High Strength-Ductility Nb-microalloyed Low Martensitic Carbon Steel: Novel Process and Mechanism

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Received: 3 August 2015 / Revised: 16 September 2015 / Published online: 12 October 2015
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Abstract A low-carbon Nb-microalloyed Fe–Mn–Si-based steel was treated by a novel quenching–partitioning–tempering (Q–P–T) process as a modified quenching and partitioning (Q&P) process. After processed by Q–P–T treatment, this steel exhibits excellent mechanical properties such as high product of strength and elongation. The addition of Nb markedly raises both the yield strength and tensile strength of Q–P–T martensitic steel, especially the yield strength, which can be attributed to the strong grain refinement strengthening and precipitation strengthening of Nb. The Nb addition can also lead to a little increase in ductility. The Nb-microalloyed steel treated by Q–P–T process displays much higher ductility than that treated by traditional quenching and tempering (Q&T) process. The mechanisms of Q–P–T process on ductility enhancement were fully analyzed and can be attributed to high quenching temperature and considerable amount of retained austenite.

KEY WORDS: Heat treatment; Mechanical properties; Microstructure; Microalloyed steel; Martensitic transformation

1 Introduction

In the past decade, more and more advanced high-strength steels (AHSS) are being used in the automotive industry for reducing vehicle weight and increasing fuel efficiency without sacrificing passenger safety [1]. The development of AHSS is focused on high strength together with sufficient ductility. Therefore, several kinds of low-alloying Fe–Mn–Si-based AHSS have been studied intensively, such as dual-phase (DP) steels consisting of ferrite and

martensite [2] and transformation-induced plasticity (TRIP) steels consisting of ferrite, bainite and retained austenite [3, 4], quenching and partitioning (Q&P) steels consisting of martensite and retained austenite [5–9] as well as quenching–partitioning–tempering (Q–P–T) steels consisting of martensite, retained austenite and carbide [10]. In AHSS, Q&P steels and Q–P–T steels show the highest strength and enough ductility because the martensite matrix has higher strength than ferrite matrix in DP or TRIP steels; meanwhile, considerable amount of retained austenite plays an important role in ductility enhancement. Speer et al. [5] proposed novel Q&P process in 2003, where a Si-containing steel was subjected to quenching from austenitizing temperature to a temperature (T_q) between the martensite-start (M_s) temperature and martensite-finish (M_f) temperature, followed by a “partitioning” treatment either at (one-step Q&P) or above (two-step Q&P) T_q temperature. During “partitioning,” the carbon diffuses from the supersaturated martensite phase to the untransformed austenite phase, and thus carbon-

Available online at <http://link.springer.com/journal/40195>

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enriched retained austenite can be stabilized in subsequent cooling to room temperature. For low- or medium-carbon Q&P steels, the quenching temperature T_q is usually much higher than room temperature [5], and thus considerable retained austenite can be obtained. Based on Q&P process and constrained carbon paraequilibrium (CCE) theory, Hsu [10] proposed the Q–P–T process in 2007. Additional carbide-forming elements such as Nb and Mo are added into Q–P–T steels, which are not permitted in CCE theory of Q&P process, and the addition of these elements leads to carbides formation and grain refinement for precipitation and grain refinement strengthening [11]. Since the Q–P–T process contains the essentials of Q&P process: quenching temperature (T_q) determined by combination of CCE theory and Koistinen–Marburger (K–M) equation [12], the Q–P–T martensitic steels also have considerable retained austenite comparing with traditional quenching and tempering (Q&T) steels (where T_q is room temperature in Q&T process). In investigating the first Q–P–T steel, the precipitation strengthening of Nb by tempering was determined as 130 MPa [13]. Although this medium-carbon steel treated by Q–P–T process exhibits the tensile strength of high up to 2000 MPa, high Nb content (0.2 wt%) results in poor ductility of about 10%, and the product of strength and elongation (PSE) is only about 20 GPa% [13]. After that, high strength–ductility medium-carbon Q–P–T steel with over 30 GPa% [14] and low-carbon Q–P–T steel with over 20 GPa% [15] were developed. In developing high strength–ductility Q–P–T steels, the effect of retained austenite on ductility enhancement was focused and studied. Zackay *et al.* [16] proposed TRIP effect in 1967, where the strain-induced martensitic transformation from retained austenite relaxes the stress concentration and redistributes the stresses during plastic deformation and thus delays the necking till the high strain region [17]. The occurrence of TRIP effect in Q–P–T was verified by experiments. For the effect of retained austenite on the toughness of high-strength steels, Webster proposed that the retained austenite blocking crack propagation (BCP) in 1968 [18], i.e., the cracks either propagate continuously in the austenite or trace a zigzag path through interlocking martensite laths, both of which will require an increased energy for fracture. The thin austenite layers between martensite laths could reduce the stress concentration at the crack tip and improve the fracture toughness [19, 20]. Recently, we found a new effect of retained austenite on ductility enhancement in studying the medium-carbon Q–P–T martensitic steel. Based on the measurement of average dislocation densities in both martensite and retained austenite by using X-ray diffraction line profile analysis (XLPA) [21], a named DARA (dislocation absorption by retained austenite) effect has been proposed, i.e., dislocations in martensite move into nearby

retained austenite through martensite-retained austenite interfaces, and these dislocations are absorbed by retained austenite during the deformation process. The possibility of dislocations transmitted from bcc-phase into the fcc-phase has been verified by molecular dynamic simulations in Cu–Nb bi-layered film [22]. DARA effect makes hard phase, martensite, be a “softening” state during the deformation and thus effectively enhances the deformation ability of martensite matrix. Two empirical conditions for remarkable DARA effect were further proposed [23]: (1) enough amount of retained austenite, larger than 10% in volume fraction; (2) coherent or semi-coherent interface between two phases. These two conditions were confirmed by low-carbon Q–P–T steel [21], bainitic steel [24] and high-carbon Q–P–T steel [25], respectively, where the coherent or semi-coherent interface between martensite (or bainite) and retained austenite shows either K–S or N–W orientation relationship. Besides, the dynamic compression behavior [26] and the elevated temperature properties of low-carbon Q–P–T steel with addition of Nb element [27] were further investigated for extending their possible application in engineering.

So far, there are still three issues to be solved in the study of Q–P–T steels: (1) Does the strengthening effect of Nb stem mainly from precipitation or grain refinement strengthening? (2) Does the addition of Nb certainly result in the loss of ductility? (3) Why novel Q–P–T process can markedly enhance ductility? In this study, therefore, two kinds of steels (Nb-microalloyed and Nb-free) were designed and treated by the Q–P–T, Q&P and Q&T processes, respectively. The above issues will be further explored by characterization of their mechanical properties and microstructures.

2 Experimental

Two kinds of low-carbon steels containing Nb or not are abbreviated as Nb03 and Nb00, respectively, and their chemical compositions are listed in Table 1 where their A_{c3} , M_s and M_f temperatures are included measured by a Formaster FII dilatometer. The composition design for the two kinds of steels is based on the following aspects. The addition of Mn is to stabilize retained austenite and provides adequate hardenability, while the addition of Si is to inhibit the formation of brittle Fe_3C Carbide. The addition of 0.03 wt% Nb to Nb03 steel was used to further raise strength by precipitation and grain refinement strengthening. The designed steels were melted in a laboratory medium frequency furnace and hot-rolled to a slab of 12 mm thickness. The samples of 2 mm in thickness were cut from a hot-rolled plate and then were treated by Q–P–T, Q&T and Q&P processes, respectively. Three different

Table 1 Chemical compositions of experimental steels (in wt pct)

Steel	C	Mn	Si	Nb	Fe	A_{c3} (°C)	M_s (°C)	M_f (°C)
Nb00	0.2	1.54	1.58	—	Bal.	904 ± 5	415 ± 5	173 ± 3
Nb03	0.19	1.52	1.57	0.029	Bal.	911 ± 5	395 ± 5	171 ± 3

heat treatment processes are schematically shown in Fig. 1. The samples were austenitized at 940 °C for 300 s, followed by quenching into salt bath at 300 °C for 15 s and subsequent partitioning/tempering at 450 °C for 30 s in molten salt, and finally quick quenching into water. The above quenching temperature (300 °C) was determined based on CCE theory/K–M equation after a slight modification. The above process is called Q–P–T process for Nb-microalloyed samples, and Q&P process for Nb-free samples. For Q&T process, the Nb00 steel and Nb03 samples were austenitized at 940 °C for 300 s, followed by water quenching to room temperature, then tempering at 450 °C for 30 s in molten salt. The difference between Q–P–T (or Q&P) process and Q&T process just lies in quenching temperature (T_q), and T_q is room temperature in Q&T process.

The samples for tensile test were prepared with a gauge length of 15 mm and width of 5 mm. Tensile tests were carried out on a Zwick T1-FR020TN A50 testing machine with a strain rate of 10^{-3} /s at room temperature. The tensile axis was parallel to the rolling direction. The fracture morphologies were observed on a JSM-6460 scanning electron microscope (SEM). The microstructure of the specimens was further characterized by transmission electron microscopy (TEM, JEM-2100F, 200 kV) after electropolishing by a twin-jet polisher in 4% perchloric acid

and 96% ethanol solution at –20 °C. The volume fraction of retained austenite was determined by X-ray diffraction (XRD) with CuK_α radiation in a D/max-2550 X-ray diffractometer on the basis of direct comparison of integrated intensity of the austenite peaks $(200)_\gamma$, $(220)_\gamma$ and $(311)_\gamma$ with martensite peaks $(200)_\alpha$ and $(211)_\alpha$.

3 Results

The tensile curves of the two kinds of low-carbon steels treated, respectively, by Q–P–T, Q&P and Q&T processes are shown in Fig. 2. Table 2 lists the mechanical properties of the specimens treated by Q–P–T, Q&P and Q&T processes, including yield strength ($\sigma_{0.2}$), tensile strength (σ_b), uniform elongation (δ_u) and total elongation (δ_b), as well as PSE ($\sigma_b \times \delta_b$). The results indicate that the strength of Nb-microalloyed steels is always higher than that of Nb-free steel, especially the yield strength. For example, the $\sigma_{0.2}$ and σ_b of Nb03 Q&T sample are 1227 and 1337 MPa, respectively, which are higher than 1149 and 1266 MPa for Nb00 Q&T sample, respectively. For the same process of Q–P–T and Q&P, the $\sigma_{0.2}$ and σ_b of Nb03 Q–P–T sample are 1146 and 1247 MPa, respectively, which are also higher than 1094 and 1236 MPa of Nb00 Q&P sample, respectively. Besides, the elongation of Nb03 sample is slightly higher than that of Nb00 sample, e.g., the δ_u and δ_b of Nb03 Q&T sample are 4.0 and 12.2%, respectively,

Fig. 1 Schematic heat treatment diagram of the Q–P–T, Q&P and Q&T processes, where the abbreviation of M.S.Q. and W.Q. represent molten salt quenching and water quenching, respectively

Fig. 2 Tensile engineering stress and strain curves of the Nb00 steel and Nb03 steel treated by Q–P–T, Q&P and Q&T processes

Table 2 Mechanical properties of the steels treated by Q&T, Q&P and Q–P–T processes

Steel	Process	$\sigma_{0.2}$ (MPa)	σ_b (MPa)	δ_u (%)	δ_b (%)	$\sigma_b \times \delta_b$ (GPa%)
Nb00	Q&T	1149 ± 7	1266 ± 9	3.8 ± 0.3	11.7 ± 0.8	14.8
Nb03	Q&T	1227 ± 13	1337 ± 14	4.0 ± 0.1	12.2 ± 0.5	16.3
Nb00	Q&P	1094 ± 17	1236 ± 9	5.4 ± 0.8	13.9 ± 0.8	17.2
Nb03	Q–P–T	1146 ± 15	1247 ± 15	6.0 ± 0.3	15.0 ± 0.5	18.7

slightly higher than 3.8 and 11.7% of Nb00 Q&T sample, while for the Nb03 Q–P–T sample, the values of δ_u and δ_b are 6.0 and 15.0%, respectively, slightly higher than 5.4 and 13.9% for Nb00 Q&P sample. Therefore, Q–P–T steel exhibits the highest PSE (18.7 GPa%), the second Q&P steel (17.2 GPa%), the third Nb-microalloyed Q&T steel (16.3 GPa%) and finally the Nb-free Q&T steel (14.8 GPa%). The PSE (energy unit), as a comprehensive property, is traditionally considered to represent the properties of toughness [28].

Figure 3 shows the XRD spectra of Nb00 and Nb03 samples treated by Q&T, Q&P and Q–P–T processes, respectively. It is clear that only bcc-phase appears in the Q&T samples, indicating no fcc-phase peak, while the diffraction peaks of fcc-phase are clearly visible in both Q&P (Nb00) and Q–P–T (Nb03) samples in addition to strong diffraction peaks of bcc-phase. And the fractions of fcc-phase are determined to be 5.5 vol% for Q&P (Nb00) sample and 6.8 vol% for Q–P–T (Nb03) sample, respectively. The TEM observation for the Nb00 Q&T and Q&P specimens (Fig. 4) further reveals that the bcc-phase is dislocation-type martensite and the fcc-phase is retained austenite. Moreover, the film-like retained austenite distributes between the martensite laths, which are verified by bright-field (BF) images (Fig. 4a, c) and the dark-field (DF)

images (Fig. 4b, d). The inserted selected area electron diffraction (SAED) patterns indicate that the orientation relationships between martensite and retained austenite are $(\bar{1}\bar{1}0)_\alpha // (\bar{1}\bar{1}1)_\gamma$ and $[001]_\alpha // [011]_\gamma$ N–W orientation relationship (Fig. 4b). There is a coexistence of N–W and K–S orientation relationship, i.e., $(\bar{1}\bar{1}0)_\alpha // (\bar{1}\bar{1}1)_\gamma$ and $[\bar{1}11]_\alpha // [011]_\gamma$ (Fig. 4d). It is worth noting that TEM images exhibit film-like retained austenite in Q&T specimen. But, retained austenite could not be detected in XRD spectrum (Fig. 3), indicating that the volume fraction of retained austenite in Q&T sample is too less to be determined. Figure 5 shows the TEM micrographs of the Nb03 steel treated by Q&T and Q&P processes. It can be seen from Fig. 5b, d that there are large numbers of fine spherical precipitated carbides with average particle size about 5–10 nm dispersedly distributed in the martensite matrix. These spherical carbides are identified as NbC with fcc lattice structure by SAED inserted in Fig. 5b. Moreover, the NbC carbides and martensite matrix take Bake–Nutting (B–N) orientation relationship as $(01\bar{1})_\alpha // (\bar{2}00)_{\text{NbC}}$ and $[011]_\alpha // [0\bar{1}\bar{1}]_{\text{NbC}}$.

4 Discussion

4.1 Effects of Nb on Strength and Ductility

By comparing Nb-microalloyed steels with Nb-free steels, we can find that the Nb-microalloyed steels have higher strength, and the increment of yield strength is more obvious in Nb-microalloyed steels compared to the samples without Nb. For instance, the $\sigma_{0.2}$ of Nb03 Q&T steel was increased by 78 MPa as compared to that of Nb00 Q&T steel, while σ_b was increased by 71 MPa. In particular, the $\sigma_{0.2}$ of Nb03 Q–P–T steel was increased by 52 MPa when compared with that of Nb00 Q&P steel, while the σ_b was increased only by 11 MPa. It is well known that the increase in yield strength depends on grain refinement according to Hall–Petch relation [29]. The increase in tensile strength depends on the size, amount and distribution of spherical carbides in matrix based on Zener equation [30] and grain size as well. Therefore, it is reasonable to believe that the grain refinement strengthening of Nb is stronger than its precipitation strengthening. In the previous investigation [31], precipitation temperature of Nb is about 1200 °C and thus the

Fig. 3 XRD patterns of the Nb00 steel and Nb03 steel treated by Q–P–T, Q&P and Q&T processes

Fig. 4 TEM micrographs of the Nb00 steel treated by Q&T and Q&P processes, respectively: **a** Q&T, bright-field image; **b** Q&T, dark-field image and $\bar{1}\bar{1}1_\gamma$ and $1\bar{1}1_\gamma$ diffraction spots of retained austenite; **c** Q&P, bright-field images; **d** Q&P, dark-field image and $\bar{1}\bar{1}1_\gamma$ and $1\bar{1}1_\gamma$ diffraction spots of retained austenite

pinning effect of Nb on grain boundaries results in refinement of austenite grain during hot-rolling. During subsequent austenitizing, the pinning effect of Nb on grain boundaries results in further refinement of austenite grain. The refinement of austenite grains gives rise to the decrease in martensite packet and block sizes [32]. This is the reason that Nb can markedly enhance the yield strength of steel. The Nb-carbides precipitate during austenitizing and partitioning/tempering, respectively, in which Nb-carbides have larger size during austenitizing, about several tens nanometer, and Nb-carbides have smaller size, about several nanometer [33]. As a result, the tensile strength increase is mainly attributed to fine Nb-carbides precipitated from martensite matrix during partitioning/tempering. The strengthening effect from the precipitation of carbides will always cause the loss of ductility because they impede the movement of dislocations [34]. However, the refinement of grains will possibly lead to of ductility increase. Because the grain boundaries impede the movement of dislocations, and

smaller grains accompanying with more grain boundaries, which will result in the ductility decrease. In other hand, the refinement of grains increases the ductility based on effect of grain size on Lüders elongation [35]. Therefore, it is reasonably concluded that the effect of Nb on ductility by grain refinement compensates impeding effect of Nb-carbides on dislocation movement in this work, and thus the elongation of Nb-microalloyed steels is slightly higher than that of Nb-free steels. It is worth noting that low- and medium-carbon martensitic steels (<0.5 wt%) may have tempering brittleness when they were tempered between 300 and 500 °C due to precipitation of brittle phase Fe_3C carbides [36]. However, the addition of Si element can suppress the precipitation of the brittle Fe_3C carbides [37]. So, in designing Fe–Mn–Si-based Q–P–T steel, stable NbC carbides precipitate from martensite matrix and no formation of brittle Fe_3C carbides during tempering of 450 °C. Thus, tempering brittleness will be avoided and the ductility of Q–P–T steel increased.

Fig. 5 TEM micrographs of the Nb03 steel treated by Q&T and Q&P processes: **a** Q&T, bright-field image; **b** Q&T, dark-field image and 111_{NbC} diffraction spots of retained austenite; **c** Q&P, bright-field images; **d** Q&P, dark-field image and 111_{NbC} diffraction spots of retained austenite

4.2 Mechanism for Ductility Enhancement in Q–P–T Process

As mentioned above, Q–P–T process contains the essentials of the Q&P process. Quenching temperature (T_q) is determined by both CCE theory and K–M equation, where the T_q is usually much higher than room temperature for low and medium steel. And thus, the Q–P–T martensitic steels have considerable retained austenite comparing with the traditional Q&T steels. The origin of retained austenite on ductility enhancement results from three effects: DARA effect, TRIP effect and BCP effect. The DARA effect makes the hard phase, martensite, maintain in a “softening” state during whole deformation and availably intensifies the deformation ability of the hard phase, martensite, at uniform deformation stage, and in turn evidently improves the ductility. Since not every martensite grain (lath) would have an adjacent retained austenite, only the dislocations in martensite lath neighboring retained austenite can move into the retained austenite. Meanwhile, Lu *et al.* [38] experimentally reported that the dislocation

cannot form in the retained austenite with the thickness less than 15 nm. As a result, the more the retained austenite is, the stronger the DARA effect is. With further increase in strain, the strain-induced martensitic transformation from retained austenite will occur when the stress in local area of retained austenite reaches certain critical value, and this leads to the well-known TRIP effect because strain-induced martensitic transformation relaxes the stress concentration in this area and avoids the formation of cracks effectively, and thus delays the necking of the Q–P–T steel. Obviously, the more the retained austenite is, the stronger the TRIP effect is. Under subsequent larger strain condition, once cracks form and propagate in some high stress concentration areas, the retained austenite can block further the propagation of cracks, i.e., BCP effect occurs. The BCP effect effectively postpones the fracture of sample and increases the total elongation of martensitic steels. In addition to ductility enhancement stemming from considerable retained austenite by Q–P–T process, we find that higher T_q can effectively decrease thermal stress and phase-transformation stress. It can be qualitatively

Fig. 6 SEM fractographs of the Nb03 steel treated by Q&T **a**, Q-P-T **b** processes

analyzed by the relationship between stress σ and temperature as follows [39]:

$$\sigma = \alpha_1 E (T_0 - T_f). \quad (1)$$

where E and α_1 are the elastic modulus and the coefficient of thermal expansion, respectively, and they are constant for a given material; T_0 and T_f are initial and final temperature, respectively. In the present case, $T_0 = 940$ °C and $T_f = 20$ °C (room temperature) for Q&T process and 300 °C is for Q-P-T (or Q&P) process, respectively. Obviously, the thermal stress caused by Q&T process is much higher than that by Q-P-T (or Q&P) one at quenching stage. In addition, we can know from K-M equation [12] that the T_q in Q&T process is much lower than that in Q-P-T process, and more transformation from austenite to martensite occurs in Q&T process, which will probably cause the initiation of microcracks. Figure 6 shows the SEM images of fracture morphologies of Nb03 Q&T and Q-P-T samples, respectively. They have the same composition, but the T_q in Q&T process is much lower than that in Q-P-T process. It can be seen that the number of the secondary microcracks in Q&T fracture sample (Fig. 6a) is more and the size is larger than that in Q-P-T fracture sample (Fig. 6b), which can be attributed to higher thermal stress and transformation stress caused by Q&T process. By comparison of the fracture morphologies of Q-P-T sample with Q&T sample (see Fig. 6), the tearing ridges in Q-P-T sample are more than those in Q&T sample, which probably results from much softer phase, the retained austenite. The dimples in Q-P-T sample are deeper and more uniform than those in Q&T sample, which probably results from lower dislocation density in martensite matrix in Q-P-T sample. Partitioning of carbon from martensite to retained austenite results in relative low dislocation density in martensite, and the dislocation density in martensite will further decrease due to DARA effect during deformation.

5 Conclusions

1. The addition of Nb to Fe-Mn-Si-based steels markedly raises the strength, especially the yield strength. The yield strength of Nb-microalloyed Q&T steel was increased by 78 MPa when compared with that of Nb-free Q&T steel, while that of Nb-microalloyed Q-P-T steel was increased by 52 MPa when compared with that of Nb-free Q&P steel under the same heat treatment condition. The elongation of Nb-microalloyed steels is slightly higher than that of Nb-free steels.
2. The mechanism for ductility enhancement in Q-P-T process is resulted from both considerable amount of retained austenite and high quenching temperature T_q leading to lower thermal inner stress.
3. In the designed the Fe-Mn-Si-based Q-P-T steel, stable NbC carbides precipitate in the martensite matrix and no brittle Fe_3C carbides form during 450 °C tempering. Tempering brittleness is avoided by addition of Si, and the ductility of Q-P-T steel is thus increased.

Acknowledgments The work was financially supported by the National Natural Science Foundation of China (No. 51301106).

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