Approach and mechanism of toughness enhancement for a high carbon Q-P-T steel

Shengwei Qin\textsuperscript{a}, Yu Liu\textsuperscript{a}, Jiazhi Zhang\textsuperscript{a}, Ying Wang\textsuperscript{b}, Yonghua Rong\textsuperscript{a}, Xunwei Zuo\textsuperscript{a} and Nailu Chen\textsuperscript{a}

\textsuperscript{a}School of Materials Science and Engineering, Shanghai Jiao Tong University, Shanghai, People’s Republic of China; \textsuperscript{b}School of Mechanical Engineering, Shanghai Dianji University, Shanghai, People’s Republic of China

ABSTRACT

Based on the previous study of Cr-containing Fe-0.63C-1.52Mn-1.49Si-0.62Cr-0.036Nb high carbon steel, although normalization process as a pretreatment of quenching–partitioning–tempering (Q-P-T) was performed in this high carbon steel to raise the ductility, its impact toughness only is 7.4 J/cm\textsuperscript{2}. Microstructural characterization reveals that strain-induced twin-type martensite transformed from considerable chunky retained austenite destroys toughness. Therefore, a novel Cr-free Fe-0.67C-1.52Mn-1.49Si-0.038Nb (wt.%), high carbon steel was designed and normalization process as pretreatment of Q-P-T process was still employed so that the volume fraction of retained austenite evidently decreases from 21% to 10%. This high carbon steel not only keeps high tensile strength (1660 MPa) and elongation (29.0%), but also exhibits high impact toughness of 33.2 J/cm\textsuperscript{2}. The high impact toughness is attributed to the elimination of chunky retained austenite accompanying dispersive and fine retained austenite with high mechanical stability, which effectively reduces the formation of brittle strain-induced twin-type martensite during deformation.

INTRODUCTION

Among the advanced high strength steels, quenching and partitioning (Q&P) \cite{1} steels have been researched intensively due to high strength from martensitic matrix and good ductility from considerable retained austenite. Q&P process involves \cite{1}: a fast quenching from austenitizing temperature to a quenching temperature ($T_q$) between start temperature ($M_s$) of martensitic transformation and finish temperature ($M_f$); then carbon partitioning from martensite to retained austenite at (one-step) or above $T_q$ (two-step) to obtain more carbon-enriched retained austenite at room temperature. However, Q&P process excludes the precipitation of carbides and precipitation strengthening. For the sake, Hsu proposed the quenching–partitioning–tempering (Q-P-T) process in 2007 \cite{2}. Since Q-P-T process absorbs the core idea of Q&P process: the determination of $T_q$ based on ‘constrained carbon paraequilibrium’ (CCE) theory of Q&P process \cite{1}, Q-P-T steels can also obtain considerable carbon-enriched retained austenite by the partitioning of carbon, meanwhile, during partitioning /tempering dispersive carbides precipitate from martensitic matrix. Our previous work demonstrated that Q-P-T process was applied to treat Fe-0.19C-1.52Mn-1.57Si-0.03Nb low carbon steel and Fe-0.63C-1.52Mn-1.49Si-0.62Cr-0.036Nb (wt.%), high carbon steel, respectively, and the result indicates that although retained austenite fraction of 29.1% in this high carbon Q-P-T martensitic steel is much more than 6.5% of low carbon Q-P-T martensitic steel, its total elongation of 12.4% is less than 15% of low carbon Q-P-T steel \cite{3}. The origin of this phenomenon was revealed, that is, high carbon Q-P-T steel has considerable chunky retained austenite with low mechanical stability, rather than film-like retained austenite with high mechanical stability in low carbon Q-P-T steel, and thus the strain-induced twin-type martensite transformed from chunky retained austenite severely harms ductility. Therefore, it is an effective approach for ductility enhancement to raise the mechanical stability of retained austenite. Based on the idea, a normalization process (cooling in air) was added as pretreatment of Q-P-T process for this high carbon steel. The result indicates that the total elongation of high carbon Q-P-T steel rises from 12.4% to 29.0% since the addition of normalization process results in more dispersive and fine retained austenite with high mechanical stability in almost same volume fraction of retained austenite \cite{3}. Moreover, the high carbon Q-P-T steel exhibits the tensile strength of 1860 MPa and product of strength and elongation (PSE) of about 54GPa%. \cite{3,4}. The PSE is energy per volume, and thus is traditionally considered to represent the properties of toughness \cite{5}. However, the average toughness value of three impact samples
with notch for this high carbon Q-P-T steel only is 5.9 J (7.4 J/cm²), while three samples without notch were impacted, they were all unbroken [4]. Microstructural characterization reveals that although the addition of normalization process as pretreatment of Q-P-T process refines retained austenite and raises its mechanical stability accompanying the improvement of ductility, but this high carbon Q-P-T steel still has considerable chunky retained austenite. This finding indicates that the poor impact toughness of this high carbon Q-P-T martensitic steel results from strain-induced twin-type martensite to the notch sensitivity. Therefore, the elimination of chunky retained austenite is a required condition for toughness enhancement.

We found a reverse relation between volume fraction of retained austenite (VA) and the impact toughness in the Q-P-T steel: for Fe-0.1C low alloy Q-P-T steel, the VA and impact toughness were 6% and 100 J [6]; for Fe-0.2C low alloy Q-P-T steel, the VA and impact toughness were 11% and 46 J [7]; for Fe-0.4C low alloy Q-P-T steel, the VA and impact toughness were 15% and 20 J [8]; for Fe-0.6C low alloy Q-P-T steel, the VA and impact toughness were 30% and 4 J [4]. It is quite clear that the impact toughness decreases with the increase of the VA in Q-P-T steels. This finding gives us an inspiration: the normalization process as pretreatment of Q-P-T process can effectively refine retained austenite [4], but cannot eliminate considerable chunky retained austenite. As a result, an approach of toughness enhancement was proposed in this work, namely, chunky retained austenite will be reduced by decreasing VA to a proper volume fraction. A detailed way is that the Cr element stabilizing austenite in the high carbon Q-P-T steel will be removed and the normalization process as pretreatment of Q-P-T process will be kept. The objective of this work is to check the correctness of this approach on toughness enhancement of high carbon steels.

**Experimental procedure**

The chemical composition of the designed steel is analyzed as Fe-0.67C-1.52Mn-1.49Si-0.038Nb (wt.%). The addition of Si can suppress the precipitation of brittle cementite (Fe₃C) [9]. The addition of Nb can promote the formation of stable carbides and refine grains effectively [2]. The Ms and Mf temperature of this high carbon steel were respectively determined as 209°C and 75°C by JMatPro simulation software. Rectangular tensile specimens with the gauge length of 15 mm, width of 5 mm and thickness of 1.5 mm were cut from the hot-rolled plate immediately. Charpy V-notch standard impact samples with 55 × 10 × 10 mm were cut along the rolling direction according to the method described in ISO 148-1:2006. V-notch impact samples were performed on a PTM2200-D1 pendulum impact testing machine. In order to reveal the effect of normalization process on the mechanical stability of retained austenite, specimens were subjected to the two heat treatment processes respectively: sample-A was subjected to single Q-P-T process, that is, it was heated 830°C for 300 s. Subsequently, sample was quenched in a salt bath at 180°C (Tq) for 10 s, then partitioned/tempered at 400°C for 600 s in another molten salt bath, and finally water quenched to room temperature. While sample-B was subjected to normalization process as pretreatment of Q-P-T process, that is, it was heated to 850°C for 3600 s, then air cooled to room temperature, finally the same Q-P-T process as sample-A was performed. Tensile specimens were conducted at room temperature using a Zwick/Roell Z100 universal testing machine at a strain rate of 2 × 10⁻³ s⁻¹. The VA was determined by X-ray diffraction (XRD) with CuKa radiation using a D/max-2550 X-ray diffractometer based on a direct comparison method of the integrated intensity of the austenite (200)γ, (220), and (311)γ peaks and the martensite (200)α and (211)α peaks. Specimens for transmission electron microscopy (TEM) were prepared by mechanically polishing and then electropolished using a solution of 4% perchloric acid and alcohol at −20°C ∼ −30°C in a twin-jet polisher. TEM observation was performed on a JEOL-2100F microscope operated at an accelerating voltage of 200 kV. The 7600F scanning electron microscope equipped with a field emission gun and a TSL system was used to examine microstructure and obtain electron backscatter diffraction (EBSD) map of the samples.

**Results**

**Mechanical properties**

The tensile curves of sample-A and sample-B were presented in the **Figure 1**. The tensile strength, total elongation and PSE of sample-A and sample-B were 1600 MPa, 27.0%,43.2GPa% and 1660 MPa, 30.9%, 51.3GPa%, respectively. It is clear that the mechanical properties of sample-B are much better than those of
sample-A. Considering the effect of tensile specimen size on elongation, the elongation of sample-B by the conversion of Oliver formula used in the international standard [10] is 23.0%, and PSE is 38.2 GPa%, which is superior to the 30 GPa% of the third generation advanced high strength steels. Besides, the average impact toughness value of three impact samples with notch only is 8.1 J/cm² for sample-A, while the average impact toughness value is 33.2 J/cm² for sample-B. The impact toughness of sample-B is about quadruple that of sample-A.

**Microstructural characterization**

The TEM characterization of sample-A and sample-B are presented in Figure 2. The microstructures of both

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**Figure 2.** TEM micrographs of undeformed Q-P-T sample, (a) BF image of sample-A, (b) DF image of chunky retained austenite and inserted SAED pattern of the retained austenite and martensite (incident beam direction $z \approx \{011\}$ or $\{111\}$); (c) BF image of sample-B, (d) DF image of flaky retained austenite and inserted SAED pattern of the retained austenite and martensite (incident beam direction $z \approx \{011\}$ or $\{111\}$); (e) twin-type martensite, BF image of sample-A, (f) twinning martensite, DF image of sample-A and inserted SAED pattern (incident beam direction $z \approx \{113\}$ or $\{113\}$); (g) twin-type martensite, BF image of sample-B, (h) twin-type martensite, DF image of sample-B and inserted SAED pattern (incident beam direction $z \approx \{113\}$ or $\{113\}$).
sample-A and sample-B consist of retained austenite and dislocation-type martensitic matrix. Figure 2(a,b) is bright field (BF) image and dark field (DF) image of chunky retained austenite for sample-A, respectively. Figure 2(c, d) is BF and DF images of flaky retained austenite for sample-B, respectively. Inserted selected area electron diffraction (SAED) pattern in Figure 2(b) and (d) indicates there are \((\overline{1}10)_{\alpha}/(\overline{1}11)_{\gamma}\), \([\overline{1}11]_{\alpha}/[011]_{\gamma}\) (K-S) and\((\overline{1}10)_{\alpha}/(\overline{1}11)_{\gamma}\), \([001]_{\alpha}/[011]_{\gamma}\) (N-W) orientation relationships between martensite and retained austenite. The width of retained austenite in sample-A is high up to about 1 \(\mu\)m (Figure 2(b)), which is about 10 times larger than that in sample-B (Figure 2(d)). While dislocation-type martensite laths in both sample-A and sample-B also exist an evident difference in size, the average size (width and length) of martensite laths of sample-B is much less than that of sample-A. Especially, the majority of retained austenite in sample-B mainly exhibits flaky rather than chunky. Therefore, the strain-induced twinline or flake-like retained austenite between martensite laths cannot be detected because scanning step length of EBSD is 0.1 mm, which is larger than the nanosized-width of film-like or flake-like retained austenite. Figure 3(c) shows the film-like or flake-like retained austenite between martensite laths cannot be detected because scanning step length of EBSD is 0.1 mm, which is larger than the nanosized-width of film-like or flake-like retained austenite. Figure 3(c) shows the grain size distribution of retained austenite measured by Image-Pro Plus. It is clear that the retained austenite in sample-B is not only in volume fraction much less than that of sample-A, but also is in size much smaller than that of sample-A, in which the V\(_{RA}\) of two samples is qualitatively consistent with the XRD results. A great amount of micron-sized chunky retained austenite is observed in sample-A, in contrast, the retained austenite in sample-B is dispersive and fine distribution, and chunky retained austenite is almost eliminated. In order to verify the higher mechanical stability of dispersive retained austenite than chunky retained austenite, the V\(_{RA}\) in the same strain for sample-A and sample-B was measured by XRD. The result indicates that there is the V\(_{RA}\) of 7.1% at 20.0% fracture strain
for sample-A, while there is the $V_{RA}$ of 4.9% at the same strain (unfractured) for sample-B. It indicates that the $V_{RA}$ of 14.5% in sample-A transforms to twin-type martensite, but only the $V_{RA}$ of 5.7% transforms to twin-type martensite in sample-B, implying that the retained austenite in sample-B exhibits higher mechanical stability than sample-A.

**Discussions**

**Effect of normalization process**

In order to reveal the effect of normalization process as a pretreatment of Q-P-T process, the microstructures of the high carbon steel in hot rolled state and in subsequent normalization process state were characterized, respectively. Figure 4(a) demonstrates that the microstructure in hot rolled sample almost consists of single coarse pearlite with wide lamellar spacing and large colony. Figure 4(b) shows that the microstructure after normalization process of the hot rolled sample consists of ferrite and martensite whose average sizes are smaller than the size of pearlite colony. The Cr-free Fe-0.67C-1.52Mn-1.49Si-0.084Nb (wt.%) has the $V_{RA}$ of 21.6% in undergoing single Q-P-T process (sample-A), and Cr-containing Fe-0.63C-1.52Mn-1.49Si-0.62Cr-0.036Nb has the $V_{RA}$ of 29.1%, meaning that the removal of Cr element makes the $V_{RA}$ reduce by about 8%. The addition of normalization process as pretreatment of Q-P-T process makes sample-B further reduce from $V_{RA}$ of 21.6% to 10.6%. It is known from K-M equation [11] that the relative amount of martensite and retained austenite depends on the difference between $T_q$ and $M_s$. Sample-B has the same quenching temperature ($T_q$: 180°C) as sample-A, and thus lower $V_{RA}$ in sample-B implies that sample-B has higher $M_s$ than sample-A. Since long time annealing of normalization process (heated to 850°C for 3600 s) makes the uniform distribution of carbon in high-temperature austenite due to fully dissolution of Fe$_3$C in pearlite in hot rolled microstructure so that ferrite and martensite (less than 0.67 wt% C) are constituent in sample-B before Q-P-T process. While the microstructure in sample-A before Q-P-T process is pearlite consisting of ferrite and Fe$_3$C (6.69 wt% C), a short time of 300s at 830°C in Q-P-T process cannot make a full diffusion of carbon in high-temperature austenite after dissolving Fe$_3$C, as a result, more carbon-enriched area in sample-A than in sample-B leads to more retained austenite in sample-A than in sample-B after Q-P-T process.

In addition, plenty of relative small ferrite and martensitic packets and blocks formed by normalization process for sample-B replace coarse pearlite colonies in hot rolled sample, which effectively increases the nucleation sites of austenite during austenitising temperature in Q-P-T process. Therefore, the sample-B with normalization process exhibits smaller retained austenite and martensitic matrix after Q-P-T process than the sample-A. In general, the removal of Cr element and normalization process as pretreatment of Q-P-T process effectively reduces the $V_{RA}$ which eliminates chunky retained austenite and refines retained austenite and martensitic matrix.

**Origin of toughness enhancement**

As mentioned above, our previous work demonstrated that when normalization process as pretreatment of Q-P-T process was used for Cr-containing high carbon steel, the total elongation of this high carbon Q-P-T steel rises from 12.4% to 29.0%, since the addition of normalization process results in more dispersive and fine retained austenite with high mechanical stability in almost same volume fraction. Moreover, the high carbon Q-P-T steel exhibits a high PSE. The PSE is energy per volume, and thus is traditionally considered to represent the properties of toughness [5]. However, the average toughness value of three impact samples with notch for this high carbon Q-P-T steel only is 7.4 J/cm$^2$, while three samples without notch were impacted, they were all unbroken. Microstructural characterization reveals that although the addition of normalization process as pretreatment of Q-P-T process refines retained austenite and raises its mechanical stability accompanying the improvement of ductility, but this high carbon Q-P-T steel still has considerable chunky retained austenite. It can reasonably be concluded by comparing with the experimental

![Figure 4](image-url)
results of notch-sample’s impact with notch-free sample’s impact that the strain-induced martensite transformed by chunky retained austenite is more detrimental to toughness than ductility due to the sensitivity of strain-induced twin-type martensite to the notch. How to further decrease the amount of chunky retained austenite, our measure is to reduce the volume fraction of retained austenite by removal of Cr element stabilizing austenite and keep normalization process as pretreatment of Q-P-T process. As a result, a Cr-free Fe-0.67C-1.52Mn-1.49Si-0.038Nb (wt.%) was designed and normalization process as pretreatment of Q-P-T process was still employed. XRD verifies the decrease of the volume fraction of retained austenite from 29.1% to 10.6% comparing with Cr-containing high carbon steel, and EBSD demonstrates that chunky retained austenite is almost eliminated accompanying with the formation of dispersive and fine retained austenite. The notch effect toughness lowers from 7.4 to 33.2 J/cm². For Fe-0.67C-1.52Mn-1.49Si-0.038Nb (wt.%), when single Q-P-T process was used (sample-A), the \( V_{RA} \) is still high up to 21.6% accompanying with considerable chunky retained austenite, and thus the impact toughness only is 8.1 J/cm².

The result confirms the correctness of our idea: the elimination of chunky retained austenite with poor mechanical stability is the origin of toughness enhancement. It is worthy to point out that although the evident decrease of retained austenite for a Fe-0.67C-1.52Mn-1.49Si-0.038Nb (wt.%) subjected by normalization process as pretreatment of Q-P-T process, high elongation is kept and high up to 29.0%, which is attributed to the enhancement effect of 10% retained austenite on ductility by dislocation absorption by retained austenite (DARA) effect [12] during deformation and the decrease of brittle twin-type martensite during deformation due to flaky retained austenite with high mechanical stability.

Conclusions
A Cr-free Fe-0.67C-1.52Mn-1.49Si-0.038Nb (wt.%) high carbon steel was designed based on the previous study of Cr-containing high carbon steel and treated by normalization process as pretreatment of Q-P-T process (sample-B) and single Q-P-T process (sample-A) for comparison, respectively. Their mechanical properties were measured and microstructures were characterized by XRD, SEM, EBSD and TEM, and main conclusions are described as follows.

1. The impact toughness of sample-B is 33.2 J/cm², while that of sample-A is only 8.1 J/cm² and that of Cr-containing sample is 7.4 J/cm².
2. The \( V_{RA} \) of sample-B is 10.6%, and such an effective reduction of retained austenite fraction is attributed to the removal of Cr comparing with Cr-containing high carbon steel and the addition of normalization process as pretreatment of Q-P-T process comparing with sample-A treated by single Q-P-T process.
3. The elongation of sample-B is high up to 29.0%, which is attributed to (DARA) effect of retained austenite and high mechanical stability of retained austenite during deformation, in which the latter markedly reduces brittle twin-type martensite transformed from retained austenite.

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