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Abstract

Low-carbon alloy steel specimens were prepared by utilizing the combined effects of hot deformation and a two-step quenching and partitioning (Q&P) process. Different hot deformation temperatures were designed to investigate the effects of stress and plastic deformation induced by loading (to achieve 30% deformation) on the martensite transformation start temperature (Ms), retained austenite content, and mechanical properties under the Q&P process. The results show that, compared with the conventional two-step Q&P process, the microstructure is refined under the combined effects, particularly becoming more pronounced as the deformation temperature decreases, with martensite laths exhibiting a curved morphology. As the deformation temperature increases, Ms rises, yet the amount of martensite transformation decreases. This is because stress-induced dislocations predominantly appear at the grain boundaries of the austenite parent phase, serving as preferential nucleation sites for martensite transformation; however, once transformation occurs, a certain plastic strain enhances the stability of intragranular austenite, thereby promoting an increase in retained austenite content. The mechanical properties of specimens under the combined effects are also improved, with the highest hardness achieved at a deformation temperature of 650 °C, and the best ductility at 750 °C.

Full Text

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Effect of the Combined Action of Hot Deformation and Quenching-Partitioning Treatment on the Martensitic Transformation Mechanism of Low-Carbon Alloy Steel*

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Abstract Low-carbon alloy-steel specimens were prepared using the combined action of hot deformation and a two-step quenching and partitioning (Q&P) process. Different hot-deformation temperatures were designed, and the effects of the stress and plastic deformation induced by loading (to obtain 30% deformation) on the martensitic transformation start temperature ((M_s)), the retained-austenite content, and the mechanical properties under the Q&P process were investigated. The results show that, compared with the conventional two-step Q&P process, the combined action significantly refines the microstructure; in particular, refinement becomes more pronounced as the deformation temperature decreases, and the martensite laths exhibit a bent morphology. As the deformation temperature increases, (M_s) rises, whereas the amount of martensitic transformation decreases. This is because stress-induced dislocations mostly appear at the austenite grain boundaries, which become preferred nucleation sites for martensitic transformation; once transformation occurs, a certain amount of plastic strain increases the stability of the surrounding austenite, thereby promoting an increase in the retained-austenite content. The mechanical properties of the specimens are also improved under the combined action: the hardness of the specimen deformed at 650 ℃ is the highest, whereas the specimen deformed at 750 ℃ exhibits the best plasticity.

Keywords hot deformation; quenching and partitioning; martensitic transformation; stress

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THE COMBINED EFFECT OF HOT DEFORMATION PLUS QUENCHING AND PARTITIONING TREATMENT ON MARTENSITE TRANSFORMATION OF LOW CARBON ALLOYED STEEL

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ABSTRACT A combined process of hot deformation with different deformation temperatures plus two-step quenching and partitioning (Q&P) treatment was applied to low carbon alloyed steel. The effect of stress

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(30% plastic deformation) on the start temperature of martensite transformation (M_s), volume fraction of retained austenite and mechanical properties was analyzed. It found that comparing with specimen treated by conventional two-step Q&P process, the microstructure of steel treated by combined process was

finer and finer with the decreasing hot deformation temperature, and the typical curved micromorphology of martensite exists. Moreover, the M_s of specimen treated by combined process is increased with the increasing of deformation temperature. The effect of stress on the M_s can be attributed to the effect of stress on the grain boundaries of austenitic parent phase, where a large amount of dislocation induced by the stress is prior to occur so as to promote formation of martensite. However, the stability of untransformed austenite was improved by the plastic deformation when matensite transformed so as to get the more retained austenite (the highest volume fraction of retained austenite obtained by combined process of hot deformation at 750 ℃ is 17.2%). Moreover, the mechanical properties were improved by the combined process, namely, the highest hardness of specimen were obtained when hot deformation at 650 ℃ and the highest plasticity were obtained when hot deformed at 750 ℃.

KEY WORDS hot deformation, quenching and partitioning, martensite transformation, stress

High-strength quenched martensitic steels are rarely used directly in the as-quenched condition without tempering[^1,^2]. The conventional quenching and tempering (Q-T) process is a commonly used strengthening-and-toughening method. Tempering can improve the plasticity and toughness of martensitic steel, but it also causes a substantial reduction in strength and hardness[^3,^4]. In 2003, Speer et al.[^5] first proposed the quenching and partitioning (Q&P) process for martensitic steels; that is, the steel is first quenched to an isothermal quenching temperature (quenching temperature, QT, $M_s > \mathrm{QT} > M_f$, where M_s is the martensite transformation start temperature and M_f is the martensite transformation finish temperature), so that a portion of martensitic microstructure is obtained. The partitioning of C from the preformed martensite to the untransformed austenite is then controlled, and finally the steel is quenched to room temperature, yielding a multiphase microstructure mainly composed of martensite and carbon-enriched retained austenite[^6-^{15}].

In 2009, Speer and Matlock[^{16}] proposed the concept of directly performing Q&P treatment after hot rolling to produce high-performance hot-rolled sheets. Chandra et al.[^{17}], using 0.2C–2.0Mn–1.5Si–0.6Cr steel as the research material, performed quenching and partitioning after hot deformation. Their study found that the yield strength of the steel exceeded 1100 MPa and that, compared with samples treated by conventional heat treatment, it exhibited better elongation and impact toughness. Similar results were also obtained in Refs. [18] and [19]. It can be seen that quenching and partitioning after hot deformation has already been well verified as a means of improving steel-sheet performance. Because Q&P-treated steel consists of primary quench martensite (formed during the first quench), secondary quench martensite (formed during the second quench), and retained austenite, the martensite content during processing plays a very important role in the evolution of the steel microstructure and its mechanical properties.

In 1959, Koistinen and Marburger[^{20}] proposed the relationship between

martensite content and M_s under variable-temperature conditions:

$$\xi = 1 - \exp[-\theta(M_s - T)] \tag{1}$$

where ξ represents the amount of martensitic transformation, θ is a material constant reflecting the martensitic transformation rate, and T denotes a temperature below M_s. At this point, the martensitic transformation considers only the effect of the temperature field on the amount transformed.

In 1973, Guimaraes and Shyne[^{21}] simplified Eq. (1) as follows:

$$\xi = 1 - \exp[-0.011(M_s - T)] \quad (-80\,^{\circ}\mathrm{C} < T < M_s) \tag{2}$$

In the equation, θ in Eq. (1) can be regarded as the constant 0.011.

Equations (1) and (2) are very widely used in major mainstream stamping simulation software packages. For example, LS-DYNA still uses Eq. (1) to calculate the martensite content in geometrical structural components such as automobile B-pillars; to a certain extent, this can reflect the characteristics of martensitic transformation. However, the application of the above equations also has major limitations. On the one hand, M_s is set as a constant, so the martensite content depends only on temperature changes, while the influence of stress on M_s is ignored. On the other hand, when quantifying martensite content in the software, the influence of stress on the martensitic transformation rate is not considered. These factors cause the simulation accuracy to be unable to truly reflect the transformation process. In the present work, CrNi3Si2MoV steel is used as the research material, and hot deformation + Q&P treatment is carried out to study the effects of the deformation-transformation coupling on the martensitic transformation mechanism, microstructure, and mechanical properties, and to clarify the transformation behavior characteristics of low-carbon alloy steel treated by Q&P under conditions in which stress caused by loading is present.

1 Experimental method

The chemical composition (mass fraction, %) of CrNi3Si2MoV steel is: C 0.21, Si 1.75, Mn 0.29, Cr 1.03, Ni 2.86, Mo 0.31, V 0.08, Nb 0.049, S 0.0007, P 0.006, and Fe balance. The dilatometric method was used to measure M_s as 375 ℃ and M_f as 175 ℃. The specimens were cylindrical, with dimensions of 10 mm in diameter × 80 mm in length. Two processing routes were designed using a Gleeble 3800 thermal simulation testing machine, as shown in Fig. 1. Figure 1a shows the coupled process of hot deformation + Q&P treatment: the specimen was first heated to 900 ℃ and held for 5 min, then cooled separately to 850, 750, and 650 ℃ and held for 10 s; it was then compressed by 30% at a strain rate of 1 s^{-1}, cooled to 330 ℃ and held for 1 min, reheated to 500 ℃ and held for 1 min, and finally quenched to room temperature. Figure 1b shows the conventional

Fig. 1 Schematic of hot deformation and Q&P treatment process

Figure 1: Fig. 1 Schematic of hot deformation and Q&P treatment process

Fig. 2 Dimension of the specimen in uniaxial tension test

Figure 2: Fig. 2 Dimension of the specimen in uniaxial tension test

two-step Q&P treatment process. The heating and cooling rates used in the experiments were 10 ℃/s.

A PHILIPS APD-10 X-ray diffractometer (XRD,

Fig. 1 Schematic of hot deformation and Q&P treatment process (Q&P—quenching and partitioning)
(a) combined process
(b) conventional two-step Q&P process

The residual austenite fraction was measured by CoKα. The deformed portion of the hot-compressed specimen was cut longitudinally with a wire-cutting machine. After mechanical grinding and polishing, it was etched with 2% nitric acid alcohol (volume fraction), and the microstructure was observed using an S-4300 field-emission scanning electron microscope (FE-SEM) and a JEM-2010 transmission electron microscope (TEM). The specimen was machined into a small cylinder 3.5 mm in length and 7.5 mm in diameter. The martensitic transformation during quenching after austenitization was observed in situ using a VL2000DX-SVF17SP&15FTC high-temperature laser scanning confocal microscope (HTL-CM). In order to clearly observe the austenite grain boundaries and the martensitic transformation process, the specimen was first heated to 1300 °C, held for 1 min, and then cooled to room temperature at a rate of 400 °C/min, during which the martensitic transformation was observed. The hardness of the specimens was measured using a VH-5 Vickers hardness tester. The samples treated on the Gleeble thermal simulator were machined into the non-standard specimens shown in Fig. 2 to evaluate uniaxial tensile properties.

Fig. 2 Dimension of the specimen in uniaxial tension test
(unit: mm)

2 Experimental Results and Discussion

2.1 Observation by high-temperature laser confocal microscopy of the preferred nucleation sites and formation process of martensite during cooling

HTLCM was used for in situ observation of the martensitic transformation process during cooling of austenite, and the results are shown in Fig. 3. It can be seen that, as the temperature decreases: (1) martensite preferentially nucleates at austenite grain boundaries and grows into the grains, as indicated by arrow

1 in Fig. 3a, passing through the entire grain; it also promotes martensitic transformation in adjacent grains, as indicated by arrow 2 in Fig. 3b; (2) after martensite nucleates, it grows into the austenite interior at a certain angle (60° or 120°) to the martensite that formed first, as indicated by arrow 3 in Fig. 3b; (3) martensite nucleates at corners, as indicated by arrow 4 in Fig. 3b. Martensitic transformation is a displacive transformation, and the increase in the martensite amount does not depend on an increase in the length and thickness of martensite laths, but rather on the continuous batch formation of new martensite laths as the temperature decreases. There are mainly three mechanisms by which newly formed martensite laths relate to previously formed martensite laths: (1) adjacent and parallel, as indicated by arrow 5 in Fig. 3c; (2) non-adjacent but parallel, as indicated by arrow 6 in Fig. 3d; (3) approximately parallel, as indicated by arrow 7 in Fig. 3d. It can also be found from Fig. 3 that, during quenching, the formation of martensite is not uniformly distributed; instead, it increases in batches in certain regions, continuously dividing the untransformed austenite into several regions of different sizes and showing microstructural inhomogeneity. Studies indicate that this microstructural inhomogeneity plays a positive role in obtaining more retained austenite in steels treated by the Q&P process.

2.2 Effect of hot deformation before transformation on the (M_s), microstructure, and mechanical properties of Q&P steel

2.2.1 Microstructure Figure 4 shows SEM images of the microstructures of austenitized specimens after hot deformation at 650 and 750 °C followed by the Q&P process, as well as after the conventional two-step Q&P process. It can be seen that, after different processing routes, in addition to lath martensite, each specimen also contains blocky structures with sizes of 1–3 μm. These structures are difficult to etch. Studies [22] have shown that the etched structures are the primary quenched martensite formed during the Q&P process (M1, Fig. 4a); because tempering occurs during partitioning, they are relatively easy to etch. The blocky retained structures are secondary quenched martensite (M2, Fig. 4a), formed during the second quenching process; because they are in the as-quenched state, they are not easily etched. IPWin60C software was used to statistically analyze the average size of secondary quenched martensite in the microstructure. The results show that, compared with 0.179 μm (Fig. 4c) in the specimen treated by the conventional Q&P process, the size is refined to 0.157 μm (Fig. ···

Fig. 3 In-situ observation of the preferential nucleation sites of martensite and the growth process of transformed nuclei during cooling

Fig. 3 HTLCM images show the formation and growth of martensite during the quenching treatment at 362.9 °C (a), 358.5 °C (b), 356.6 °C (c), and 350.7 °C (d). Arrow 1 indicates martensite nucleates at the grain boundary; arrow 2 indicates martensite transformation promoted in the adjacent grain; arrow 3 indicates newly formed martensite; arrow 4 indicates martensite nucleates at the

corner of an austenite grain; arrow 5 indicates adjacent and parallel martensite; arrow 6 indicates non-adjacent but parallel martensite; and arrow 7 indicates approximately parallel martensite.

Fig. 4 SEM images of the microstructures after hot-deformation + Q&P treatment and conventional Q&P treatment

Fig. 4 SEM images of samples treated by Q&P process after hot deformation at 650 °C (a), 750 °C (b), and conventional Q&P process (c). M1—initial martensite; M2—fresh martensite.

4a) and 0.169 μm (Fig. 4b), indicating that the size was significantly refined.

The TEM images of the samples after hot-deformation + Q&P treatment at different temperatures and after conventional Q&P treatment are shown in Fig. 5. It can be seen that, under different experimental conditions, the microstructure of the steel is mainly composed of refined lath martensite (M), film-like retained austenite (γ), and fine carbides. During the subsequent Q&P treatment process, the deformed supercooled austenite matrix underwent martensitic transformation, but still retained the characteristics of the deformed microstructure. For example, the martensitic lath morphology with curved features shown in the boxed regions of Fig. 5a and b becomes more pronounced as the deformation temperature decreases; this phenomenon is absent in the sample treated by the conventional Q&P process. The retained austenite in samples treated by different processes exists mainly in the form of thin films, with a width less than 100 nm.

Fig. 5 TEM images of lath martensite and retained austenite after hot deformation at different temperatures + Q&P treatment and after conventional Q&P treatment

Fig. 5 Bright-field (a–c, e) and dark-field (d, f) TEM images of samples treated by the Q&P process after hot deformation at 650 °C (a), 750 °C (b), 850 °C (c, d), and by conventional Q&P (e, f)

2.2.2 Contents of M_s and retained austenite

Regarding the influence of stress caused by loading on martensitic transformation, Olson and Cohen[^{23}] and Xu Zuyao[^{24}] successively proposed that stress can induce or suppress non-diffusional transformations in alloys. In fact, the effect of stress on M_s can be attributed to the effect of stress on the austenite parent-phase grain boundaries[^{17}]. Figure 3a shows that martensite preferentially nucleates mainly at the austenite parent-phase grain boundaries. Therefore, when the amount of deformation caused by stress is not very large, the large number of dislocations concentrated in the austenite parent phase as a result of the stress will be the main source inducing phase transformation and increasing M_s. Under the combined effect of hot deformation at different temperatures (30% deformation) + Q&P process, the variation curves of the radial volume of the specimens with temperature are shown in Fig. 6. The M_s of this

steel measured using a dilatometer is 375 °C, whereas Fig. 6 shows that, when stress (30% deformation) is present, M_s increases; moreover, with increasing hot-deformation temperature, the M_s of the specimens increases. Specifically, the M_s values at hot-deformation temperatures of 650, 750, and 850 °C are 395, 410, and 421 °C, respectively. It can be seen that, under the present experimental conditions, hot deformation can promote martensitic transformation, and there is a certain relationship between stress and M_s. In addition, with ···

As the hot-deformation temperature increases, the expansion of the specimen decreases; that is, the tendency toward explosive transformation is correspondingly reduced. The reason is that, although hot deformation promotes an increase in (M_s), at higher deformation temperatures partial recrystallization occurs. The recrystallized grains are distributed at the prior-austenite grain boundaries and consume the dislocations generated by hot deformation, thereby reducing the number of sites at grain boundaries where martensite preferentially nucleates.

XRD results show that the retained-austenite content of the specimen treated by the conventional Q&P process is 14.0%. In comparison, the hot-deformation + Q&P process produced more retained austenite: the 650, 750 and 850 °C deformation + Q&P treatments yielded retained-austenite contents of 14.3%, 17.2% and 16.4%, respectively. Thus, after hot-deformation + Q&P processing, the specimens contain more retained austenite in their microstructures. From Fig. 6, although after deformation at 750 and 850 °C the (M_s) values are higher than that after deformation at 650 °C—that is, martensitic transformation occurs earlier under the action of stress—the larger retained-austenite content reduces the martensitic transformation conversion fraction.

Liu Chuncheng et al. ([25]) proposed that the stress induced by loading before transformation changes the morphology of smooth and flat intergranular boundaries into irregular wavy boundaries, thereby locally producing small regions with relatively high grain-boundary energy at the grain boundaries. These regions serve as preferential nucleation sites for martensitic transformation and thus influence (M_s), which is consistent with the experimental phenomena in this work. In addition, once plastic deformation occurs, grain-boundary nucleation sites are gradually consumed. In other words, the sites favorable for nucleation are quickly consumed. The corresponding plastic strain can increase the yield strength of the austenite parent phase within the grains, while martensitic transformation is a shear-type transformation. An increase in the parent-phase strength hinders the shear process, which is equivalent to increasing the resistance to transformation; conversely, it reduces the martensitic transformation rate and transformation conversion fraction, making the parent phase more stable, i.e., mechanical stabilization occurs. Therefore, although the combined action of 750 and 850 °C hot deformation + Q&P processing increases (M_s), the plastic strain before transformation also significantly improves the stability of the austenite parent phase, leading to an increase in retained-austenite content.

Fig. 6 Effect of deformation temperature on start temperature of martensite transformation (M_s)

Figure 3: Fig. 6 Effect of deformation temperature on start temperature of martensite transformation (M_s)

Fig. 7 Uniaxial tensile curves of specimens obtained by different processes

Figure 4: Fig. 7 Uniaxial tensile curves of specimens obtained by different processes

2.2.3 Mechanical properties

The test results show that the hardnesses of the specimens under different conditions are 395 HV (Q&P), 426 HV (650 °C + Q&P), 412 HV (750 °C + Q&P) and 405 HV (850 °C + Q&P), respectively. Compared with the conventional Q&P process, the specimens treated by hot-deformation + Q&P have higher hardness. As the deformation temperature decreases, the hardness of the hot-deformation + Q&P specimens gradually increases. The uniaxial tensile curves characterized using nonstandard specimens are shown in Fig. 7. It can be seen that, compared with the conventional Q&P process, the hot-deformation + Q&P specimens not only show improved strength but also a markedly increased elongation. The large number of dislocations generated by deformation are inherited by martensite during quenching, and the microstructural refinement caused by deformation is an important reason for the increase in strength ([26]). The hot-deformation + Q&P specimens contain more retained austenite than the conventional Q&P specimens. Studies in Ref. [22] have shown that the plasticity of Q&P-processed steel increases linearly with increasing retained-austenite content. The transformation-induced plasticity (TRIP) effect of retained austenite is the main reason for the improvement in steel plasticity. In particular, the specimen treated by 750 °C deformation + Q&P has the highest retained-austenite content, 17.2%, and also the highest plasticity level.

Fig.6 Effect of deformation temperature on start temperature of martensite transformation (M_s)

Fig.7 Uniaxial tensile curves of specimens obtained by different processes

3 Conclusions

(1) Compared with specimens prepared by the conventional two-step Q&P process, under the combined action of hot deformation (30% deformation) + Q&P processing, the microstructure of the specimens is refined, including refined martensite laths and film-like retained austenite. The refinement of the microstructure becomes more pronounced as the deformation temperature decreases. In addition, the martensite laths exhibit typical curved morphological characteristics.

(2) Under the combined treatment, increasing the hot-deformation temperature promotes an increase in the martensitic transformation start temperature (M_s). Dislocations induced by stress appear at the austenite parent-phase grain boundaries and become preferential nucleation sites for martensitic transformation. However, once transformation begins, a certain amount of plastic strain improves the stability of the intragranular austenite, promoting an increase in retained-austenite content and reducing the martensite content. For example, under 750 °C hot deformation, the retained-austenite content can reach 17.2%.

(3) Compared with the conventional Q&P process, the combined thermomechanical deformation + Q&P process can serve as a method for preparing steel materials with higher strength and higher ductility.

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Figure 7

Figure 5: Figure 7

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Figures