The twice softening of martensitic matrix in Q–P–T steels and its effect on ductility

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ABSTRACT

When a Si-containing steel is subjected to quenching-partitioning-tempering (Q–P–T) process, it will become a martensitic steel with considerable volume of retained austenite, which is attributed to the partitioning of carbon from martensite to retained austenite leading to carbon-depletion in martensitic matrix and carbon-enrichment in the retained austenite. The softening of the martensitic matrix caused by carbon-depletion markedly enhances its deformation ability and raises the ductility of Q–P–T steels. During deformation dislocation absorption by retained austenite (DARA) effect will occur, that is, the dislocations in martensitic matrix will move into nearby retained austenite through the martensite-austenite interface and be absorbed by the retained austenite. This second softening of the martensitic matrix also enhances its deformation ability and effectively raises the ductility. The effect of DARA on ductility during deformation is more effective than the transformation-induced plasticity (TRIP) effect caused by martensitic transformation of retained austenite, because during the TRIP effect the twin-type martensite produced by strain-induced martensitic transformation of retained austenite is very detrimental to ductility.

KEYWORDS

Q–P–T process; ductility; softening of martensite; retained austenite; DARA effect

Introduction

In advanced high strength steels (AHSSs), martensitic steels have the highest strength. In 2003, Speer et al. [1] proposed a novel heat treatment process named as quenching–partitioning (Q&P) process. The Q&P process involves a Si-containing steel being quenched from the austenitizing temperature to a temperature (T_q) between the martensite-start (M_s) and martensite-finish (M_f) temperature, followed by a ‘partitioning’ treatment either at (one-step Q&P) or above T_q temperature (two-step Q&P). During ‘partitioning’ carbon diffuses from the supersaturated martensite phase to the untransformed austenite phase; therefore, carbon-enriched retained austenite can be stabilized in subsequent cooling to room temperature. For low-carbon or medium-carbon Q&P steels, the T_q is usually much higher than room temperature, and thus considerable retained austenite can be obtained. Based on the CCE theory of the Q&P process [1] the precipitation of carbides is not permitted during Q&P process, and therefore it excludes precipitation strengthening. For this reason, Hsu [2] proposed the quenching–partitioning–tempering (Q–P–T) process in 2007. Additional carbide-forming elements like Nb are added in Q–P–T steels, which can lead to carbide formation and grain refinement for precipitation strengthening and refined grain strengthening [3].

Since Q–P–T process absorbs the core idea of the Q&P process: quenching temperature (T_q) determined by the combination of CCE theory and K–M equation [4], obviously, the Q–P–T martensitic steels also have higher retained austenite comparing with traditional quenching and tempering (Q&T) process (its T_q is room temperature). Our previous researches [5–8] indicate that low-carbon or medium-carbon Q–P–T steel exhibits much better product of strength and elongation (PSE) than traditional low-carbon or medium-carbon Q&T steels due to more retained austenite. The ductility enhanced by retained austenite stems not only from the well-known transformation-induced plasticity (TRIP) effect [9] proposed 50 years ago but also a new effect: dislocation absorption by retained austenite (DARA) effect proposed by us in 2011 [8]. TRIP effect enhances the ductility by strain-induced martensitic transformation (SIMT) of retained austenite, which relieves the stress within untransformed retained austenite and its adjacent martensite during deformation [10], and thus the TRIP effect delays the necking. We proposed the DARA effect in medium-carbon Q–P–T steel based on the measurement of average dislocation densities in both martensite and retained austenite during deformation by...
X-ray diffraction line profile analysis (XLPA). During deformation the dislocations in the martensitic matrix will move into nearby retained austenite through the martensite-austenite interface and be absorbed by retained austenite. Two empirical conditions required for the remarkable DARA effect were further proposed: [6,7] (1) retained austenite, being larger than 10% volume fraction; and (2) that the interface between two phases should be coherent or semi-coherent. These two conditions were confirmed by low-carbon Q–P–T steel [7] and low-carbon bainitic steel [6], respectively, in which the interface between martensite (or bainite) and retained austenite was coherent or semi-coherent at K–S or N–W orientation relationship. The DARA effect makes hard phase martensite a ‘softening’ state during deformation and effectively enhances the deformation ability of martensite matrix. Of the two effects mentioned above, the TRIP effect is most paid attention by investigators, and is usually considered as a main origin of ductility for martensitic steels or bainitic ones. However, we note that usually considered as a main origin of ductility for martensitic steels or bainitic ones. However, we note that.

Specimens for transmission electron microscopy (TEM) were prepared by mechanically polishing and then electropolishing in a twin-jet polisher using 4% perchloric acid and 96% ethanol at −20°C. TEM was performed in a JEOL-2100F microscope operated at 200 kV. The volume fractions of retained austenite (VRa) in the Q–P–T samples unloaded at different strain stages were measured by X-ray diffraction (XRD) with CuKα radiation using a D/max-2550 X-ray diffractometer based on a direct comparison method of the integrated intensity of the austenite peaks and the martensite peaks [8]. The average dislocation densities in both martensite and retained austenite were measured by X-ray diffraction line profile analysis (XLPA) [14,15], rather than TEM because the TEM method produces a localized value without statistical information.

Results

Heat treatment process

When low carbon, medium carbon and high carbon steels are subjected to a Q–P–T process, these Q–P–T steels will all exhibit a carbon-depleted martensitic matrix and a carbon-enriched retained austenite, moreover, the amount of retained austenite is much higher than that of martensitic steels treated by traditional Q&T process. For examples, Fe–0.19C–1.52Mn–1.57Si–0.03Nb (wt%) steel (Fe-0.2C-0.03Nb) [16] was treated by Q–P–T process, that is, the low carbon steel was austenitized at 960°C for 300 s, followed by quenching into a salt bath at 300°C (Tq) for 15 s, then by partitioning and tempering at 450°C for 30 s and finally water quenched to room temperature. This low carbon Q–P–T steel has a VRa of 13.8%. While when this low carbon steel was subjected to the Q&T process it had hardly any retained austenite. Fe–0.42C–1.46Mn–1.58Si–0.028Nb (wt%) steel (Fe-0.4C-0.03Nb) [16] was subjected to the Q–P–T process which involved austenitising at 850°C for 300 s, followed by quenching into salt bath at 200°C (Tq) for 15 s, and then by partitioning and tempering at 450°C for 30 s in molten salt, then cooling to room temperature in water. The medium carbon the Q–P–T martensitic steel has VRa of 16%. While when this low carbon steel was subjected to the Q&T process it had hardly any retained austenite. Fe–0.63C–1.52Mn–1.49Si–0.62Cr–0.036Nb (wt%) steel (Fe-0.6C-0.03Nb) [11] was subjected to the Q–P–T process, which involved austenitising at 830°C for 300 s, followed by quenching in a salt bath at 160°C (Tq) for 10 s, then partitioning and tempering at 400°C for 60 s in molten salt, and finally quenching to room temperature in water. This high carbon Q–P–T steel has a VRa of 29.1%. While when this high carbon steel was subjected to the Q&T process it had a VRa of only 7.1%. When this high carbon steel was quenched in liquid nitrogen (LN) it had hardly any
retained austenite. In order to refine the microstructure consisting of coarse ferrite and pearlite in a hot-rolled plate, the pretreatment of the normalization process as Q–P–T process was designed for Fe–0.6C–0.03Nb [17]. That is, samples were heated at 850°C for 1 h and then cooled in air to room temperature. Then, these specimens were subjected to a Q–P–T process, and marked as sample-B, while samples without pretreatment were marked as sample-A.

**Mechanical properties**

The mechanical properties of Fe–0.2C–0.03Nb, Fe–0.4C–0.03Nb and Fe–0.6C–0.03Nb steels are shown in Figure 1 [11,16]. The tensile strength of Q&T steels is all higher than that of Q–P–T steels, but the elongation is much lower than that of Q–P–T steels. It is worth to point out that the tensile strength (1558 MPa) and total elongation (20.3%) of medium carbon Q–P–T steel are higher than the 1242 MPa and 15.0% of low carbon Q–P–T steel because of increased carbon content. However, high carbon Q–P–T steel exhibits tensile strength of 1950 MPa and total elongation of 12.4%, as shown in Figure 1(b) [11]. Elongation is lower than that of medium carbon steel. When the pretreatment of the normalization process as Q–P–T process was added, this high carbon Q–P–T steel (sample-B) exhibits tensile strength of 1860 MPa and total elongation of 29.0%, as shown in Figure 2 [17]. So far, we have realized the enhancement of both strength and elongation with the increase of carbon content from low to high carbon through medium carbon.

**Microstructural characterization**

Based on the XRD spectra of low carbon, medium carbon [16] and high carbon Q–P–T samples [11] before tensile test (0% strain) in Figures 3 and 4, their microstructures were determined as body centered cubic (bcc) phase and face-centered cubic (fcc) phase. While in corresponding low carbon and medium carbon Q&T samples there were no diffraction peaks of retained austenite. In high carbon Q&T (quenching in water) sample there was a $V_{RA}$ of 7.1%, but in sample quenched in liquid nitrogen (LN) there was no diffraction peak of retained austenite (Figure 4). TEM characterization reveals that Q–P–T steel consists of the bcc dislocation-type martensite laths and fcc film-like retained austenite (several nanometer width) for low carbon steel (Figure 5(b)) or fcc flake-like retained austenite (several tens of nanometers width) for medium carbon steel (Figure 5(d)). For high carbon Q–P–T steel, some thin plates of twin-type martensite are embedded in dislocation-type martensitic matrix (Figure 6(b)), and fcc retained austenite exhibited chunky characteristics of several hundred nanometers width (Figure 6(d)). Selected area electron diffraction (SAED) patterns inserted in Figure 5(b,d) and Figure 6(d), show the orientation relationship between martensite and retained austenite: 

$$\{110\}_a/\{1\overline{1}1\}_y, \quad <111>_a/\langle 011 \rangle_y, \quad (K-S) \quad \text{and} \quad \{110\}_a/\{1\overline{1}1\}_y, <\overline{1}01>_a/\langle 011 \rangle_y, \quad (N-W).$$

**Discussion**

**Softening of martensitic matrix caused by the partition of carbon**

As mentioned above, during the Q–P–T process, the partitioning of carbon from supersaturated martensite to the
untransformed austenite will lead to a carbon-depleted martensitic matrix and carbon-enriched retained austenite. Here, we take Fe–0.6C–0.03Nb Q–P–T steel as an example to demonstrate the softening of martensitic matrix caused by the partition of carbon. According to the $C_g = (\alpha - 3.547)/0.046$ equation [18,19], the carbon content in retained austenite can be determined by the lattice constant of austenite ($a_g = 3.61094 \times 10^{-10}$ m) measured from the (200)$_g$ diffraction peak. For Q–P–T steel with partitioning/tempering at 400°C for 600 s, the carbon content of the retained austenite in Q–P–T steel was calculated as 1.39 wt%, and thus the carbon content in the martensite was estimated as 0.27 wt%, which is based on both the carbon content (0.63 wt%) of this steel and the ratio of bcc and fcc phases in volume fraction if the reduction of carbon due to the precipitation of carbide from the martensitic matrix is ignored. It is clear that the carbon content in martensite decreases from 0.63–0.27 wt%, while the carbon content in the retained austenite increases from 0.63–1.39 wt%. With the reduction of carbon partitioning time, the amount of retained austenite remains almost unchanged, but the degree of carbon depletion will be lowered. For example, when the partitioning time decreases from 600 to 60 s, the carbon content in the retained austenite falls from 1.39–1.37 wt% accompanied by an increase of carbon content in martensite from 0.27–0.31 wt%. With the decrease of partitioning time, the tensile strength increases accompanied by a reduction of elongation. The mechanical properties of Q–P–T samples with different partitioning/tempering times at 400°C are shown in Figure 7 [13]. The tensile strength and elongation are 1860 MPa and 28.9% for partitioning time with 600 s, 1910 MPa and 21.7% for 300 s, and 1920 MPa and 18.8% for 60 s, respectively. It is clear that with prolonging partitioning time, the degree of softening of the martensite matrix rises accompanied by an increase in elongation. For Q&T steel, since no retained austenite exists and no partitioning of carbon occurs, the carbon content in martensite remains 0.63 wt%. The carbon content of 0.63 wt% in martensite without softening corresponds to the average dislocation density of $7.69 \times 10^{14} \text{ m}^{-2}$, and the carbon content of 0.27 wt% in softening martensite corresponds to $5.45 \times 10^{14} \text{ m}^{-2}$. The carbon depletion in martensite

![Figure 3. XRD spectra of Fe–0.2C–0.03Nb and Fe–0.4C–0.03Nb samples after Q&T or Q–P–T treatment, respectively.](image)

![Figure 4. XRD spectra of the Fe–0.6C–0.03Nb Q–P–T and Q&T tensile samples at different strain stages (from 0% to 12.4% strains).](image)
by partitioning of carbon leads to a decrease by dislocation density of $2.24 \times 10^{14} \text{ m}^{-2}$, which is over the increment ($1.90 \times 10^{14} \text{ m}^{-2}$) of the average dislocation density in the pure aluminum at 8% true strain [20]. It can be imagined that the evident decrease of dislocation density in the martensitic matrix will effectively relieve

**Figure 5.** Bright field (a, c) and 111̅, diffraction spot formed dark field (b, d) TEM images of Fe–0.2C–0.03Nb (a, b) and Fe–0.4C–0.03Nb (c, d) samples after Q–P–T treatment, inserted SAED patterns.

**Figure 6.** TEM micrographs of undeformed high carbon Q–P–T sample: (a) twinning martensite, BF image, (b) twinning martensite, DF image and inserted SAED pattern, (c) BF image of Q–P–T sample, (d) DF image of retained austenite and inserted SAED pattern of the retained austenite and martensite.
the stress concentration and delay crack initiation caused by tangle dislocations during deformation, and thus such a softening of martensite resulting from partitioning of carbon will markedly raise the deformation ability of the martensitic matrix in Q–P–T steel, and in turn markedly enhance the ductility.

**Softening of martensitic matrix caused by DARA effect**

We at first measured the variations of average dislocation densities within both martensitic matrix and retained austenite in Fe–0.4C–0.03Nb samples unloaded at different deformation stages by XPLA and found that during deformation the dislocations in martensitic matrix will move into nearby retained austenite through the martensite-austenite interface and be absorbed by retained austenite and named it the DARA effect. The DARA effect was also found in low-carbon Q–P–T steel [7] and bainitic steel [6], respectively. Since in a high-carbon Q–P–T steel twin-type martensite will partially replace dislocation-type martensite, whether DARA effect is suppressed is in question. For the sake, we recently measured the average dislocation densities in martensite and retained austenite in Fe–0.6C–0.03Nb Q–P–T samples and Q&T samples for comparison [13]. For the high-carbon Q&T sample quenched in liquid nitrogen (LN), the average dislocation $\bar{\rho}_M$ in martensite increases with strain monotonically (Figure 8(a)), which is in accordance with our expectation because of dislocation multiplication. However, for the Q–P–T sample, $\bar{\rho}_M$ in martensite did not change in the same way. The $\bar{\rho}_M$ in martensite reduces with increasing strain in initial deformation. For example, before the tensile test, $\bar{\rho}_M$ was $5.45 \times 10^{14}$ m$^{-2}$, while $\bar{\rho}_M$ was $5.28 \times 10^{14}$ m$^{-2}$ at 3% strain, which was less than that before the tensile test; then after 5% strain, the $\bar{\rho}_M$ in martensite gradually rises with increasing strain, as plotted in Figure 8(a). The $\bar{\rho}_A$ in the retained austenite rapidly increased with the strain, as shown in Figure 8(b). Obviously, such a phenomenon cannot be explained by the TRIP effect. Our explanation for this phenomenon is described as follows.

During initial deformation, the $\bar{\rho}_M$ in martensite reduces with increasing strain because dislocations in martensite

**Figure 7.** Comparison of mechanical properties of high-carbon Q–P–T martensitic steel with other AHSSs with different tensile sample sizes reported.

**Figure 8.** The variation of average dislocation density with strain in martensite (a) or retained austenite (b) in Q–P–T sample or Q&T one.
move to nearby retained austenite through the interface between martensite and retained austenite. Moreover, the number of dislocations in martensite transported to the retained austenite is larger than the scale of dislocation multiplication which leads to the decrease of dislocation density in martensite in the initial deformation stage. After 5% strain, the $\bar{\rho}_m$ in martensite gradually rises with increasing strain, indicating that the number of dislocations in martensite transported to retained austenite is less than the scale of dislocation multiplication in martensite. Furthermore, the number of dislocations in martensite transported to retained austenite could be estimated as follows: The increment of the average dislocation density in martensite is about 8% true strain [20].

As mentioned above, the decrease of the dislocation multiplication when compared with that in the undeformed Q&T sample is about 1.08 x 10^{14} m^{-2}. The average dislocation density of 3.92 x 10^{14} m^{-2} is about twice (1.90 x 10^{14} m^{-2}) of the average dislocation density in the pure aluminum at 8% true strain [20]. As mentioned above, when the partitioning time decreases from 600 to 60 s, the carbon content in retained austenite lowers from 1.39 to 1.37 wt% accompanied by an increase of carbon content in martensite from 0.27 to 0.31 wt%. Corresponding average dislocation density in martensite decreases by 2.24 x 10^{14} m^{-2} and elongation increases by 10%. Therefore, it is reasonably believed that, the reduction (3.92 x 10^{14} m^{-2}) of average dislocation density caused by the DARA effect will at least rise above 10% of elongation.

**Effect of SIMT on ductility**

As well known, TRIP effect enhances the ductility by strain-induced martensitic transformation (SIMT), which relieves the stress within untransformed retained austenite and its adjacent martensite during deformation [10], and thus the TRIP effect delays the necking. The enhancement effect of SIMT from retained austenite on ductility has been verified in low carbon and medium carbon martensitic steels [5-8]. However, we also know that the twin-type martensite plates produced by SIMT are very brittle and are very detrimental to ductility. Therefore, the SIMT has two opposite effects of positive and negative on ductility. In our previous study, the variation of $V_{RA}$ with strain in medium-carbon Q-P-T steel with 20% total elongation exhibits a remarkable TRIP effect [16], namely, the $V_{RA}$ gradually decreases from 16%, 13%, 10%, and 5% to 3% with the increase of strain from 0 (before deformation), 3%, 7%, and 11% to 15%, respectively. The $V_{RA}$ (29.1%) in above high-carbon Q-P-T steel is much more than 16% in the medium carbon Q-P-T steel before tensile test, and the issue we face is: whether more retained austenite can also produce more remarkable TRIP effect in high-carbon Q-P-T steel than in medium-carbon Q-P-T steel or not? To answer this question, the $V_{RA}$ in tensile samples unloaded at different strains from 0% to 12% was measured by XRD [11]. It is quite clear that the $V_{RA}$ gradually decreases from 29.1%, 25.9%, 20.4%, 17.9%, and 15.1% to 10.4% with increasing strain from 0%, 2%, 4%, 6%, and 8% to 12.4% (fracture strain), respectively, which is attributed to SIMT. The SIMT effect gradually occurs during deformation accompanying with the decrease of the $V_{RA}$. Since retained austenite in Q-P-T steel has more than 0.8 wt% carbon content, the strain-induced martensite is twin-type martensite. Figure 9 shows TEM micrographs of strain-induced twinning martensite in deformed Q-P-T specimen after fracture [13]. Based on the following fact, although the $V_{RA}$ (29.1%) in the high carbon Q-P-T martensitic steel is much higher than 16% in medium carbon Q-P-T martensitic steel, the elongation (12.4%) is much less than 20% of the medium carbon Q-P-T martensitic steel. This means that the detrimental effect of SIMT on ductility is greater than the enhancing effect of SIMT on ductility in this high carbon Q-P-T martensitic steel. In view of the detrimental effect of SIMT on ductility, we proposed

**Figure 9.** TEM micrographs of strain-induced twinning martensite in deformed Q-P-T specimen: (a) bright-field image and (b) dark-field image and inserted SAED pattern.
the design idea of Anti-TRIP [13]. We designed another process in which the normalization (NOR) process was taken as a pretreatment for the Q–P–T process [13]. We named the above high carbon Q–P–T sample without or with pretreatment as sample-A or sample-B. It is worth pointing out that the morphologies of the retained austenite were mainly chunky (see Figure 6(d)), which was different from low carbon Q–P–T steels with its film-like retained austenite (Figure 5(b)) and medium carbon Q–P–T steels with flake-like retained austenite (Figure 5(d)). So far, we cannot find the obvious difference of microstructures of sample-A and sample-B from SEM and TEM observation as well as XRD measurement expect for the martensite’s average size. Therefore, the microstructures in sample-A and sample-B were further characterized by EBSD [13]. Figure 10(a,b) show the color code phase map, in which red color corresponds to bcc lattice (martensite) and blue color corresponds to fcc lattice (austenite). In Figure 10, the flake-like (or thinner film-like) retained austenite between martensite laths cannot be seen because the scanning step length of EBSD is 0.1 mm, which is larger than the nanosized-width of flake-like retained austenite. By comparing Figure 10(a) with (b), it can be found that sample-B has more dispersive and finer retained austenite than sample-A. The grain size distribution of retained austenite was quantitatively measured by Image-Pro Plus, as shown in Figure 10(c). It is clear from Figure 10(c) that the retained austenite in sample-B is more dispersive and of smaller size than that of sample-A with the smallest in the 0.4–0.8 mm range although their volume fraction of retained austenite is almost the same. In order to verify whether the relative dispersive and fine retained austenite has a higher mechanical stability, the variation of retained austenite fraction with strain in different deformation stages was measured by XRD. The result indicates that there is \( V_{RA} \) of 9.1% at 16.9% fracture strain for sample-A, while there is \( V_{RA} \) of 11.9% at the same strain for sample-B. This experiment fully verifies that dispersive and fine retained austenite has higher mechanical stability. Since retained austenite has higher mechanical stability in sample-B than in sample-A, during deformation strain-induced twin-type martensite in sample-B is less than that in sample-A, which leads to higher elongation (29%) of sample-B martensitic steel than the 16.9% of sample-A. In general, twin-type martensite produced by SIMT from chunky retained austenite is very detrimental to ductility, while dispersive and fine retained austenite with high mechanical stability is expected to improve ductility.

**Dominant mechanism of high ductility**

Comparing the mechanical properties of Q–P–T steels with those of Q&T steels with the same composition, we found that the total elongation of a Q–P–T steel was much higher than that of Q&T steel with the same composition. Obviously, the high ductility stems from much more \( V_{RA} \) in Q–P–T steels than in Q&T steels. The above results indicate that the ductility is enhanced by retained austenite through two approaches: the DARA effect and the TRIP effect. The DARA effect means that the hard phase martensite in a ‘softening’ state during whole deformation intensifies the deformation ability of the hard phase martensite, and in turn evidently improves the ductility. Since not every martensite grain (lath) would have an adjacent retained austenite, the dislocations only in martensite lath neighboring retained austenite can move into the retained austenite. As a result, the more the \( V_{RA} \) is, the stronger

![Figure 10. EBSD analysis of retained austenite in sample-A (a) and sample-B (b), respectively; (c) grain size distribution of retained austenite.](image)
the DARA effect is. With further increase of strain, the strain-induced martensitic transformation from retained austenite will occur when the stress caused by high density of dislocation in local area reaches certain critical value, and this leads to TRIP effect because SIMT relaxes the stress concentration in this area and avoids the initiation of cracks, and thus delays the necking of the Q–P–T steel. However, the twin-type martensite produced by SIMT severely damages ductility of high carbon Q–P–T martensitic steels, and thus the SIMT should as possible be avoided to occur in retained austenite. The high ductility of Q–P–T steels is not only connected to more retained austenite with high mechanical stability, but also connected to lower dislocation density in martensitic matrix than Q&T steels with the same composition.

In general, the high ductility of a martensitic steel is mainly not dependent on the amount of soft phase retained austenite, but is dependent on the deformation ability of martensitic matrix and the ability of hindering crack formation in the hard phase martensitic matrix. The deformation ability of martensitic matrix is attributed to the twice softening of martensitic matrix caused by the partitioning of carbon during Q–P–T process and the DARA effect during deformation. Besides, in the enhancement effects of retained austenite on ductility the DARA effect is more effective than the TRIP effect.

**Disclosure statement**

No potential conflict of interest was reported by the authors.

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