to represent the state before partitioning step indirectly (hereafter referred to as "as-quenched specimen" or "partitioned for 0s"), and others were held at 380 °C for 10-3600s and then quenched to room temperature by water. Three specimens were prepared for each heat treating condition.

Microstructures were characterized by scanning electron microscopy (SEM) after mechanical polishing and etching in a 4% natal solution. Electron backscatter diffraction (EBSD) measurement (step size: $0.05\,\mu m$; tilt angle: 70°) were carried out after electrolytic polishing in an electrolyte solution at ambient temperature (7 parts alcohol and 1 part perchloric acid). The acquired data was post processed by Channel 5 software. Transmission electron microscopy (TEM) samples were obtained by punching from the same region of SEM and EBSD in

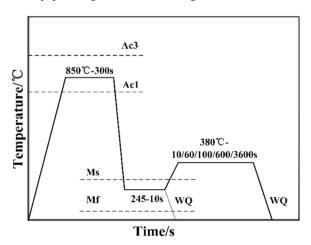


Fig. 1. Scheme of the applied Q&P heat treatment: Ac1, pearlite dissolution start temperature; Ac3, austenite formation finish temperature; M_s , martensite start temperature; M_f , martensite finish temperature; WQ, water-quenching.

specimens, and then ground to a thickness of 50 μ m followed by double-jet thinning at $-25\,^{\circ}$ C with operating voltage of 32 V using 10 vol% perchloric acid solution. The amount of retained austenite and corresponding average carbon concentration were measured by X-ray diffraction (XRD) with Cu K α radiation at room temperature. Samples were scanned in a 20 range from 40° to 101° at a scanning rate of 2°/min. The specimens were prepared the same as EBSD samples (operated at 10 v for 25 s). Quantification of the retained austenite volume fraction was obtained by collecting the peak intensities of $(200)_{\gamma}$, $(220)_{\gamma}$, $(311)_{\gamma}$, $(200)_{\alpha}$ and $(211)_{\alpha}$. The austenite carbon concentration was calculated using the following equation [12].

$$C_{\gamma} = (\alpha_{\gamma} - 3.547)/0.046$$
 (1)

Where C_{γ} and α_{γ} are the carbon concentration of austenite in weight percent and the lattice parameter of austenite in Angstroms, respectively. The lattice parameter could be obtained based on a certain austenite peak which is (200) in this article and the method is presented as Eq. (2):

$$\alpha_{\gamma} = \lambda \sqrt{h^2 + k^2 + l^2} / (2\sin\theta_{hkl}) \tag{2}$$

Where λ , (h k l) and θ_{hkl} are the wavelength of the radiation, the three Miller indices of a plane and the Bragg angle, respectively.

The volume fraction (V) and the mean planar intercept diameter (D) of carbides were obtained based on 15 different TEM micrographs as the distribution of the carbides was inhomogeneous. Note that, only the data of carbide precipitated inside ferrite had been measured, whereas that of carbide inside martensite was too difficult to measure effectively. The precipitation strengthening effect (σ_P) of the microalloyed carbides could be calculated by the following equation [13].

$$\sigma_{\rm p} = 6.66/\text{L} \cdot \ln(\text{D}/(4.96 \times 10^{-4})) \tag{3}$$

Where L is the surface to surface carbide spacing in micro meter and can be calculated from the following equation.

$$L = D[(\pi/4V)^{0.5}-1]$$
 (4)

The work hardening behavior was studied by using Hollomon equation [14]:

$$\sigma_{t} = K \varepsilon_{t}^{n_{i}} \tag{5}$$

Where σ_t and ϵ_t are the true stress and true strain, respectively, K is the strength coefficient, and n_i is the instantaneous work hardening exponent.

The value of n_i can be deduced from Eq. (6):

$$n_{i} = (\varepsilon_{t}/\sigma_{t})(d\sigma_{t}/d\varepsilon_{t})$$
(6)

3. Results

3.1. Theoretical analysis of microalloyed carbides precipitation and corresponding trapped carbon contents

Fig. 2a shows the variation of phase fraction as a function of temperature by Thermal-calc calculation. Two kinds of carbides are found to be existed in 850 °C, which can be identified to be VC and (Ti,Nb)C₂ from the atom ratios of corresponding element components. The carbon that trapped in VC and (Ti,Nb)C₂ can be calculated from the result of Fig. 2a. Assumed that all of the carbon in ferrite is transferred into austenite or trapped in micro-alloyed carbides and the carbide precipitation is proceed completed, the trapped carbon content in micro-alloyed carbides can be estimated to be $\sim\!0.05$ wt% as Fig. 2b shows. The corresponding carbon concentration in austenite ($X_{\rm C}^{\rm RA}$) is ~0.3 wt% when annealed at 850 °C, whereas the initial carbon concentration in alloy ($X_{\rm C}^{\rm alloy}$) is 0.2 wt%.

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