Effect of One-Step Q&P Process on Microstructure and Mechanical Properties of Dual Martensite Steel (Postprint)

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Abstract

The microstructure and mechanical properties of dual-martensite experimental steel processed by a one-step quenching and partitioning (Q&P) treatment were investigated and compared with those subjected to direct quenching and quenching and tempering (Q&T) treatments, to preliminarily elucidate the effects of the one-step Q&P process on the material's microstructural and mechanical characteristics. The results demonstrate that the microstructure consists primarily of lath martensite, plate martensite, and interlath film-like retained austenite. With prolonged partitioning time, the plate martensite content initially increases and subsequently decreases, whereas the retained austenite content progressively increases and approaches a stable value. Compared with conventional direct quenching and Q&T processes, the experimental steel subjected to the one-step Q&P treatment exhibits superior comprehensive mechanical properties, combining high strength with good ductility. The strength-ductility product, tensile strength, and elongation reach values of 21774.2 MPa • %, 1442 MPa, and 15.1%, respectively. Furthermore, with increasing partitioning time, the tensile strength gradually decreases while the elongation progressively increases and stabilizes.

Full Text

Title and Authors

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Abstract

In response to increasing demands for automotive lightweighting and safety, advanced high-strength steels (AHSS) have attracted considerable attention due to their significant potential for weight reduction and energy absorption during collisions. The quenching and partitioning (Q&P) process, proposed by Speer et al. based on conventional martensitic and TRIP steels, offers a novel approach to developing high-strength, high-ductility martensitic steels. This process involves quenching from the austenite region to a temperature between the martensite start (Ms) and finish (Mf) temperatures, followed by isothermal holding at this temperature (one-step Q&P) or slightly above it (two-step Q&P) to allow carbon partitioning from supersaturated martensite to austenite, and final quenching to room temperature to obtain a multiphase microstructure comprising martensite and carbon-enriched retained austenite. Compared with conventional martensitic and TRIP steels, the Q&P process can effectively increase the volume fraction of retained austenite, achieving both high strength and good ductility.

Since its introduction, extensive research has investigated the influence of Q&P process parameters on microstructural evolution and mechanical properties. Bagliani et al. developed a high-strength automotive steel with a product of tensile strength and elongation exceeding 20000 MPa·% using a 0.28C-1.4Si-0.67Mn-1.49Cr-0.56Mo steel. With advances in characterization techniques, researchers have refined microstructural analysis of Q&P steels. Wang et al. distinguished primary quenched martensite (M1) from secondary quenched martensite (M2) based on formation timing and corrosion resistance, noting that M1 undergoes tempering during partitioning and is more susceptible to corrosion than fresh M2. Santofimia et al. used electron backscatter diffraction (EBSD) to characterize M1 and M2 in medium-high carbon Q&P steels, revealing significant differences in crystal structure and morphology, with M1 being larger in size and exhibiting less lattice distortion. However, few studies have focused on plate martensite that may form during secondary quenching. Plate martensite possesses high strength but extremely poor ductility, significantly affecting the overall mechanical properties of Q&P steels. Therefore, investigating plate martensite formation is crucial.

Most research has concentrated on two-step Q&P processes, which are complex and unsuitable for large-scale industrial production. This study focuses on the one-step Q&P process, analyzing microstructural evolution through crystallographic characterization of transformation products and comparing the results with direct quenching and quenching & tempering (Q&T) processes to develop a more energy-efficient, practical Q&P approach.

Experimental Procedures

Materials and Heat Treatment

The experimental steel had a chemical composition (wt.%) of: C 0.19, Si 1.55, Mn 1.53, Ni 0.95, Cr 1.01, Mo 0.45, Cu 1.01, Ti 0.033, B 0.0027, Al 0.025, Fe balance. The steel was melted in a 150 kg vacuum furnace under Ar atmosphere to prevent oxidation. The ingot was heated to 1200 °C for 2 h for homogenization, then hot-rolled in seven passes to a final thickness of ~5 mm with a finishing temperature of (950$±$10) °C, followed by air cooling to room temperature.

Critical transformation temperatures were measured using a Formaster-FII dilatometer. Standard samples (3 mm diameter × 10 mm length) were prepared. The measured Ac1, Ac3, Ms, and Mf temperatures were 703 °C, 880 °C, 362 °C, and 152 °C, respectively. To ensure complete austenitization without excessive grain growth, an austenitization temperature of 950 °C was selected. Heat treatment parameters for all samples are summarized in .

Microstructural Characterization

Heat-treated samples were prepared for optical microscopy (OM), transmission electron microscopy (TEM), and X-ray diffraction (XRD). OM samples were mechanically ground, polished, and etched with 4% (vol.) nital solution, then examined using an OLYMPUS GX71 inverted microscope. Phase volume fractions were analyzed using Image-Pro Plus software.

TEM samples were mechanically thinned to 0.05 mm, then twin-jet electropolished in a solution of 9% (vol.) HClO4 and C2H5OH. Microstructures were observed using a Tecnai G2 F20 TEM.

XRD samples were ground with sandpaper and electropolished to eliminate residual stress from mechanical grinding, which could affect retained austenite measurements. The electropolishing solution had a volume ratio of C2H5OH:HClO4:H2O = 13:2:1, with a voltage of 20 V for 25 s. Retained austenite volume fraction was measured using a D/max 2400 XRD system with Cu Kα radiation at 56 kV and 182 mA, with a step size of 0.02°. The martensite (200)α and (211)α peaks and austenite (200)γ, (220)γ, and (311)γ peaks were selected to determine diffraction angles (2) and integrated intensities (I). The retained austenite volume fraction was calculated using:

$$V_\gamma = \frac{I_\gamma}{I_\alpha G} \times 100\%$$

where V_γ is the retained austenite volume fraction, I_α is the integrated intensity of any martensite {200} or {211} diffraction peak, I_γ is the integrated intensity of any austenite {200}, {220}, or {311} diffraction peak, and G is the intensity factor ratio between specific austenite and martensite crystal planes.

Five diffraction peak combinations were measured: (200)α-(200)γ, (200)α-(220)γ, (200)α-(311)γ, (211)α-(200)γ, (211)α-(220)γ, and (211)α-(311)γ. The corresponding G values were 2.50, 1.38, 2.02, 1.19, 0.06, and 0.96, respectively. Each V_γ was calculated and averaged to obtain the final retained austenite volume fraction.

Carbon content in retained austenite was determined from the austenite lattice parameter (a_γ), which increases by 0.00044 nm for every 0.1 wt.% increase in carbon content:

$$a_\gamma = 3.555 + 0.044x_C$$

where x_C is the carbon content (wt.%) in austenite.

Mechanical Testing

Tensile specimens were machined along the rolling direction according to GB/T228-2010 standard with a gauge length of 25 mm. Tensile tests were conducted on a SANS5105 universal testing machine at a strain rate of 0.002 s^{-1} at room temperature.

Results and Discussion

2.1 Microstructure

[Figure 1: see original paper] shows OM images of samples after different heat treatments. Figures 1a-e present microstructures of one-step Q&P processed samples. The dark regions represent low-carbon lath martensite formed during initial quenching, while the white regions are high-carbon plate martensite formed after partitioning, with volume fractions of 2.6%, 4.9%, 9.4%, 5.2%, and 1.9%, respectively. With increasing partitioning time, plate martensite content first increases then decreases, reaching a maximum at 10 min (Sample No.6). Literature indicates that the minimum carbon content for plate martensite formation is 0.60 wt.%, while the minimum carbon content for retained austenite can be estimated using the Ms empirical formula:

$$Ms = 539 - 423x_{Cr} - 7.5x_C - 30.4x_{Mo} - 7.5x_{Mn} - 17.1x_{Si} + 30x_{Al}$$

where x_{Mn}, x_{Ni}, x_{Cr}, x_{Mo}, x_{Si}, and x_{Al} are the weight percentages of respective elements in austenite. Calculation yields a minimum carbon content of 1.05 wt.% for retained austenite. Thus, untransformed austenite with carbon content >1.05 wt.% remains as retained austenite, 0.60-1.05 wt.% transforms to plate martensite, and <0.60 wt.% transforms to lath martensite during subsequent quenching.

For partitioning times of 2-10 min (Figs. 1a-c), plate martensite content increases with time because the chemical potential of carbon in martensite exceeds that in untransformed austenite, driving carbon diffusion from martensite to austenite. This enriches the carbon concentration in untransformed austenite (0.60-1.05 wt.%), which transforms to plate martensite upon cooling, peaking at 10 min. Further increasing partitioning time (Figs. 1d-e) decreases plate martensite content, requiring further investigation.

[Figure 1: see original paper]f shows Sample No.2 microstructure, consisting primarily of lath martensite. After complete austenitization, the uniform carbon concentration of 0.19 wt.% prevents plate martensite formation. However, TEM analysis is needed to confirm the presence and morphology of retained austenite.

To clarify the decrease in plate martensite with extended partitioning time, Sample No.8's heat treatment was simulated using a dilatometer. The sample was heated to 950 °C at 10 °C/s, held for 3 min, cooled to 300 °C at 50 °C/s, held for 30 min, then cooled to room temperature with He gas. The dilatation curve is shown in [Figure 2: see original paper]. Region 1 represents the quenching process after austenitization, where curve elevation indicates lath martensite formation. Regions 2 and 3 show the isothermal holding process. No curve elevation appears in Region 2, but after 16 min (11 min holding time), the curve begins rising until the end of holding. Two explanations exist for dilatation increase during isothermal holding: (1) carbon partitioning continuously enriches untransformed austenite, lowering Ms below 300 °C, effectively causing bainitic transformation; (2) Ms remains above 300 °C, and the dilatation increase results from martensite nucleation.

[Figure 3: see original paper]a-c shows TEM images and selected area electron diffraction (SAED) patterns of Samples No.1-3. All microstructures consist primarily of lath martensite, with SAED patterns showing only bcc diffraction spots, indicating minimal retained austenite. Measured average lath widths are 120 nm, 140 nm, and 260 nm, respectively, demonstrating that higher cooling rates produce finer martensite laths. Increased cooling rate enhances martensite nucleation driving force and nucleation sites, activating all four possible habit planes $\{111\}\gamma$ within austenite grains. This subdivides the prior austenite grains, increasing the number of martensite packets. Since austenite grain size remains unchanged, martensite packet size decreases with increasing cooling rate.

[Figure 4: see original paper] shows TEM images and SAED patterns of Sample No.6. The microstructure contains two distinct martensite morphologies and interlath retained austenite films. The arrowed region in [Figure 4: see original paper]a shows twinned martensite corresponding to the white plate martensite regions in OM images. [Figure 4: see original paper]b shows the SAED pattern of bcc twin martensite along the [110] zone axis, confirming the twin plane as (112). [Figure 4: see original paper]c-e show bright-field, dark-field images, and SAED pattern of lath martensite and retained austenite. The orientation relationship between austenite and martensite follows the K-S relationship:

(101)α//(111)γ and [111]α//[101]γ, where (111)γ and [101]γ represent close-packed planes and directions in austenite, respectively. This satisfies the lattice matching principle, where similar atomic arrangements at a crystal plane or direction between parent and product phases minimize nucleation energy. [Figure 4: see original paper]d is the centered dark-field image of [111]α and [101]γ diffraction spots, where bright contrast regions correspond to retained austenite.

[Figure 5: see original paper] shows TEM images of Sample No.8. [Figure 5: see original paper]c is the SAED pattern of the darker lath region (arrowed) in [Figure 5: see original paper]a, showing both bcc and fcc phase diffraction patterns along [111] zone axes that also satisfy the K-S relationship. [Figure 5: see original paper]b is the centered dark-field image of (111)γ, where bright regions correspond to fcc phase at lath edges (retained austenite), while the central regions are bcc phase.

2.2 Retained Austenite Content

[Figure 6: see original paper] shows XRD patterns of different samples. Samples No.2 and No.10 show almost no retained austenite peaks, while Sample No.6 exhibits distinct retained austenite diffraction peaks. Using JADE software and equations (1) and (2), retained austenite contents in Samples No.4-8 were calculated as 5.1%, 6.4%, 8.1%, 8.2%, and 8.3%, respectively, with carbon contents of 1.14%, 1.35%, 1.38%, 1.38%, and 1.39%. One-step Q&P processed samples exhibit two key features: (1) high retained austenite content (up to 8.3 vol.%), resulting from carbon partitioning that enriches austenite and stabilizes it at room temperature; (2) both retained austenite content and its carbon content increase with partitioning time, stabilizing after 10 min.

As shown in [Figure 1: see original paper], plate martensite content peaks at 10 min partitioning time, then decreases with further holding, while retained austenite content increases only slightly after 10 min. This is attributed to phase transformation occurring after 10 min, as evidenced by the dilatation curve in [Figure 2: see original paper]. TEM analysis confirms this transformation product as bcc phase ([Figure 5: see original paper]). The competition between carbon partitioning and phase transformation during isothermal holding determines the final contents of plate martensite and retained austenite.

2.3 Mechanical Properties

Mechanical properties of different samples are listed in . Comparing Samples No.1-3 reveals that tensile strength (Rm) increases with cooling rate, while elongation shows the opposite trend, due to combined effects of quenching stress and grain refinement. Samples No.4-8 all exhibit Rm >1200 MPa and product of strength and elongation >20000 MPa·%. With increasing partitioning time, tensile strength gradually decreases while elongation increases and stabilizes. The strength decrease arises from: (1) carbon depletion in martensite due to partitioning, reducing solid solution strengthening; (2) decreased dislocation density and dislocation straightening in lath martensite, reducing dislocation and internal strain strengthening. Elongation is primarily determined by retained austenite content, showing corresponding trends. Samples No.9-11 exhibit intermediate properties between direct quenching and one-step Q&P, consistent with literature.

Sample No.5 demonstrates the optimal comprehensive properties, achieving a product of tensile strength and elongation of 21774.2 MPa·%, tensile strength of 1442 MPa, and elongation of 15.1%. This represents significant improvement over direct quenching and Q&T processes, and higher efficiency than two-step Q&P processes for similar steel compositions reported in literature.

Conclusions

(1) One-step Q&P processing yields a microstructure primarily consisting of lath martensite, plate martensite, and interlath retained austenite films. Plate martensite content first increases then decreases with partitioning time, exhibiting characteristic twinning features with twin plane (112) in TEM images. Retained austenite distributes as thin films between lath martensite, maintaining a K-S orientation relationship with martensite. Direct quenched microstructures consist mainly of lath martensite, with lath width decreasing as cooling rate increases.

(2) Compared with direct quenching and Q&T processes, one-step Q&P processed samples exhibit superior comprehensive mechanical properties, achieving a product of tensile strength and elongation of 21774.2 MPa·%, tensile strength of 1442 MPa, and elongation of 15.1%. With increasing partitioning time, tensile strength gradually decreases while elongation increases and stabilizes.

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Note: Figure translations are in progress. See original paper for figures.