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Effects of Compositional Inhomogeneity on the Microstructures and Mechanical Properties of a Low Carbon Steel Processed by Quenching-Partitioning-Tempering Treatment

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Abstract: Quenching-partitioning-tempering (Q-P-T) heat treatment is a relatively novel approach to attain excellent ductility in high-strength steels. In the present work, the microstructural evolution and the mechanical properties of a low carbon microalloyed advanced steel were systematically investigated after the Q-P-T process. The microstructural evolution was explored by employing X-ray diffraction, transmission electron microscopy and scanning electron microscopy. The results indicate that the multiphase microstructures strongly depend on both the initial microstructure and the processing parameters of the quenching and partitioning process, especially the quenching temperature. Compositional inhomogeneity during the Q-P-T process results in multiphase microstructures, in which the mechanical properties of the quenching and partitioning steels may be strongly impacted by the distribution of heterogeneous austenite phase in the steel matrix.

Keywords: microstructure evolution; advanced high-strength steel; SEM; TEM; orientation relationship



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1. Introduction

The third generation of advanced high-strength steels (AHSSs) was developed to lighten automobiles and improve fuel efficiency [1–3]. Several types of Fe-Mn-Si-based steels have been studied intensively, such as dual-phase steel [4,5], transformation-induced plasticity steel (TRIP) [6,7], and twinning-induced plasticity steel (TWIP) [8]. However, the strengths of the advanced high-strength steels are typically lower than 1000 MPa [9]. A new heat treatment technique was proposed for enhancing the mechanical properties of the steels, namely the quenching and partitioning (Q&P) process [10–12]. In the Q&P process, a Si-containing steel is rapid cooled (first quenching) from an austenitizing temperature to a quenching temperature (T_q) between the martensite-start temperature (M_s) and martensite-finish temperature (M_f), followed by a ‘partitioning’ treatment, where the carbon atoms can diffuse from the martensite phase to the adjacency untransformed austenite phase during the isothermal holding. When the partitioning temperature equals the quenching temperature, it is denoted as “one step quenching and partitioning” treatment; otherwise, it is named “two step quenching and partitioning” treatment, where the partitioning temperature is usually above the martensite-start temperature. After the partitioning treatment, the carbon-enriched retained austenite can be stabilized in the subsequent quench (final quench) to room temperature. Compared with other AHSSs, the strength of Q&P steel can be increased while the plasticity of the steels can be guaranteed due to the martensite plus retained austenite dual phase structure. [13–15].

Afterwards, in order to utilize the carbide precipitation for strength enhancement, a innovative heat treatment process, namely quenching-partitioning-tempering (Q-P-T) was proposed by T. Y. Hsu [16], which also can obtain the martensite plus retained austenite

dual phase structure in microalloyed steels. The following are the primary distinctions between the Q&P and Q-P-T treatments: (i) alloying elements such as Nb and V, which can cause carbide formation and grain refinement in steel, are added to Q-P-T steels but not to Q&P steels; (ii) tempering temperature depends on the temperature for better carbide precipitation, during which the partitioning of carbon atoms can take place. This implies tempering includes partitioning of carbon atoms [16,17]. The Q-P-T steel matrix consists of the dual microstructure with martensite laths and film-like retained austenite located between the martensite laths, which can lead to the good combination of high strength and adequate plasticity. In addition, the alloy carbides with controlled particle sizes distributed in the martensite lath make the strength and hardness of Q-P-T steel higher than the Q&P steel. According to earlier research, the martensite/austenite fraction of the steels can be tuned to optimize their mechanical properties [18–20]. However, the microstructural evolution of the Q-P-T steels under the process of heat treatment is rather complicated, as there can be several competing reactions/phase transformations during the quenching-partitioning-tempering treatment [10,21,22]. Qin et al. [23,24] investigated the microstructural evolution and mechanical properties of a high carbon steel (C~0.6 wt.%) subjected to Q-P-T treatment and found that the ductility of steels could be enhanced by a relatively high-volume fraction of retained austenite using two strategies: one is the dislocations absorbed by the retained austenite (DARA) effect, and the other is the TRIP effect during deformation. The DARA effect can enhance the deformation ability of the hard martensite, and the strain-induced martensitic transformation of austenite can relax the stress concentration and thus prevent the formation of cracks effectively. However, the effect of retained austenite related to ductility in low carbon steel might be somewhat different from that of the high carbon steels because the amount of retained austenite is relatively low, and the morphology and distribution of retained austenite are also different. The microstructural evolution can determine the mechanical properties of the steels. Thus, a high-strength low-carbon steel was quenched to different T_q and then partitioned at medium temperature (e.g., 400 °C) for various times to reveal the relationship between mechanical properties and microstructural evolution for advanced high-strength steel. The microstructural evolution of the treated steel is discussed with the purpose of controlling and optimizing the mechanical properties of the quenching partitioning steels.

2. Material and Methods

2.1. Material and Specimens

The alloy composition of the microalloyed steel (in weight %) studied is Fe-0.21C-1.8Mn-1.51Si-0.028Nb-0.15Mo-0.025Cr, as listed in Table 1. Here, Si is used to suppress the formation of cementite, therefore to boost the partitioning of carbon from the martensite phase to the austenite phase during the heat treatment [3]. Mn can increase the stability of the austenite phase and decrease the martensite transformation temperature in steel. Microalloyed elements such as Nb, Mo, and Cr [25,26] can help to decrease the primary austenite grain size by pinning the grain boundaries and make a contribution to carbide dispersion precipitation strengthening in the steels. The steel was melted in a laboratory medium frequency furnace in the Technology Center of Baosteel Group (Shanghai, China). According to the actual process of production, the slab was homogenized at a temperature of 1230 °C for one hour, the ingot was forged to a thickness of 35 mm, and then the slab was hot rolled. The multi-pass hot rolling was split into 5 passes with a total reduction of 80%, starting at a temperature of 1130 °C and finishing at the temperature of 780 °C, and then air cooled down. The recrystallization temperature of the steel is about 950 °C. The austenitizing and martensite start temperatures, which were measured with a Formaster FII dilatometer (Fuji Electronic Ind. Co., Japan) are 860 °C and 380 °C, respectively. For the Q-P-T process, the as-received samples were austenitized at 910 °C for 360 s, followed by rapid quenching to a mix-salt bath at 220 °C, 250 °C and 280 °C for about 10 s respectively, and then was held at 400 °C salt bath for 12 s, 30 s, 90 s, 300 s, 600 s respectively for studying the effect of quenching temperature and partitioning/tempering time on the

completing partitioning and tempering process, and was finally water quenched. It should be pointed out that the partitioning/tempering temperature and time during Q-P-T process are selected to be favorable for the precipitation of dispersive carbides in the martensite matrix, instead of just satisfying the carbon partitioning during Q&P process [24].

Table 1. The composition of the microalloyed steel (in weight %).

Fe	C	Si	Mn	Nb	Mo	Cr	S	P
balance	0.21	1.51	1.8	0.028	0.157	0.025	0.003	0.004

2.2. Testing Conditions

The axes of the specimens for the tensile testing were cut parallel to the rolling direction. Gauge dimensions are 20 mm in width and 70 mm in length. The samples were evaluated at room temperature using a Zwick T1-FR020TN A50 machine (Zwick Roell, Ulm, Germany). The microstructural investigation was carried out for the Q-P-T treated samples. For metallographic characterization, samples were prepared by an etchant of 4% nital solution with ethanol as solvent, and then the samples were investigated using a JEOL-7500F (JEOL Ltd., Tokyo, Japan) scanning electron microscope operating at 10 kV. The distribution of austenite was characterized by electron backscattering diffraction (Oxford Instruments, Bristol, UK). The electron backscattering diffraction (EBSD) sample was electro-polished in a solution consisting of 10 vol% perchloric acid and 90 vol% glacial acetic acid at -10°C for about 20 s. The specimens for transmission electron microscopy (TEM) observation were abraded to a thickness of about 80 μm . Subsequently, these thin foils were electropolished at -25°C by an MTP-1A twin-jet polisher (Ruida, Shanghai, China) using an electrolyte consisting of 4 % perchloric acid and 96 % ethanol solution. All the chemicals used in the study were purchased from Sinopharm Chemical Reagent Co., Ltd., Shanghai, China. In the sample preparation, in order to prevent the sample from oxidizing and deteriorating to the greatest extent, the temperature of the electropolishing was set at -25°C [27]. TEM observation was performed using a JEOL-200CX (JEOL Ltd., Tokyo, Japan) microscope operating at a voltage of 200 kV. In order to determine the volume fraction of austenite in the treated samples, the X-ray diffraction (XRD) experiments were carried out in a D/max 2550 X-ray diffractometer (Rigaku Corporation, Tokyo, Japan) at a voltage of 30 kV with copper $\text{K}\alpha$ radiation. A direct comparison method [14] was used to evaluate the quantity of retained austenite in a semi-quantitative manner by comparing the integrated intensities of the 200γ , 220γ , and 311γ austenite peak and the 211α and 200α martensite peak, respectively.

3. Results and Discussion

3.1. Microstructural Evolution

The as-received steel mainly consists of elongated ferrite grains and the particles that are distributed in the ferrite grain boundaries. These particles are in irregular shapes, and the energy-dispersive X-ray spectroscopy results reveal that the particles are enriched in Mn, which implies that these particles are cementites or martensite/austenite islands according to the composition and the heat treatment procedure of the steel, as shown in Figure 1.

The OM image in Figure 2a and the SEM image in Figure 2b reveal clearly lath martensite in the quenched steel. The average grain size of the prior austenite phase is about 8 μm , which is consistent with the grain size for the as-received samples. The addition of Nb and Mo reduce the grain size of the steel as compared to lean steel without such microalloying elements heat treated by similar quenching and partitioning patterns and whose grain size is about 20 μm [28]. The microstructure of the steel grain is typical lath martensitic matrix, with a high density of dislocations (Figure 2c). The dark field image in Figure 2d shows the morphology of the retained austenite phase located between the

martensite laths has a thin film-like shape, and the austenite plate width is about 20~30 nanometers on average.

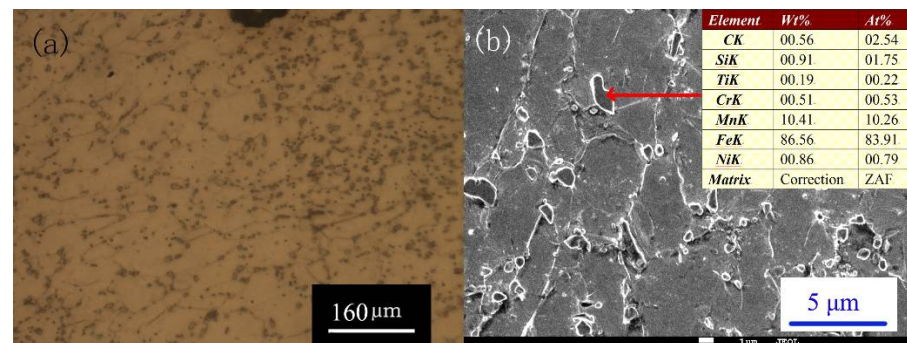


Figure 1. Optical micrograph (a), secondary electron micrograph (b), and the energy-dispersive X-ray spectroscopy results (inset) of the as-received steel.

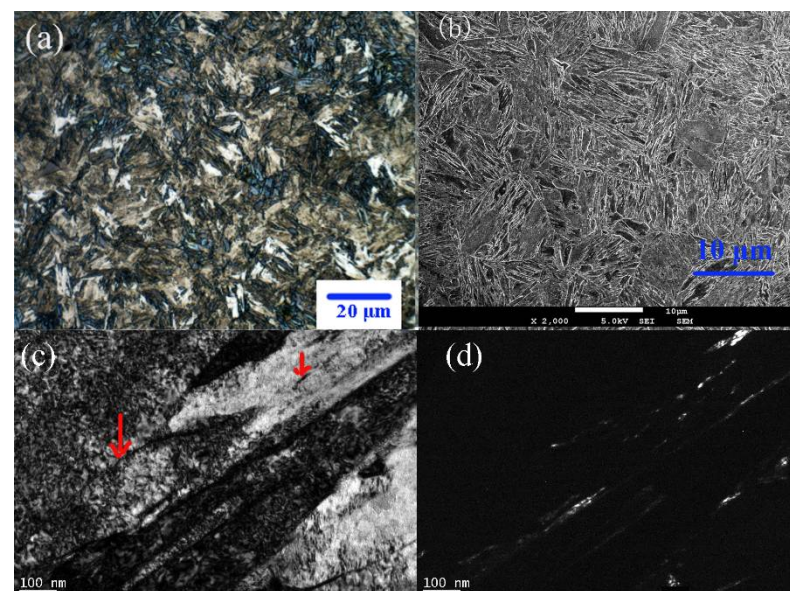


Figure 2. Optical micrograph (a), secondary electron micrograph (b), and transmission electron micrograph (c,d) of the water-quenched steel after austenization.

Figure 3 shows SEM secondary electron micrographs of the sample quenched to 220 $^{\circ}\text{C}$ and partitioned/tempered at 400 $^{\circ}\text{C}$ for 12 s, 90 s, and 600 s. For short time partitioning treatment, the general microstructural feature remains characteristic of the lath martensite in the as-quenched sample. As the partitioning and tempering time increases, the morphology of the treated samples changes from the typical lath microstructure to irregular appearance, which indicates the generation of the competing reactions [10,29]. The fresh lath martensite generated during the initial quenching to 220 $^{\circ}\text{C}$ underwent a 400 $^{\circ}\text{C}$ tempering process; thus, some carbides precipitated inside the tempered martensite lath are clearly visible when the tempering time is over 90 s (Figure 3d). Furthermore, the bainite transformation and the carbide formation can take place while the sample is subjected to Q-P-T treatment, especially during the long isothermal treatment [15,21]. Although there is no sign of bainite phase in the samples partitioned until 90 s, some bainites were found in the steels quenching and partitioned for 600 s (as indicated by the blue arrows in Figure 3f). The sample partition-tempered for 600 s displays a less “lath-like” appearance than the others, with short partitioning and tempering time. As shown by the red arrows in Figure 3f, the particles with irregular shapes appearing after isothermal holding for 600 s are identified as M/A islands, which can be attributed to the regions enriched in alloying elements of

the as-received samples (Figure 1). The origin of similar particle morphology between the as-received sample and the quenching-partitioning-tempering treated sample is rooted in the chemical heterogeneity of the austenite grains, although the samples were heated above A_3 temperature for a few minutes [3]. The final morphology of the Q-P-T treated sample being different from the original microstructure could have a significant influence on the mechanical properties of the Q-P-T steels [30,31].

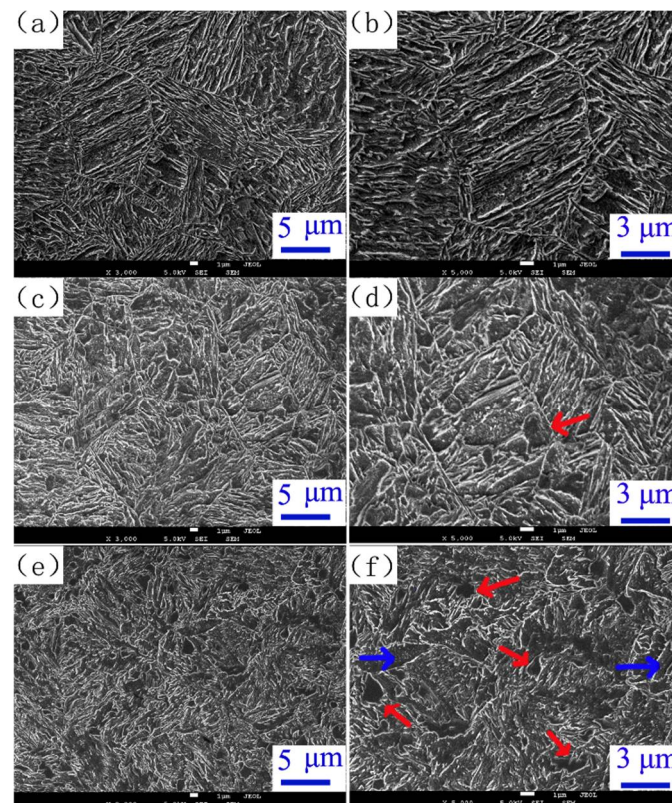


Figure 3. Secondary electron micrographs of the samples quenched to 220 °C partitioned at 400 °C for (a,b) 12 s, (c,d) 90 s, and (e,f) 600 s.

Compared with the sample quenched to 220 °C and partitioned-tempered at 400 °C for different times, the microstructures of samples quenched to 280 °C are similar, but the M/A islands appear earlier when the sample was partitioned-tempered for only 12 s (Figure 4a,b). The higher temperature favors the diffusion of carbon and alloying elements, thus accelerating the formation of M/A islands in Q-P-T steels. For longer partitioning treatment, more granular particles are present. The number of the M/A islands with irregular shapes is far greater than that of the samples quenched to 220 °C for 600 s (Figures 3f and 4f).

The film-like retained austenites of the lath martensite and the blocky retained austenite, which is typically found along the prior austenite grain boundary, are two common forms of retained austenite with different shapes. The EBSD only can qualitatively reveal the existence of the blocky retained austenite, because the EBSD technique is limited by a step size 0.1 mm larger than nanometer size of the film-like retained austenite, as shown in Figure 5c. After being quenched from the austenitizing temperature, the center of the austenite grain with a lower alloy content may firstly transform to the lath martensite phase; meanwhile, a majority of the blocky retained austenite (with higher manganese content) located at the prior austenite grain boundaries remained untransformed at the same time. This blocky retained austenite phase could be enriched in carbon atoms during the partitioning/tempering treatment; thereby, some can remain stable after the final water quenching to room temperature. This is why there are certain amounts of M/A islands

when the samples are subjected to a long-term isothermal holding treatment. With such a high amount of carbon content, the retained austenite could exist stably for a long period at high temperature without decomposition into bainite when the partitioning-tempering time is shorter than 90 s [32]. Both the SEM and the EBSD results indicate that the inhomogeneous distributions of carbon and manganese in the as-received samples have great influence on the morphology of the Q-P-T treated samples.

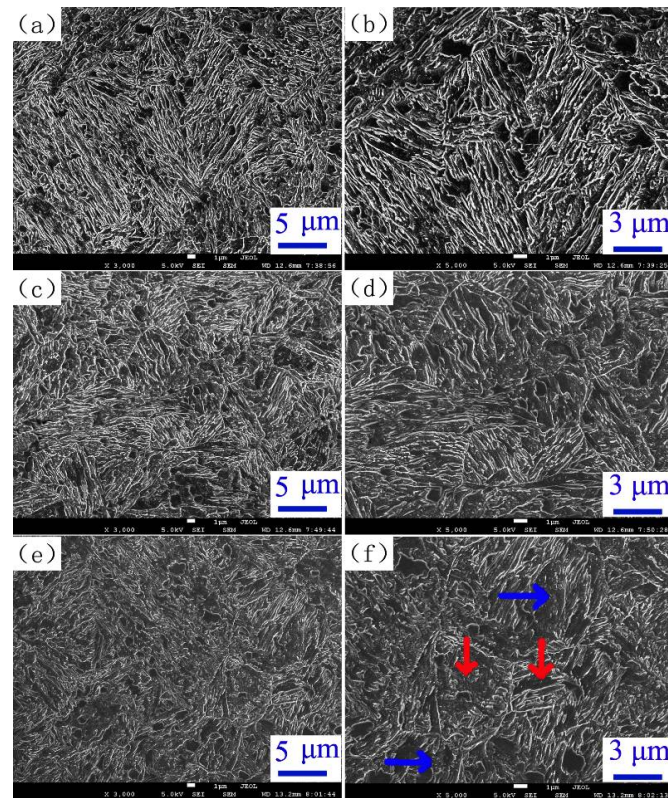


Figure 4. Secondary electron micrographs of the samples quenched to 280 °C and partitioned at 400 °C for (a,b) 12 s, (c,d) 90 s, and (e,f) 600 s.

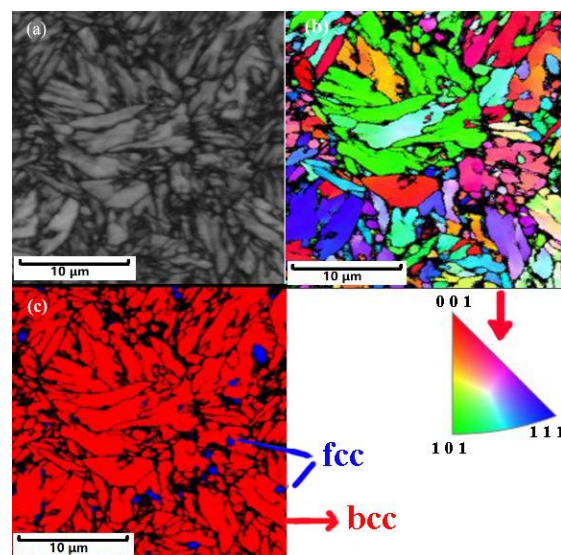


Figure 5. The electron backscattering diffraction analysis (a) overall image quality (IQ) map, (b) bcc phase inverse pole figure (IPF) map, and (c) phase map of sample quenched to 220 °C and partitioned-tempered at 400 °C for 60 s.

The effect of carbon partitioning from the initially formed martensite to austenite and the stability of the austenite phase during isothermal holding are features of the heat treatment. The TEM dark field (DF) image of the austenite in Figure 6b reveals that the width of the retained austenite is about 50 nm, which is obviously thicker than that of the water-quenched sample (Figure 2d). The corresponding selected-area electron diffraction (SEAD) embedded in Figure 6b indicates that the orientation relationship between the martensite and the retained austenite phase follows both the Kurdjumov-Sachs (K-S) relationship of $[1\ 1\ 1]_{\alpha} // [-1\ 0\ -1]_{\gamma}$, $(1\ -1\ 0)_{\alpha} // (-1\ 1\ 1)_{\gamma}$ and the Nishiyama-Wasserman (N-W) relationship: $[-1\ 0\ 0]_{\alpha} // [1\ 0\ 1]_{\gamma}$, $(0\ 1\ 1)_{\alpha} // (-1\ 1\ 1)_{\gamma}$. Besides the film-like austenite, another shape of retained austenite, namely the blocky shape, is distributed uniformly in the matrix of the steels (Figure 7), which is consistent with the EBSD results (Figure 5c).

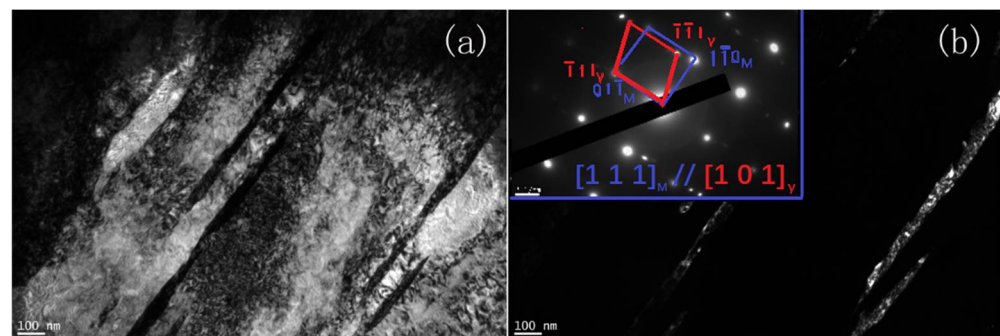


Figure 6. Bright field image (a) and dark field image (b) of the quenching-partitioning-tempering sample.

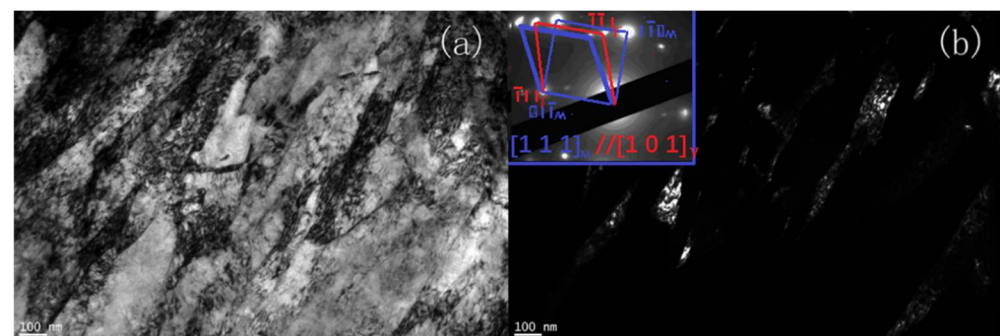


Figure 7. The transmission electron micrograph of the quenching-partitioning-tempering sample: bright field image (a) and dark field image (b).

The XRD diffraction patterns of Q-P-T steels are shown in Figure 8, and the carbon contents of austenite phases were calculated using the following empirical formula [33]:

$$a = 0.3573 + 0.00075 \text{ at.\% C (nm)} \quad (1)$$

where a is the lattice parameter of the austenite phase, which can be derived from the interplanar spacing of $(2\ 0\ 0)$ plane of austenite.

The volume fraction of the retained austenite in the samples quenched to 220 °C and 280 °C, and followed by partitioned-tempering for 12 s, 30 s, 90 s, 300 s, and 600 s, were determined using X-ray diffraction. The needed time for the carbon partitioning from martensite in Fe-0.19 C-1.59 Mn-1.63 Si steel is a few seconds, and the homogenization of carbon in the whole austenite film requires more than 10 s. In the present case, the X-ray diffraction results suggest that it may take more than half a minute for carbon diffusion and stabilizing the austenite to ambient temperature. When the sample is quenched to 220 °C and partitioned/tempered for 30 s, the largest volume fraction of retained austenite (9%) was obtained, as shown in Figure 8c. The volume fraction of retained austenite

rapidly increases with the increase in isothermal holding time until 30 s; then, it slowly decreases with the extension in isothermal holding time, regardless of the initial quenching temperature. Longer isothermal holding time leads to the decomposition of austenite and the development of both carbide and bainite, thus resulting in a decrease in austenite volume fraction [10]. With the partitioning-tempering period, austenite's carbon content initially falls and then increases, and the carbon content of the sample treated at 280 °C is a little higher than that partitioned-tempered at 220 °C (Figure 8d), indicating higher stability of the austenite after a long partitioning treatment.

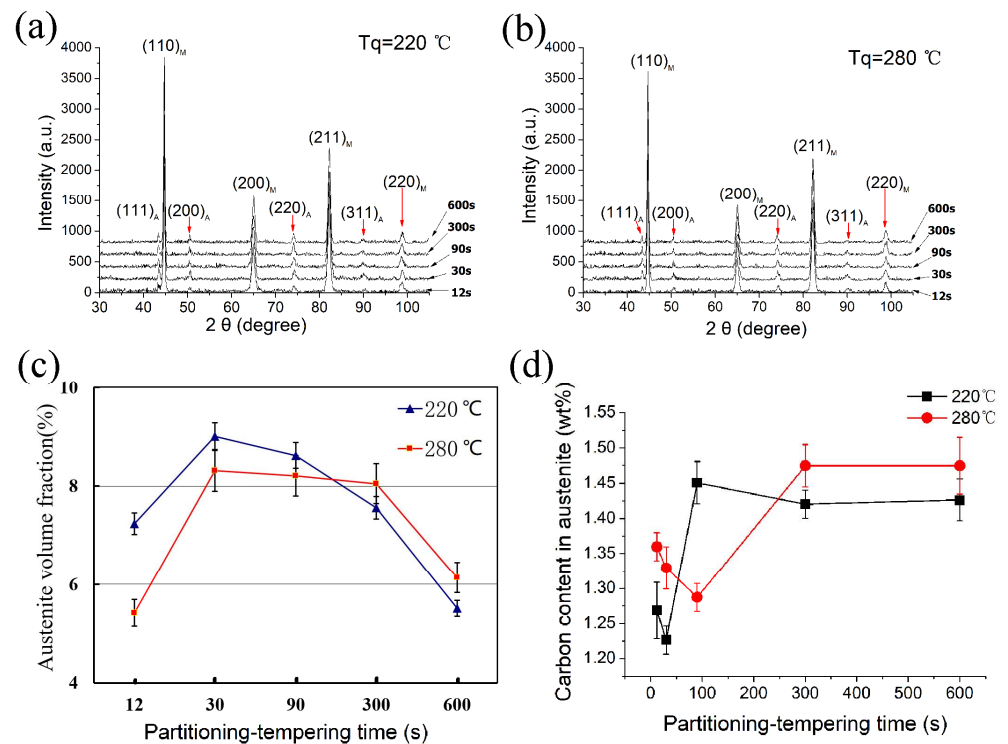


Figure 8. The XRD patterns (a,b), volume fraction of the austenite (c) and carbon content in austenite (d) in the quenching-partitioning-tempering specimens as functions of the quenching temperatures and partitioning-tempering time.

The microstructural evolution of the studied steel is expressed in Figure 9. When the as-received samples were austenitized at 910 °C, the austenite grains with an inhomogeneous distribution of alloying elements (e.g., Mn and Cr) were formed. After the first quenching to T_q , the austenite transformed into martensite, and the austenite regions with high content of alloying elements remained, but they transformed to martensite during the second quenching to room temperature (denoted as “secondary martensite”). When the partitioning tempering time was longer, the effect of diffusion of carbon atoms into the untransformed austenite region became more and more significant, and the untransformed regions with high carbon content demonstrated higher stability and transformed into M/A islands during the second quenching. However, during the long partitioning-tempering time (600 s), austenite decomposed into bainite and carbides, resulting in a lower austenite fraction [12]. In addition, the cooling rate affected the final microstructure [34], such as the stability and volume fraction of retained austenite in the Q-P-T steel; therefore, the cooling steps should be carried out as quickly as possible to achieve the desired microstructure.

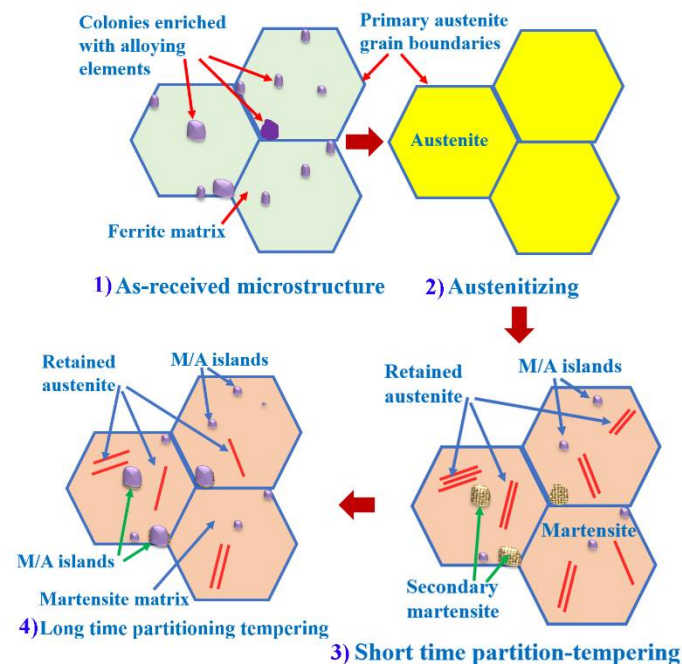


Figure 9. The schematic diagram illustrates the microstructural development of Q-P-T steel.

3.2. Mechanical Properties of the Quenching-Partitioning-Tempering Steels

When quenching at various temperatures with varying the partitioning-tempering period, the changes in final tensile strength and total elongation show a similar trend. The total elongation of samples quenched to 220 °C or 280 °C increased noticeably as the partitioning-tempering time increased from 0 to 30 s. When the sample was partitioned-tempered at 400 °C for 30 s, the total elongation could reach about 14%, which is the highest value (Figure 10). The total elongation decreased gradually with the extension of the partitioning-tempering time. The maximum value for the ultimate tensile strength of the quenching-partitioning-tempering treated steel was about 1400 MPa, and thus exhibited the highest product (19.6 GPa%) of strength and elongation (PSE); then, the ultimate tensile strength decreased slowly with the increase in partitioning-tempering time due to the following reasons: firstly, the martensite matrix gradually becomes softer with the depletion of carbon atoms during the partitioning-tempering process; secondly, decomposition of a certain amount of retained austenite occurs, accompanying the formation of carbides and bainite (indicated by blue arrows in Figures 3 and 4); thirdly, the density of M/A islands increases with the partitioning-tempering time, which could result in the deterioration of the mechanical behavior of Q-P-T steels accompanied by the decomposition of austenite in the steel matrix. In addition, the large blocky austenite that forms at low quenching temperatures is not as stable as the one that forms at high quenching temperatures due to the lower carbon content during the partitioning-tempering process; therefore, there may be a certain amount of blocky austenite transforming to martensite (TRIP effect) during the deformation.

The volume fraction of retained austenite and the total elongation change consistently, and the ultimate tensile strength also displays a similar phenomenon to the volume fraction of retained austenite. The Q-P-T process makes use of the advantage of the quenching partitioning process to increase the amount of retained austenite as much as possible at room temperature [19]. Besides the increase in ductility of Q-P-T steels that results from the softening of the martensite matrix by the depleting of carbon atoms, the quenching-partitioning-tempering treated steels have a considerable amount of retained austenite compared with the traditional quenching and tempering steels. The retained austenite can enhance the ductility of the steels as follows: (i) The retained austenite can effectively block microcrack propagation (BCP). (ii) The volume fraction of austenite in the Q-P-T

steels is estimated to be between 5% and 10% by X-ray analysis, and the fraction of blocky austenite is estimated to be about 5% by EBSD mapping. The retained austenite with blocky morphology can increase the tensile ductility through the TRIP effect during the tensile test, leading to the redistribution of stress and thus reducing the stress concentration [35,36]. (iii) The effect of dislocations absorbed by retained austenite (DARA) can result in the hard phase martensite maintaining a “softening” state during the whole deformation, thus intensifying the deformation ability of the martensite phase [12,37,38]. However, the relatively low volume fraction of austenite with a carbon content greater than 1.3 wt.% in the current Q-P-T steel is thought to have a less significant TRIP and DARA effect on ductility and elongation with respect to that of high carbon steel containing an austenite fraction of larger than 20% during deformation [23,38].

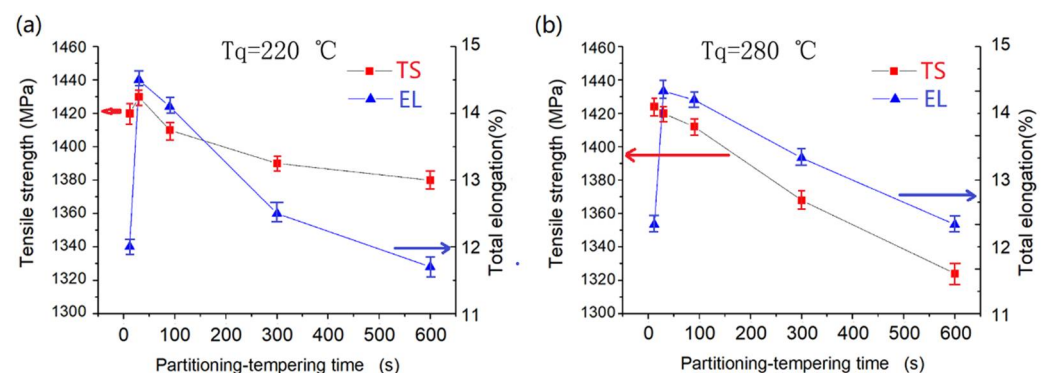


Figure 10. The evolution of ultimate tensile strength (UTS) and total elongation (TEL) of samples as functions of quenching-partitioning time for (a) quenching temperature of 220 °C and (b) quenching temperature of 280 °C.

The longer partitioning-tempering time leads to a decrease in the volume fraction of austenite. Therefore, the appearance of the M/A islands could be regarded as one kind of competing reaction during the isothermal holding process in the present work. So, the partitioning-tempering time should be restrained (less than 90 s) for attaining a greater amount of retained austenite after the partitioning-tempering process. The mechanism of microstructure inheritance should be studied further for fine tuning the microstructure and the mechanical properties of the quenching-partitioning-tempering steels.

4. Conclusions

The microstructural evolution during the quenching-partitioning-tempering treatment related to the original structure of the as-received steel was investigated, and the results are as follows.

1. The results of SEM, TEM, and XRD demonstrate that the quenching-partitioning-tempering steels mainly consist of the tempered martensite, untempered martensite, and two types of retained austenite and M/A islands inherited from the as-received steel. There is a small amount of bainite in the steels partitioned-tempered for more than 90 s.

2. The microstructural and compositional inhomogeneity of the as-received steel can have a great effect on the final microstructure of the Q-P-T steels. The number density of the M/A islands increases with the extension of the partitioning-tempering time, while the austenite fractions of Q-P-T steels first increase and then decline with the partitioning-tempering time. The high plasticity of current low carbon Q-P-T steels comes primarily from the softening of the martensite matrix, and both the BCP effect and TRIP effect may have an impact on the ductility of the steels, and the mechanism of the microstructure-properties relationship should be investigated further. Because of the optimized configuration of their multiphase microstructure, the low carbon steels partitioned-tempered for 30 s exhibit the optimal mechanical properties, with the highest amount of retained austenite (10%). The present study suggests that the partitioning-tempering process should be strictly controlled

within a certain time range for the steels with compositional inhomogeneity for obtaining good comprehensive mechanical properties.

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