On structure-property relationship in nanostructured bainitic steel subjected to the quenching and partitioning process

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Abstract

We elucidate here the mechanistic contribution of the application of quenching and partitioning (Q&P) concept to a high carbon Mn-Si-Cr steel in obtaining a multiphase microstructure comprising of martensite/austenite and nanostructured bainite (bainitic ferrite and nanometer-sized film-like retained austenite) that exhibited tensile strength of 1923MPa and total elongation of 18.3%. The excellent mechanical properties are attributed to the enhanced refinement of blocky austenite islands obtained by the Q&P process. The austenite was stabilized by both carbon partitioning from martensite and bainite transformation. Compared with conventional heat treatment to produce nanostructured bainite, the total time is significantly reduced without degradation of mechanical properties.

Keywords: bainitic steel; Q&P process; Retained austenite; Mechanical property

1. Introduction

The advanced high strength steels (AHSSs) with high ductility have been extensively studied with the aim of saving energy and raw materials and conserving the environment by decreasing the weight of steel components [1-4]. In this context, fine and dispersed retained austenite (RA) plays a significant role in balancing strength and ductility, which is attributed to transformation-induced plasticity (TRIP) effect [5-9].

A number of studies on microstructure-mechanical property relationship indicated that a major factor that influences the mechanical properties of steel is the stability of the retained austenite. Moreover, the stability of retained austenite is influenced by (i)

the local carbon content in austenite [10-11], (ii) the constraining effect of phases surrounding the austenite [12-13], (iii) the grain volume of austenite [14], and (iv) the morphology [15-16]. Recently, some studies suggested that the size and morphology of RA are dominant factors in the stability of austenite because the carbon-content in the center of the blocky austenite was relatively low, and the larger blocky austenite easily transformed to martensite at small strains [17-19]. Consequently, a number of studies were carried out to tailor the morphology and size of the austenite by changing process parameters [15-19].

Quenching and partitioning (Q&P) process is viewed as an effective heat treatment to obtain microstructure with adequate amount of retained austenite [6, 9, 20]. Q&P heat treatment involves the following steps: (i) an initial quenching step to a temperature (T_q) between martensite-start temperature (M_s) and martensite-finish temperature (M_f) to form a certain fraction of carbon saturated martensite ; (ii) a partitioning step at T_q or higher than T_q to allow carbon to diffuse from martensite to austenite to enhance the stability of austenite. Consequently, a microstructure consisting of carbon-depleted martensite and carbon-stabilized austenite is obtained.

Some studies also focused on refining austenite through alloy design and tailoring the process parameters (such as quenching temperature and partitioning temperature). However, the Q&P process was mainly used to enhance the mechanical properties of low and medium carbon steels, and the application of Q&P process to high carbon steels is rare [21-25].

In the present study, the Q&P concept was successfully applied to a high carbon Mn-Si-Cr steel (0.66C-2.2Mn-1.7Si-0.5Cr) in obtaining a multiphase microstructure comprising of martensite/austenite and nanostructured bainite (bainitic ferrite and nanometer-sized film-like retained austenite). The primary aspect of the process is that an appreciable fraction of nanostructured bainite was obtained during the partitioning step. The martensite plates not only divided the untransformed austenite but also accelerated the bainitic transformation. More importantly, the austenite was refined by the application of Q&P process. We elucidate here the mechanism of stabilization of austenite and the relationship between microstructure and mechanical properties. To compare and contrast, conventional quenching-tempering and bainite austempering processes were also simultaneously studied.

2. Experimental procedure

The nominal chemical composition of the experimental steel was Fe-0.66C-2.2Mn-1.7Si-0.5Cr (in wt. %). Si was added to suppress the precipitation of carbides during quenching and isothermal heat treatment. The alloy studied was in the form of forged plate. The forging process was carried out as follows: An ingot of 50 kg was reheated to 1200°C and forged to 30mm thickness and the finishing-forging temperature was ~950°C. The forged bars were subjected to homogenization at 1200°C for 12 h. After homogenization, the bars were annealed at 900°C for 2h, followed by furnace cooling. The microstructure of the samples after the annealing treatment

consisted of ferrite and pearlite (i.e. prior to all the following experiments), as shown in Fig.1. The annealing is employed before heat treatment in order to eliminate the microstructure heredity and obtain relatively refined microstructure [26].

The dilatometry experiments were carried out to capture the transformation temperature and microstructural evolution using a dilatometer (Bähr D805L) equipped with quartz push-rods. The steel samples were machined to cylindrical specimens of dimension 4 mm diameter and 10 mm height. To control the temperature, thermocouples of type S were welded to the surface of the specimens in the center.

Four different heat treatments were carried out with the specimens (15 mm × 30 mm × 70 mm in size), as shown in Fig.2. The conventional Q&T process was as follows: cooling to ambient temperature (30°C) at a cooling rate of about 1-2 °C /s after austenization at 880 °C for 45 min and then tempered at 250°C for 12 h (Fig.2a). The BAT-12 samples correspond to an isothermal bainite transformation process: after austenitization at 880 °C for 45 min, the samples were cooled to 250°C (above the M_s temperature), and then austempered for 12 h (Fig.2b). Compared to the BAT-12 sample, the austempering time of BAT-24 samples was extended to 24h (Fig.2c). The QPB process consisted of cooling to 130°C (below M_s temperature) after austenitization, followed by an isothermal heat treatment at 250°C for 12 h.

Microstructural characterization was carried out using a combination of scanning electron microscopy (SEM; Zeiss EVO18, 20 kV) and transmission electron microscope (JEOL 2010, 200 kV). The samples for SEM were polished and etched with

2% nital solution. TEM studies were carried out using thin foils electropolished at -40°C using 4% perchloric acid solution. The microstructure is analyzed through image processing software (Photoshop 7.0).

The volume fraction of retained austenite (RA) was measured by X-ray diffraction (XRD; Rigaku Smartlab, Cu K α radiation) at a step of 0.02° and a counting time of 2 s per step. Rietveld analysis with MAUD software was used to calculate the diffraction data. The volume fraction of retained austenite was calculated after collecting the peak intensities of (200) γ , (220) γ , (311) γ , (210) α and (211) α . Since the carbon concentration plays an important role in governing mechanical and thermal stability of retained austenite, the carbon content in retained austenite was calculated using the following equation [27]:

$$x_c = \frac{\frac{\lambda_{k\alpha}}{2\sin\theta} \times \sqrt{8} - 3.572 - 0.0012x_{Mn} + 0.00157x_{Si} - 0.0056x_{Al}}{0.033} \quad (1)$$

where θ and $\lambda_{k\alpha}$ stand for the angle of the (220) γ plane and the wavelength of X-ray Cu target with a value of 0.15406nm; x_c , x_{Mn} , x_{Si} , x_{Al} are the chemical composition in retained austenite (in wt%).

Standard tensile samples with a gage diameter of 5 mm and gage length of 25 mm were used to determine the mechanical properties of the steels after each heat treatment using a SUNS 5305 tensile tester (MTS System, China). Three samples were tested for each condition and the average values obtained. An extensometer and a force sensor were used. The work hardening behavior was studied by applying the following classical Hollomon equation:

$$\sigma_t = K \varepsilon_t^{n_i} \quad (2)$$

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 instantaneous work hardening exponent, n_i , deduced from equation (2) is:

$$n_i = \left(\frac{\varepsilon_t}{\sigma_t} \right) \left(\frac{d\sigma_t}{d\varepsilon_t} \right) \quad (3)$$

3. Results

3.1 Microstructure

The microstructures of steels after each heat treatment are presented in Fig. 3 and 4. The microstructure of Q&T samples comprised of martensite, austenite, and a very small amount of nanostructured bainite (bainitic ferrite plus RA) (Fig. 3a). The bainitic ferrite (marked in Fig. 3a) was formed during tempering [28]. The BAT-12 microstructure consisted of thin bainitic ferrite and austenite which included film-like austenite and blocky austenite (not M/A island, analyzed in section 4.1) (Fig. 3b). The size of film-like austenite was around 20-100 nm (shown by TEM image, Fig.4a), while that of blocky austenite was around 1-2 μ m. It is suggested from Fig.3b and c that there were insignificant differences in the microstructure of BAT-12 and BAT-24 samples, and is discussed below. The microstructure of QPB samples (Fig. 3d) comprised of martensite and nanostructured bainite and RA. The nanostructured bainite (bainitic ferrite plus RA) was produced during partitioning step (shown by TEM images in Fig.4b). From the SEM micrographs, we can identify some blocky austenite in the BAT samples, while the blocky austenite was reduced and even eliminated in QPB samples,

consistent with the previous work [29]. There was small fraction of lath austenite (as marked in Fig. 3d) whose size in terms of width was about 0.2-0.4 μ m. The underlying reasons are: (i) the formation of martensite provides more nucleation sites for bainitic ferrite; (ii) during initial quenching step some austenite transformed to martensite, which divides the austenite grains and reduces the size of untransformed blocky austenite; (iii) the formation of martensite results in the plastic deformation of austenite near the pre-formed martensite and plastic deformation promotes transformation of austenite to bainite and refines the austenite [30-31].

3.2 Volume fraction of retained austenite and its carbon content

The total volume fraction of retained austenite obtained from XRD and the carbon content in austenite was calculated using Equation 1 after each heat treatment and is presented in Fig. 5. The volume fraction of austenite in Q&T samples was consistent with the K-M equation and the carbon content in austenite was higher than the average amount in the alloy (analyzed below). Compared with the BAT-12 sample, the amount of carbon in retained austenite in BAT-24 sample was higher but the volume fraction of austenite was lower. But the fraction of austenite and the enrichment of carbon in austenite in BAT-12 and QPB samples were similar. Although the fraction of austenite in QPB was large than that in BAT-24, the carbon content in austenite of BAT-24 samples was higher than that in QPB samples.

3.3 Mechanical properties

The mechanical properties after each heat treatment are listed in Table 1. The

strength (yield strength and ultimate tensile strength) of Q&T specimens was highest among all the samples and the strength of BAT samples was higher than QPB specimens. However, the ductility (elongation) varied depending on the heat treatment. The QPB samples showed an excellent ductility, which contributed to favorable combination of strength and ductility such that the product of strength and elongation (PSE) was 35.2GPa%. Even though the fraction of austenite in BAT-12 samples was higher than BAT-24 samples, the elongation of BAT-24 was larger than BAT-12 sample (discussed below). However, the ductility of Q&T samples was poor.

4. Discussion

4.1 The volume fraction of blocky and filmy austenite in BAT-12 samples

Generally speaking, the formation of isothermal bainite promotes two morphologies of untransformed austenite: blocky and film-like austenite [32-33]. The film-like austenite is located between the nanostructured bainitic ferrite laths (Fig.4a), while the blocky austenite is located between bainitic sheaves (consist of a group of bainitic ferrite lath and RA located parallelly, marked by dotted line in Fig.3b). In general, the carbon content in blocky austenite is low, which affects the stability of austenite during deformation [34]. Thus, the fraction of blocky and film-like anstenite in BAT-12 samples was calculated and the relation between them can be expressed as follows [35]:

$$\frac{V_{f\gamma}}{V_{b\gamma}} = \frac{0.15V_{BF}}{1 - 0.15V_{BF}} \quad (4)$$

where $V_{f\gamma}$ and $V_{b\gamma}$ are the volume fraction of film-like austenite and blocky austenite,

respectively, and V_{BF} is that of bainite ferrite. $V_{f\gamma}$ and $V_{b\gamma}$ were calculated to be 0.12 and 0.11.

4.2 The carbon enrichment of austenite

As mentioned above, the stability of austenite is influenced by the carbon-content in austenite. Thus, the mechanism of carbon enrichment needs some discussion. Bainitic ferrite can be formed during tempering and the carbon can be partitioned from martensite to austenite, which makes the carbon content in retained austenite higher than the average amount in the alloy. The relative change in length ($\Delta L/L_0$; ΔL refers to the change in length during Q&T heat treatment and L_0 is the initial length of the dilatometry specimens) with respect to the temperature of Q&T heat treatment simulation is presented in Fig. 6a. It is suggested that length reduces during heating from the ambient temperature to the tempering temperature (indicated with arrow). It is believed that it is mainly due to segregation of carbon around the dislocations because of low temperature formation of Fe-C clusters and carbides in martensite. The formation of high content of martensite results in high dislocation density. Timokhina's research showed that the high density of dislocation promotes the formation of Fe-C clusters and even carbides in BCC phase (bainitic ferrite or martensite) [36]. This can also explain the reason why both the fraction of austenite and carbon content in austenite is lower than BAT and QPB samples.

The dilatometry plot in Fig. 6b represents the simulation of BAT heat treatment. There was no trace of martensite transformation during the cooling step after isothermal

transformation at 250°C for 12 h, which suggests that the carbon atoms diffused to austenite from bainitic ferrite and stabilized the austenite to the ambient temperature during the finally cooling stage. Fig. 6d indicated that the bainite transformation was nearly completed after isothermal treatment at 250°C for 12 h, which is also the reason why the microstructure of BAT-12 and BAT-24 samples was similar. However, it takes a long time to homogenize carbon in blocky austenite and the martensite start temperature of untransformed austenite varies with isothermal time [37]. The average time required for carbon diffusion in austenite (carbon homogenization in austenite) can be estimated by equation (4) [38]:

$$t = \frac{\bar{l}^2}{6D} \quad (5)$$

where $\bar{l}$ is the average diffusion distance (here defined as the half-width of and blocky austenite, namely, $\bar{l}_{\gamma BAT} = 1\mu m$, D is the diffusion coefficient of carbon which equals $D_0 \exp(-Q/RT)$, where D_0 is a constant ($D_{0\gamma} = 0.62 * 10^{-6} m^2 / s$ [39]), Q is the activation of carbon diffusion ($Q_{\gamma} = 135.7 kJ / mol$ [39]), R is the gas constant, and T is the absolute temperature, here, $T = 523K$ (250°C).

From equation (4), the average time required for carbon homogenization in blocky austenite was calculated to be 168h for BAT samples. Thus, some carbon atoms in BAT samples may still be trapped between bainitic ferrite and its adjacent block austenite and the carbon content in the center of blocky austenite was low such that the austenite transforms to martensite at small strain [40-41].

However, the mechanism of enrichment of carbon atoms in retained austenite is

different for QPB and BAT samples. It is discussed in detail as follows:

The amount of martensite and austenite at each quench temperature below the M_s temperature (prior to partitioning of carbon during partitioning step) can be predicted using the K-M relationship:

$$f_m^{Tq} = 1 - \exp(\alpha_m * (Ms - Tq)) \quad (6)$$

where f_m^{Tq} refers to the fraction of martensite at the quench temperature (Tq). α_m is the rate parameter that depends on the chemical composition [42]:

$$\alpha_m = 27.2 - \sum_i S_i x_i - 19.8[1 - \exp(-1.56x_c)] \quad (7)$$

$$\sum_i S_i x_i = 0.14x_{Mn} + 0.21x_{Si} + 0.11x_{Cr} + 0.08x_{Ni} + 0.05x_{Mo} \quad (8)$$

where x_c , x_{Mn} , x_{Si} , x_{Cr} , x_{Ni} , x_{Mo} are the chemical composition in austenite (in weight) and α_m is in $10^{-3} K^{-1}$. When the sample is quenched to the ambient temperature ($30^\circ C$), the fraction of retained austenite is ~ 0.17 which is similar to the XRD data (0.18). Consequently, the K-M relationship can be expressed as:

$$f_m^{Tq} = 1 - \exp(-0.013 * (160 - Tq)) \quad (9)$$

For QPB samples, the quench temperature was $130^\circ C$ and the fraction of martensite ($f_m^{130^\circ C}$) was ~ 0.32 and at this point the fraction of untransformed austenite is ($f_\gamma^{130^\circ C}$) was about 0.68.

Theoretical amount of retained austenite anticipated after partitioning was calculated assuming full partitioning of carbon from martensite to austenite during the partitioning step of QPB process or by assuming carbide-free bainite formation during the partitioning step [43].

K-M relationship was applied to initial quenching and final quenching step to ambient temperature, and a theoretical final austenite fraction (solid line) at each quench temperature based on full partitioning of carbon from martensite to austenite during partitioning step is presented in Fig.7.

From the results of BAT-12, it can be deduced that ~ 0.77 ($V_{BF}=1-V_{\gamma}$, and $V_{\gamma}=0.23$) austenite transformed to bainitic ferrite during the isothermal heat treatment at 250°C for 12 h in BAT-12 sample. Thus, if the effect of prior martensite on transformation of austenite to bainitic ferrite (such as diffusion of carbon from martensite to austenite) during isothermal (partitioning) stage is ignored, the fraction of bainitic ferrite (f_{α}^{BF}) can be calculated by the following equation:

$$\frac{0.77}{1} = \frac{f_{\alpha}^{BF}}{f_{\gamma}^{130^{\circ}\text{C}}} \quad (10)$$

From Fig. 6c, we can deduce that martensite was not formed during the second quenching step. Thus, the fraction of austenite (f_{γ}) must satisfy the following equation:

$$f_{\alpha}^B + f_m^{130^{\circ}\text{C}} + f_{\gamma} = 1 \quad (11)$$

The fractions of austenite and bainitic ferrite are 0.15 and 0.52 (in Fig. 7, open circle), respectively.

Experimental austenite fraction determined from XRD was 0.24 (Fig.7, open triangle). The comparison in Fig. 7 shows that the experimental austenite fraction was higher than the value predicted based on bainite transformation and is lower than the value predicted based on complete carbon partitioning from martensite, which suggests that either bainite transformation or complete partitioning of carbon from martensite

was responsible for the experimentally determined enriched austenite. Thus, the experimental austenite was stabilized based on both carbon partitioning from martensite and bainite transformation.

Similarly, the homogenizing time for carbon in lath austenite in QPB samples can be predicted to be 7h by equation 5. Thus, the carbon atoms in QPB samples are more homogeneous than BAT samples.

4.3 The relationship between microstructure and properties

From the foregoing discussion, the phases present after each heat treatment and the size of austenite as well as the carbon content in austenite is presented in Table 2.

Given that the strength of martensite is higher than bainite, the strength (yield strength and ultimate tensile strength) of Q&T specimens was highest among all the samples. However, the behavior of ductility (elongation) was different because of different heat treatments. Compared with BAT and Q&T samples, the ductility of QPB samples was the highest. The relatively large fraction of austenite in QPB led to superior elongation. This suggests that the TRIP effect plays a vital role in elongation. Although the fraction of austenite in BAT-12 samples was higher than BAT-24 samples, the elongation of BAT-24 was larger than BAT-12 sample, which is mainly because the austenite in BAT-24 was more stable because of higher carbon content. The formation of carbon clusters as well as carbide formation during tempering of Q&T samples reduced the ductility of Q&T samples.

The true strain-tress plot and instantaneous work hardening exponent as a function

of true strain are presented in Fig. 8. The true stress-strain plot of QPB indicated a long flat stage with increase in strain. This is primarily because of the TRIP effect of retained austenite. The work hardening parameter was constant in QPB even at relatively large strain. This implied that austenite transformed to martensite continuously during deformation. But the work hardening exponent of BAT-12 samples was larger than QPB specimens at small strain, which suggested that during initial deformation, more austenite in BAT-12 samples transformed to martensite, and contributed very less to the TRIP effect. Although the carbon in austenite was similar between BAT-24 sample and QPB samples, but the morphology of austenite was significantly different. Blocky austenite was observed in BAT samples. Many studies have shown that the blocky austenite is relatively unstable compared to film-like austenite, which also explains why the ductility of QPB was better than BAT-24. The film-like austenite was more stable so that the austenite could transform to martensite (contribute to TRIP effect) at large strain during deformation. This is the underlying reason for good ductility. Last but not least, compared with Q&T samples, if we consider the transport of carbon from martensite to austenite during the partitioning step, martensite after QPB treatment is relatively ductile [44], which is also the reason why the elongation is the highest.

In summary, the carbon content in retained austenite and the morphology of retained austenite influences the stability of austenite and the film-like austenite with relatively high carbon content contributed to the excellent mechanical properties.

4. Conclusions

A high carbon nanostructured bainitic steel processed by a novel quenching and partitioning process exhibited excellent combination of strength and ductility (PSE: 35.2 GPa%). A multiphase microstructure was obtained by the novel quenching and partitioning process of the nanostructured bainitic steel that contained martensite and nanostructured bainite (nanoscale bainitic ferrite and austenite). Importantly, the blocky austenite was rare after the novel quench and partitioning treatment. The film-like austenite with relatively high stability transformed to martensite continuously during deformation, with consequent increase in ductility. Compared with conventional direct isothermal nanostructured bainite, the isothermal time for nanostructured bainitic ferrite formation of QPB was reduced from 24 h to 12 h without loss in mechanical properties.

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Fig.1 The microstructure of the experimental steel after the annealing treatment.

Fig.2 Schematic illustration of different heat treatment: (a) Q-T, (b) BAT-12, (c) BAT-24 and (d) QPB (RT: room temperature about 30°C).

Fig.3 SEM micrographs of (a) Q-T, (b) BAT-12, (c) BAT-24 and (d) QPB samples; M: martensite, RA: retained austenite, B: nanostructured bainite, BF: bainitic ferrite; the dotted line in Fig.3b showing the bainitic sheaf

Fig.4 TEM micrographs of (a) BAT-12 and (b) QPB sample, RA: retained austenite, BF: bainitic ferrite

Fig.5 Volume fraction of austenite and its carbon content after each heat treatment.

Fig.6 Experimental dilatometric plots of different processes:(a) the relative change in length as function of temperature of Q-T; (b) the relative change in length as function of temperature of BAT; (c) the relative change in length as function of temperature of QPB; (d) the relative change in length as function of time during isothermal stage of BAT and QPB.

Fig.7 Comparison of experimental austenite fraction with the calculated austenite fraction assuming the two mechanisms and the data obtained from XRD.

Fig.8 (a) true strain-stress plots and (b) instantaneous work-hardening exponent as a function of true strain for QT, BAT-12, BAT-24 and QPB samples

Table 1. Mechanical properties of Q-T, BAT-12, BAT-24 and QPB samples.

Samples	Rp0.2_(MPa)	Rm_(MPa)	UEL_(%)	TEL_(%)	PSE_(GPa.%)
Q-T	1638 $\pm$ 28	2055 $\pm$ 12	3.7 $\pm$ 0.1	3.9 $\pm$ 0.2	8.0
BAT-12	1287 $\pm$ 15	2008 $\pm$ 17	11.7 $\pm$ 0.2	12.7 $\pm$ 0.4	25.5
BAT-24	1378 $\pm$ 9	1932 $\pm$ 7	11.3 $\pm$ 0.1	15.1 $\pm$ 0.2	29.2
QPB	1285 $\pm$ 16	1923 $\pm$ 2	14.6 $\pm$ 0.2	18.3 $\pm$ 0.8	35.2

Rp0.2: yield strength; Rm: tensile strength; UEL: uniform elongation; TEL: total elongation; PSE: the product of tensile strength and total elongation.

Table 2. The phases and the size of austenite after each heat treatment.

Samples	Size of RA (μm)		Volume fraction of each phase					C
	Filmy RA	Blocky RA	V _γ	V _{BF}	V _M	Vf _γ	V _{bγ}	(wt.%)
Q&T	-	-	0.18	0	0.82	-	-	0.99
BAT-12	0.1-0.7	1-2	0.23	0.77	0	0.12	0.11	0.15
BAT-24	0.1-0.7	1-2	0.21	0.79	0	0.11	0.10	1.30
QPB	0.2-0.4	-	0.24	0.44	0.32	0.24	0	1.25