Enhanced Stability of Retained Austenite by Quenching and Double Partitioning Process

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Abstract: In the present study, a novel quenching and double partitioning (Q&DP) process is proposed. The quenching and partitioning (Q&P) process was accompanied by a second partitioning process, which led to the enhanced stability of retained austenite. The chemical composition of the investigated steel was 0.24C-1.9Mn-1.85Si, without an excess addition of alloying elements. This process aims to enhance the stability of retained austenite by controlling martensitic transformation and carbon partitioning. By applying this process, the uniform and total elongation is increased, without obvious reduction in ultimate tensile strength. An optimum combination of strength and ductility (ultimate tensile strength: 1304MPa; total elongation: 23%) was achieved by quenching to 65°C and subsequent second partitioning treatment, after the first quenching to 300°C and partitioning process. The enhanced mechanical properties were attributed to the increased amount of stabilized film-like austenite.

1. Introduction
The quenching and partitioning(Q&P) process, a promising method to produce materials with high strength and excellent ductility [1], has attracted increasing attention in the past decade. The Q&P process starts from either a full austenization state or an intercritical annealing state. Then, the steel is quenched to a temperature(QT) between martensite starting transformation temperature (MS) and martensite finishing transformation temperature(Mf), followed by a partitioning process either at or above the quenching temperature, and finally quenched to room temperature [2]. The austenite is enriched with carbon during the partitioning treatment and stabilized to room temperature [3].

Based on the CCE model, there is an optimum quenching temperature(OQT) at which the maximum austenite stabilization and retention can be obtained [4]. Fresh martensite, containing a higher concentration of carbon than tempered martensite, is detrimental for ductility [5]. It has been reported [6] that the presence of fresh martensite readily triggers microcrack and subsequently deteriorates toughness; and it also has a negative influence on the stability of austenite transformation. A second martensitic transformation will apply if the austenite is still unstable after the partitioning process. Meanwhile, second martensitic transformation temperature (M₃') is reduced to a lower temperature [7]. When the unstable austenite is quenched to a temperature between the M₃' and the M₇', again, and followed by a second partitioning process, the fresh martensite is reduced to a lower fraction and the retained austenite is enriched with higher carbon content. Gao’s study [8] on the
tempering behavior of Q&P process focused on the different tempering temperature. However, there are less detailed studies about the mechanism of the subsequent process.

In this paper, the microstructural evolution before and after the second partitioning process of a 0.24C-1.9Mn-1.85Si (wt.%) steel was analyzed. The effect of the second partitioning process was investigated based on experimental investigation, and the relationship between the retained austenite and fresh martensite was discussed in detail.

2. Experimental procedure
A traditional Q&P steel with chemical composition of Fe–0.24C–1.9Mn–1.85Si (wt.%) was used in this study. An ingot with thickness of 30 mm was firstly cast after melting in vacuum induction furnace, then homogenized at 1200 °C for 2h, and finally hot rolled to 3 mm sheets followed by air-cooling to room temperature. The sheet was further cold rolled to the final thickness of 1.8 mm after pickling in 10% hydrochloric acid.

The dilatometry study was carried out on a Bähr D805L quenching device equipped with quartz push-rods. The critical temperatures of $A_{C1}$, $A_{C3}$ and $M_S$ of the investigated steel are 725°C, 875°C and 360°C respectively. Specimens with dimensions of 200×60×1.8 mm$^3$ were machined for heat treatments. The specimens were austenitized at 910°C for 5min in a tube furnace, and then quenched to 260°C, 280°C, 300°C and 320°C for 10s in a salt bath, which was marked as Q260, Q280, Q300 and Q320 respectively. After that, they were transferred to another salt bath with a temperature of 400°C for 100s. Finally, the specimens were water quenched to room temperature. Heat treatments were applied to investigate the influence of the second partitioning. After quenched to 300°C and partitioned at 400°C, the specimens were quenched to 18°C (room temperature) and 65°C again, which was marked as Q300-18 and Q300-65 respectively. After that, the samples were partitioned at 400°C for 100s again, followed by quenching to room temperature.

Tensile specimens with 25mm in gauge length and 12.5mm in gauge width were prepared along the rolling direction, and the tensile tests were performed at room temperature with the strain rate of approximately 1.33×10$^{-3}$ s$^{-1}$. Metallographic specimens were ground, polished and etched with 4% nital, and observed by field emission scanning electron microscopy (FE-SEM). The specimens for TEM observation were firstly ground to a thickness of 50 μm and then twin-jet electro-polished at -30°C in an electrolyte consisting of 8% perchloric acid 92% acetic acid. The volume fraction of retained austenite was measured via X-ray diffraction (XRD) instrument equipped with a Cu tube. Samples were scanned from 40° to 100° at a scanning rate of 2°/min. The amount of retained austenite was obtained using the following equation [9]

$$V_r=1.4I_r/(I_r+1.4 I_o)$$  \(1\)

Where $V_r$, $I_r$ and $I_o$ are the volume fraction of retained austenite, the average integral intensity of the (200), (220) and (311) austenite peaks and the average integral intensity of the (200) and (211) ferrite peaks. The carbon content was calculated by the following equation [10]

$$a_g (nm)=0.3556+0.00453XC+0.000095XMn$$  \(2\)

Where $a_g$ is the austenite lattice parameter in nm; $X_C$ and $X_Mn$ are the concentrations of carbon and manganese in austenite in wt.%, respectively.

3. Results and discussion

3.1. Mechanical properties and work hardening behavior
As is shown in Fig. 1a, the ultimate tensile strength of the samples ranges from 1300MPa to 1320MPa. The Q280 sample exhibits higher elongation than the other samples. The increase in elongation is due to the higher fraction of retained austenite which provides more effective Transformation-Induced Plasticity (TRIP) effect than the other samples. Similarly, the ultimate tensile strength of the samples changes little in Fig. 1b, whereas the Q300-65 shows better elongation, even better than Q280 which contains more retained austenite. The enhanced amount of retained austenite in the partitioning
process contributes to the TRIP effect during the deformation and also favors strength and elongation [11].

![Fig. 1](image1)

**Fig. 1** Mechanical properties of the samples: (a) Q260-Q320, (b) Q300, Q300-18 and Q300-65

The engineering stress-strain curves of Q300 and Q300-65 are illustrated in Fig. 2. It is concluded in Fig. 2a that the elongation of Q300-65 increased by 2% without apparent sacrifice in strength. The increase in elongation is caused by the improved amount of film-like austenite which provides a continuous TRIP effect and effectively enhances elongation. The film-like austenite is more stable for high strains than blocky retained austenite [12]. The fresh martensite formed in the quenching process is tempered and the rest austenite is carbon-rich. In the end, there is a slight decrease in ultimate tensile strength and a slight increase in elongation.

![Fig. 2](image2)

**Fig. 2** (a) nominal stress-strain curve and (b) work hardening of Q300 and Q300-65

The working hardening rate and true stress curves of Q300 and Q300-65 are presented in Fig. 2b. The working hardening rate descends quickly as the strain lower than 0.04, and decreases slowly at a strain higher than 0.04. The Q300-65 sample shows a steady and prolonged decline in working hardening rate than Q300 one. The transformation of the retained austenite to martensite can cause a local work hardening effect to delay the start of necking, which finally contributes to the higher uniform elongation. The TRIP effect of retained austenite improves the work hardening rate at high strain level and thus the uniform elongation as mentioned above.

3.2 Volume fractions and carbon content of retained austenite

The volume fraction and carbon content of retained austenite determined on the basis of XRD results are presented in Fig. 3. The volume fraction of retained austenite increases when the quenching temperature rises from 260°C to 280°C, and decreases gradually with further increase in quenching temperature (Fig. 3a). It has been mentioned above that there exists an optimum temperature which yields a maximum amount of retained austenite. It is evaluated by the lever rule on the base of the dilatometer data that the quenching temperature of 280°C gives rise to about 18% retained austenite, which is close to the optimum temperature[2,5]. However, XRD results of the retained austenite are below the calculated values. There are some reasons that affect the ultimate amount of retained austenite. First of all, for the calculated results, it is assumed that the carbon is all concentrated into
austenite after the partitioning process. Nevertheless, the distribution of carbon is inhomogeneous, which may increase the time needed for homogeneous state. Secondly, the formation of bainite during the partitioning process competes with carbon partitioning and leads to the decrease in the volume fraction of retained austenite. Finally, though a substantial fraction of Si was added to the steel, the formation of cementite or transition carbide in martensite is observed during the partitioning step [13], and the remaining amount of carbon in martensite is reduced.

![Figure 3](image-url)

**Fig. 3** Volume fraction and carbon concentration of retained austenite in the samples

However, as shown in Fig. 3b, when the second partitioning process is added to the Q300 process, the carbon content increases obviously, especially when the quenching temperature before second partitioning is 65°C. A comparison between Q300 and Q300-65 indicates that both the volume fraction and the carbon content increase in the latter process. The second partitioning process enriches the austenite with carbon and increases the amount of the film-like austenite.

### 3.3 Microstructure properties relationship

Fig. 4 shows the TEM micrographs of samples treated by Q300 and Q300-65 process. Fig.4a shows the grain boundary of Q300. Martensite laths are parallel, and no retained austenite is identified. Fig. 4b–g shows the original austenite grain boundary of Q300-65. There is film-like austenite around the grain boundary, which is not found in Q300 (Fig.4b and e). Fig.4c and f are the dark field images of Fig.4b and e, in which the retained austenite is white. Fig.4d and g are the corresponding selected area electron diffraction (SAED) images of Fig.4b and e. It demonstrates that the second partitioning process increases the fraction of film-like austenite by controlling the martensitic transformation, especially the microstructure around the grain boundary, which is supposed to be blocky martensite by traditional process. The partially transformed austenite offers carbon for the untransformed austenite, and the stable austenite with higher carbon content is finally conserved to room temperature. Fig. 4c and f show that the film-like retained austenite around the grain boundary is accompanied by the martensite laths. It is considered that the film retained austenite is more stable than the blocky retained one. Additionally, the hard martensite phase surrounding film-like retained austenite provides high three-dimensional hydrostatic pressure which constrains the volume expansion and shear deformation [14], sustaining work hardening behavior at higher strain level.

In summary, the Q&DP process increases the fraction of stable film-like austenite compared with the Q&P process. The higher carbon content of the retained austenite of the Q&DP process improves the work hardening rate and the elongation by the TRIP effect. These characteristics provide the superior combination of strength and ductility (the ultimate tensile strength: 1304MPa; total elongation: 23%) by Q300-65 process.
4. Conclusions

The microstructure and mechanical properties of a traditional Q&P steel treated by Q&DP process have been studied. The main conclusions are as follows:

(1) The final microstructure of the steel is mainly composed of the initial martensite, retained austenite, fresh martensite and a part of bainite formed during the partitioning process. The Q280 process which is close to the optimum temperature yields a maximum amount of retained austenite.

(2) When the carbon is not enough to stabilize all the austenite in the first partitioning process, fresh martensite will form during the subsequent cooling process. By controlling the martensitic transformation, the second partitioning process can reduce the fraction of fresh martensite and enhance the amount of film-like retained austenite.

(3) The sample Q300-65 treated by the Q&DP process contains more film-like retained austenite, which offers a better combination of strength and ductility than the microstructure from the traditional Q&P process.

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