

Work Hardening Behavior and Stability of Retained Austenite for Quenched and Partitioned Steels

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Abstract: Both microstructure and mechanical properties of low alloy steels treated by quenching and partitioning (Q&P) process were examined. The mixed microstructure of martensite and large-fractioned retained austenite (about 27.3%) was characterized and analyzed, excellent combinations of total elongation of 19% and tensile strength of 1835 MPa were obtained, and three-stage work hardening behavior was demonstrated during tensile test. The enhanced mechanical properties and work hardening behavior were explained based on the transformation-induced plasticity effect of large-fractioned austenite.

Key words: Q&P process; work hardening; retained austenite; martensite; mechanical property; TRIP effect

Based on the requirements for vehicle's mass reduction and safety improvement, high-strength steels combined with adequate ductility and formability, such as dual-phase (DP), transformation-induced plasticity (TRIP) and complex steels, have been developed^[1,2]. The third-generation automotive steels with higher value of $R_m \times A$, i. e. the product of tensile strength (R_m) and total elongation (A), and lower cost have attracted great attention in recent years, and the mechanical properties of both R_m above 1500 MPa and $R_m \times A$ more than 30 GPa · % are the important objects for the third-generation automotive steels^[3-5].

Speer et al.^[6,7] proposed a novel heat treatment process, i. e. the so-called quenching and partitioning (Q&P) process, aiming to produce steels with mixed microstructure of martensitic matrix and carbon-enriched austenite to improve the mechanical properties of high strength steels. Q&P process involves mainly quenching step and partitioning step. The former is to quench the austenized steel to a

temperature between the M_s (martensite-start temperature) and M_f (martensite-finish temperature) so as to create a mixture of martensite phase and untransformed austenite phase; the latter is to reheat directly to a higher temperature to make the untransformed austenite stable, during which carbon is expected to be rejected from supersaturated martensite phase into austenite phase. Finally, the steel is quenched to room temperature after Q&P process. A certain volume fraction of stable retained austenite contributes to the high strength and good ductility for Q&P steels^[4,8-13].

The austenite-to-martensite transformation can induce high hardening rate during deformation, which can improve both strength and ductility^[14-16]. It is found that $R_m \times A$ increases almost linearly with increasing austenite fraction, with an increment of 0.6 – 0.7 GPa · % per 1% γ ^[4]. In this paper, the Q&P processes were applied to low alloy steels, which is an attempt to obtain large-fractioned retained austenite and improve the ductility of ultra-high strength

steel through TRIP effect of retained austenite.

1 Experimental Procedure

Two low alloy steels were melted in a vacuum induction furnace, and forged into bar with diameter of 16 mm. The compositions of tested steels are given

Table 1 Chemical compositions of tested steels

Steel	C	Si	Mn	Cr	Ni	Mo	V	Nb	S	P	mass%
A	0.2	1.75	0.290	1.00	2.86	0.30	0.08	0.05	0.001	0.006	
B	0.4	1.70	0.027	1.05	1.83	0.62	1.68	—	0.001	0.005	

The specimens went through Q&P treatment as follows: the specimen was heated to 900 °C, held for 15 min, and then quenched in salt bath to quenching temperatures of 330 °C for steel A and 250 °C for steel B, respectively, held for 1 min, partitioned at 500 °C for 1 min in another salt bath, and finally quenched into water at room temperature. For comparison, traditional quenching and tempering (Q&T) processes were carried out as well: austenitizing at 900 °C for 15 min, quenching in oil at room temperature, and then tempering in a muffle furnace at 300 °C for 2 h.

Tensile test was performed at strain rate of 10^{-3} s^{-1} on the dog-bone shaped specimens with gauge length of 25 mm and diameter of 5 mm in an Instron machine (WE-300). The volume fractions of retained austenite were measured by X-ray diffractometer (XRD). Specimens were ground and mechanically polished, and then electrolytically polished in the mixture of chromic acid and distilled water (1 : 9). Specimens were scanned over a 2θ ranging from 45° to 115° with a step size of 0.02° and dwelling time of 2 s in PHILIPS APD-10 XRD, operated at 35 V and 35 mA with a graphite monochromatic and filtered cobalt radiation. Both austenite peaks of $\{200\}$, $\{220\}$, and $\{311\}$ and ferrite peaks of $\{200\}$ and $\{211\}$ were measured so as to calculate the volume fraction of austenite of each peak V_i based on Eq. (1), which is the average of V_i .

$$V_i = \frac{1}{1 + G(I_\alpha/I_\gamma)} \quad (1)$$

where, I_α and I_γ are the integrated intensity of ferrite and austenite peaks, respectively. The G value for each peak was used as follows: 2.5 for $I_\alpha(200)/I_\gamma(200)$, 1.38 for $I_\alpha(200)/I_\gamma(220)$, 2.02 for $I_\alpha(200)/I_\gamma(311)$, 1.19 for $I_\alpha(211)/I_\gamma(200)$, 0.06 for $I_\alpha(211)/I_\gamma(220)$, and 0.96 for $I_\alpha(211)/I_\gamma(311)$. Moreover, carbon content in austenite phase was calculated by Eq. (2)^[17].

In Table 1. Different carbon contents were used to evaluate the strength of steels and the stability of retained austenite. Meanwhile, Ni was added to improve the stability of retained austenite, and Mo and V were added to improve the strength of steels. Also, Nb was applied to refine grain size.

$$a_0 = 3.555 + 0.044x \quad (2)$$

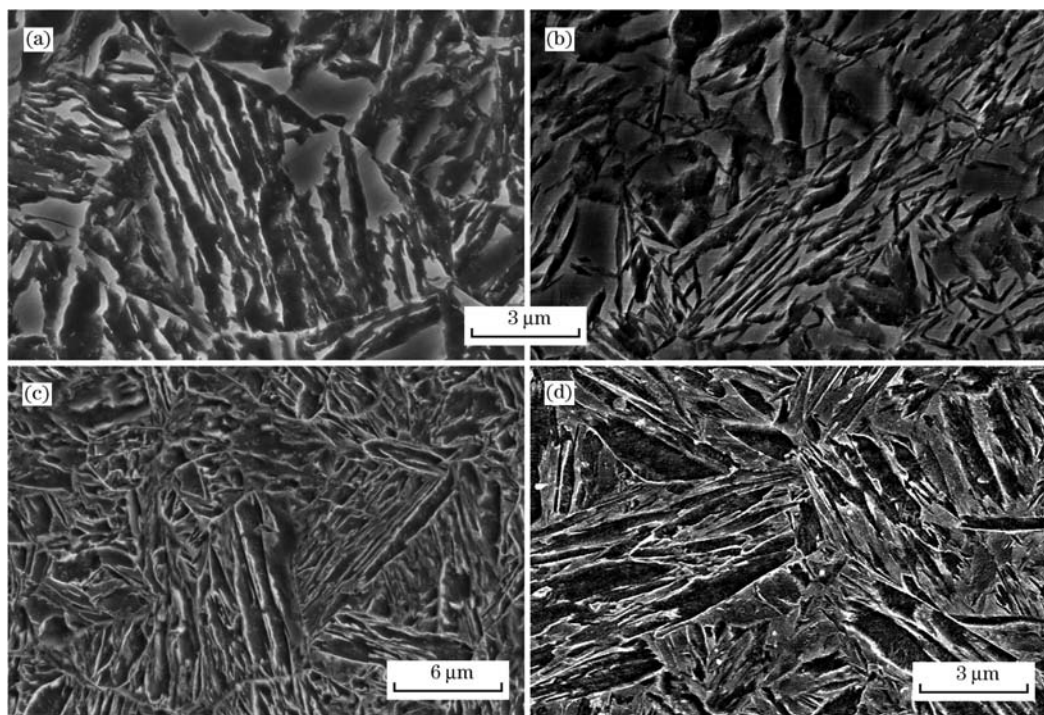
where, a_0 is austenite lattice parameter, nm; and x is carbon content, mass%. The lattice parameter was estimated from an average based on the austenite peaks of $\{220\}$ and $\{311\}$.

The microstructures were characterized by scanning electron microscope (SEM, S-4300) and transmission electron microscope (TEM, H-800), respectively. For the microstructure observation in scanning electron microscopy, samples were ground and polished mechanically, and then etched by 2% nital for 30 s. For the microstructure examination in transmission electron microscopy, samples were firstly ground mechanically to a thickness of about 0.04 mm, and then were electro-polished in a twin-jet machine in a solution of 5% perchloric acid and 95% alcohol at about -20°C .

2 Results and Discussion

2.1 Microstructure characterization

Both steels were processed by different heat treatments. The microstructures characterized by SEM are shown in Fig. 1. For the specimens treated by Q&P process (Figs. 1(a) and 1(b)), not only lath martensite but blocky phase with size of 1 μm to 3 μm can be observed, and the blocky phase is difficult to be examined without further heat treatment. For the specimens treated by Q&T process, traditional lath martensite is obtained, and the block and packet in microstructure can be clearly observed, as shown in Figs. 1(c) and 1(d). The fine microstructure of blocky phase in Q&P processed specimens can be obtained after tempering at 200 °C for 1 h (Fig. 2). It is well known that the tempered martensite is prone to be etched due to the precipitation nucleation and growth of the cementite, while the fresh martensite is difficult to be etched. According to the difference, it can be concluded that the easily etched



(a) Steel A after Q&P process; (b) Steel B after Q&P process; (c) Steel A after Q&T process; (d) Steel B after Q&T process.

Fig. 1 SEM images of steels after heat treatments

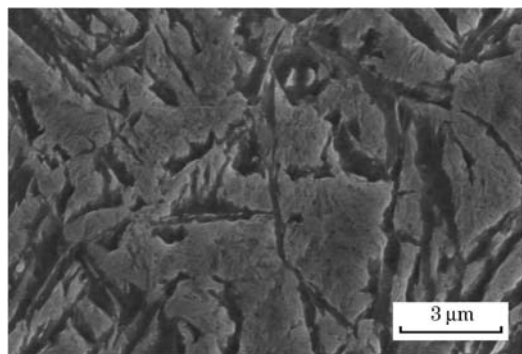


Fig. 2 SEM image of steel A tempered at 200 °C for 1 h after Q&P process

matrix in Q&P processed specimens is initial martensite formed during the first quenching, which is tempered during partitioning, and the blocky phase is the fresh martensite or M/A island formed during the final quenching of Q&P process^[18,19].

The TEM investigations indicated that the fine microstructure of Q&P processed steels consists of lath martensite and retained austenite, as presented in Fig. 3. It is interesting to find that many grains in TEM images have almost the same size and shape with those of the blocky phase in SEM images, in which only one single set of packet with the lath thickness of about 0.1–0.2 μm is identified as fresh

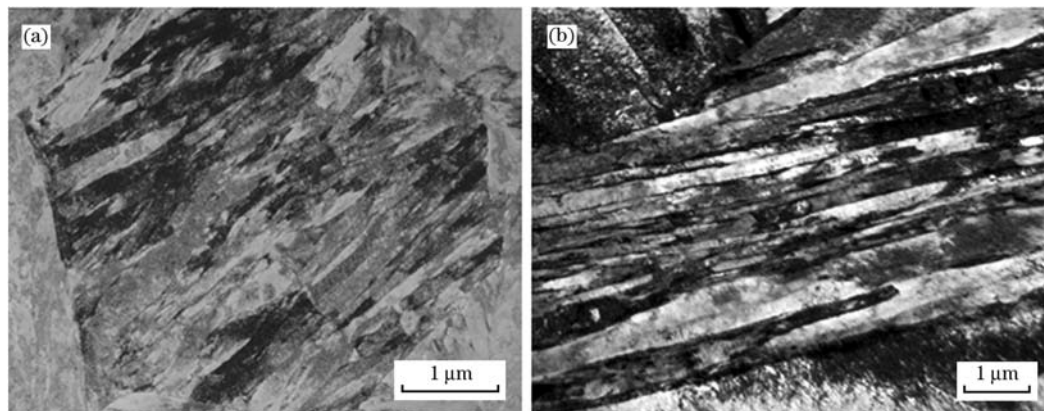
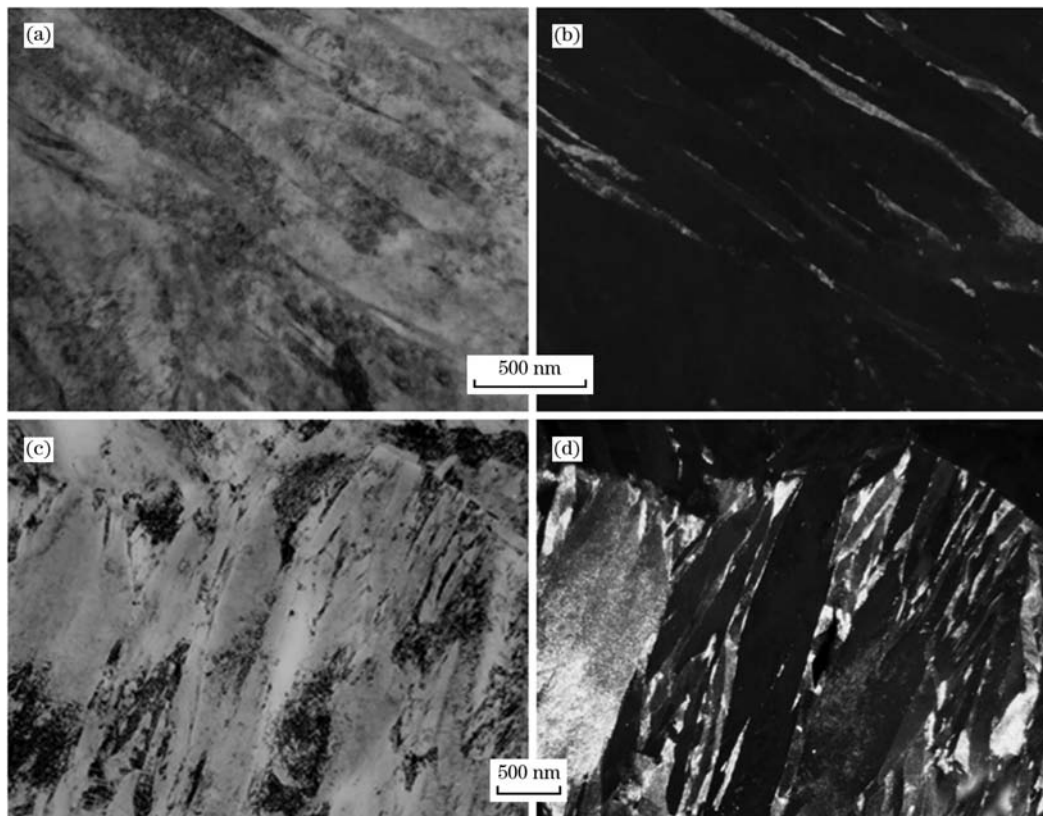


Fig. 3 TEM images of fresh martensite (a) and initial martensite (b)

martensite formed during final quenching, thinner than the initial martensite with the lath thickness of $0.2\text{--}0.3\ \mu\text{m}$ ^[18]. Because of carbon partitioning behavior, both hardness and carbon contents of fresh martensite are higher than those of martensite matrix^[18]. The austenite morphology in Q&P steels was characterized by TEM, as shown in Fig. 4. Two

types of retained austenite morphologies can be observed; one type is film-like retained austenite with the thickness less than 100 nm, and the other is fine blocky austenite. Based on TEM images, it was found that the fraction and the grain size of retained austenite in the steel B are much higher than those in steel A.



(a) Bright field image of steel A;
(c) Bright field image of steel B;

(b) Dark field image of steel A;
(d) Dark field image of steel B.

Fig. 4 Morphology of retained austenite in specimens after Q&P process characterized by TEM

The volume fractions of retained austenite were measured by XRD. It is found that the volume fraction of retained austenite is 11.9% for steel A and 27.3% for steel B processed by Q&P treatments, respectively; they are much higher than those treated by traditional Q&T process, which are 3.2% for steel A and 4.4% for steel B.

It can be demonstrated that Q&P process is an effective way to obtain mixed microstructure with characteristics of multiphase, metastable, and multi-scale. Multiphase involves the initial martensite, fresh martensite, and retained austenite. The initial martensite formed during first quenching has relatively lower carbon content and hardness after partitioning. The fresh martensite has one single set of packet and thinner lath; it was transformed from carbon-en-

riched austenite during final quenching stage and has higher carbon content and hardness. The retained austenite is finally preserved after partitioning and carbon enrichment during Q&P. For steel B, a mixed structure of initial martensite and untransformed austenite was obtained after quenching to 250 °C, and the volume fraction of each phase can be estimated by Eq. (3)^[20]. When the specimens were reheated to a partitioning temperature of 500 °C, the carbon partitioned into untransformed austenite from initial martensite and stabilized it, and much more retained austenite could be retained after final quenching, because the precipitation was effectively suppressed by silicon. However, for the specimens treated by Q&T process, almost all the austenite was transformed into martensite and only a little

austenite remained. Moreover, multi-scale micro-structural characteristics were obtained in Q&P processed specimens; not only normal sized lath, block and packet for initial martensite, but also fine sized fresh martensite coexist, and the fresh martensite has a grain size of 1–3 μm and consists of one single packet and thinner lath with the thickness of about 100 nm.

$$f_\gamma = 1 - f_M = \exp(-\alpha(M_s - M_q)) \quad (3)$$

where, f_γ and f_M are volume fraction of retained austenite and martensite, respectively; M_q is quenching temperature; and α is a constant and equal to 1.1×10^{-2} .

The average carbon content of steels greatly affects the volume fraction of retained austenite. The average carbon contents are 0.2 mass% for steel A and 0.4 mass% for steel B, and the corresponding volume fractions of the retained austenite are 11.9% for steel A and 27.3% for steel B after Q&P treatments, respectively. The higher volume fraction of retained austenite was obtained for steel B because its average carbon content equals twice that of steel A. During Q&P process, in the case of obtaining the same fraction of initial martensite and untransformed martensite after the first quenching stage, the initial martensite which has higher average carbon content can provide more carbon atoms to stabilize untransformed austenite during partitioning, and result in higher volume fraction of retained austenite. Therefore, it can be concluded that increasing average carbon content of steel is an effective way to obtain much more retained austenite.

2.2 Work hardening behavior

The average uniaxial tensile properties of two specimens for different steels and heat treatments are given in Table 2. It can be seen that excellent mechanical properties of specimens were obtained after Q&P. For steel B, the specimens treated by Q&T have ultra-high tensile strength (1945 MPa), high yield stress (1665 MPa), and low total elongation (10.5%). Compared with specimens treated by Q&T, the tensile strength and yield stress of specimens treated by Q&P process decrease to 1835 MPa and 740 MPa, respectively, while the total elongation significantly increases up to 19.0%, and the value of $R_m \times A$ reaches 34.9 GPa · %. The same change trend happened for steel A, and its value of $R_m \times A$ reaches 28.9 GPa · %. It is suggested that Q&P process can greatly decrease yield strength, increase total elongation and the value of $R_m \times A$.

Table 2 Mechanical properties of the tested steel

Steel	Process	R_m / MPa	$R_{p0.2}$ / MPa	A_{gt} / %	A / %	$R_m \times A$ / (GPa · %)
A	Q&P	1390	1025	10.5	20.8	28.9
A	Q&T	1520	1320	3.5	13.0	19.7
B	Q&P	1835	740	14.0	19.0	34.9
B	Q&T	1945	1665	3.5	10.5	20.4

Note: $R_{p0.2}$ —Yield strength; A_{gt} —Uniform elongation.

The tensile stress-strain curves and the corresponding work-hardening curves of two tested steels are shown in Fig. 5. There are great differences between specimens treated by Q&P and Q&T processes. The uniform elongation of specimens treated by Q&P process is much higher than that treated by Q&T process; that is, the work hardening behavior of specimens treated by Q&P processes occurs at a large strain range. The curves of work-hardening rate ($ds/d\varepsilon$) vs. true tensile strain ε are shown in Fig. 5(b). With the increase of tensile strain, the work-hardening behavior of specimens treated by Q&P presents three stages; the work hardening rate firstly decreases (stage I), then increases (stage II) and finally decreases again (stage III). However, compared with the above-mentioned specimens treated by Q&P, the work

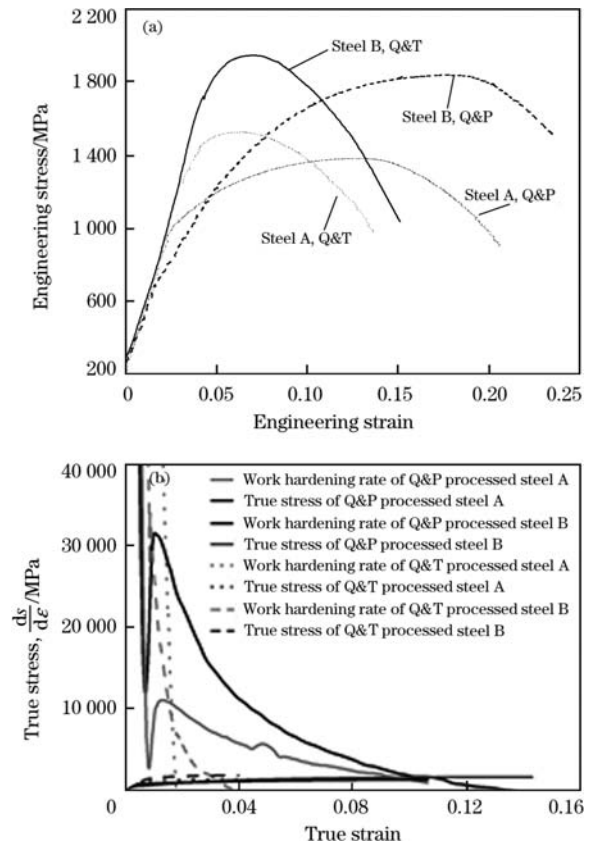


Fig. 5 Tensile stress-strain curves of tested steel (a) and corresponding work-hardening curves (b)

hardening behavior of specimens treated by Q&T processes presents only sharp decrease instead. The work hardening rate of steel B treated by Q&P is much higher than that of steel A treated by Q&P process at the same tensile strain, and the necking was precluded so as to greatly increase the uniform elongation.

The XRD measurement revealed that the austenite fraction decreases monotonically with the increase of tensile strain for the two Q&P steels, as shown in Fig. 6. It indicates that the austenite transforms into martensite during tension. Moreover, it is surprising to find that about two-thirds of retained austenite has been transformed at the strain of only 3% for steel B, and over one-third of retained austenite has transformed at the strain of only 2% for steel A. Especially, with the increase of tensile strain, the austenite transformation becomes slow. It is necessary to indicate that the stability of retained austenite in Q&P steels is worth studying because it strongly affects the TRIP behaviors.

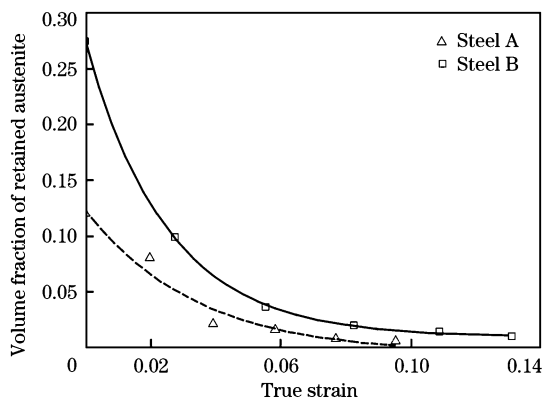


Fig. 6 Curves of volume fraction of austenite vs. true tensile strain

At the beginning of the tension, the work-hardening rate, which decreases with the increase of tensile strain, mainly derives from the dislocation hardening of initial martensite, while at stage II, the fraction and strength of martensite increase with increasing tensile strain owing to the carbon-enriched retained austenite transforming into martensite, thus increasing the work hardening rate. With the continuous transformation of retained austenite, the work hardening rate finally decreases again at stage III. The TRIP effects of retained austenite may provide work hardening rate, retard the formation of necking and improve the uniform elongation of steels.

2.3 Stability of retained austenite

The relationship between the volume fraction of

retained austenite and tensile strain can be simulated by the exponent decay law proposed by Sugimoto et al.^[21] (Eq. (4)). The constant k is important to evaluate the stability of retained austenite, and the higher the k value is, the more easily retained austenite transforms into martensite. The values of k for Q&P processed specimens are 14.94 for steel A and 10.91 for steel B, respectively, as shown in Fig. 6, and the relationship between $(\log f_{\gamma_0} - \log f_{\gamma})$ and true tensile strain is shown in Fig. 7. The k values of present Q&P steels are higher than those of TRIP steels (shown in Table 3). It indicates that the stability of retained austenite in Q&P steels is much lower than that in TRIP steels, and the stability of retained austenite in steel B is slightly higher than that in steel A treated by Q&P process.

$$\log f_{\gamma_0} - \log f_{\gamma} = k\varepsilon \quad (4)$$

where, f_{γ_0} and f_{γ} are the volume fraction of retained austenite before and after deformation, respectively.

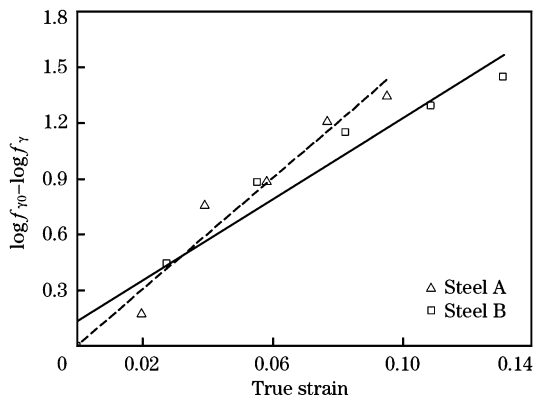


Fig. 7 Relationship between $\log f_{\gamma_0} - \log f_{\gamma}$ and ε

Table 3 k value of different steels

Steel	k value	Source
0.4%C-0.49%Si-1.48%Mn-0.96%Al	<3.5	Ref. [22]
0.2%C-1.51%Si-1.51%Mn	<6.0	Ref. [23]
Steel A	14.94	This study
Steel B	10.91	This study

It is well known that the stability of retained austenite is significantly affected by its chemical composition^[24-26], grain size^[27-29], morphology^[30] and surrounding phase. The carbon contents of retained austenite in Q&P steels were estimated at 0.99 mass% for steel A and 1.10 mass% for steel B, respectively, based on the relationship between lattice parameter and carbon content expressed as Eq. (2). Assuming that all alloying elements except carbon were homogeneously distributed, it is possible to estimate the martensitic transformation start tempera-

ture for both steels by the following empirical equation of Eq. (5)^[31], and the M_s temperatures are 45.9 °C for retained austenite in Q&P steel A and -0.6 °C for retained austenite in Q&P steel B, respectively, which indicates that both steels treated by Q&P processes have higher chemical stability, and the retained austenite in steel B is more stable than that in steel A.

$$M_s = 539 - 423w_C - 30.4w_{Mn} - 17.7w_{Ni} - 12.1w_{Cr} - 7.5w_{Mo} \quad \text{°C} \quad (5)$$

where, w_C , w_{Mn} , w_{Ni} , w_{Cr} and w_{Mo} are the mass percent of carbon, manganese, nickel, chromium and molybdenum, respectively.

Both film-like austenite and small sized blocky austenite have been observed in steels treated by Q&P process from the TEM characterization. Xiong et al.^[32] describes that film-like austenite is more stable than the blocky austenite during deformation, and when the strain exceeded 12%, all the blocky austenite had been transformed into twinned martensite, while film-like austenite was still present. It is concluded that small sized blocky austenite was transformed into martensite gradually during tensile deformation, and thus transformation increased the work hardening rate of stage II, as shown in Fig. 5(b). It demonstrates that when grain size exceeds 1 μm, the retained austenite has lower stability^[33], and the micro-sized retained austenite is stable enough to preserve even at necking location^[34,35]. The preliminary study results show that retained austenite with size of less than 0.1 μm has been observed in steel A treated by Q&P process, and the retained austenite is mainly distributed on the boundary of packet and original austenite grain^[18]. Thus, the retained austenite is stable enough in case of grain size.

Q&P process fabricates a mixed microstructure of retained austenite surrounded by high strength martensitic matrix. Thus, stress can easily transfer to retained austenite during tension, inducing its transformation into martensite at the early stage of tension strain^[36], especially for the retained austenite at the boundary of packet and original austenite grain, because it is easy to cause stress concentration in boundary region^[4]. However, for traditional TRIP steels, retained austenite is surrounded by ferrite and bainite, whose hardness is much lower than martensite in Q&P steels; thus, the relatively soft matrix reduces the stress concentration in austenite and improves the retained austenite in traditional TRIP steels with higher stability than in Q&P steel.

3 Conclusions

Excellent mechanical properties with R_m more than 1835 MPa and $R_m \times A$ above 34.9 GPa · % are obtained for low alloy steel treated by Q&P. The TRIP effect of retained austenite retards the formation of necking and greatly improves the uniform elongation. Both the hard matrix (initial martensite and fresh martensite) and ductile retained austenite give excellent combination of strength and ductility.

Due to the retained austenite in Q&P treated steel surrounded by martensite, the stress can easily transfer to retained austenite during tension, which may lead to the austenite transforming into martensite in the early stage of strain.

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