Interplay between reversed austenite and plastic deformation in a directly quenched and intercritically annealed 0.04C-5Mn low-Al steel

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Abstract

We have elucidated here the interplay between reversed austenite and plastic deformation in a directly quenched and intercritically annealed medium-manganese low-Al steel in terms of microstructural evolution and mechanical properties. The ultra-low carbon 5Mn steel was subjected to controlled rolling and direct quenching, followed by intercritical annealing at 630°C and 650°C for 30 min, respectively. The directly quenched steel consisted of thin lath-martensite of ~0.2-0.9 μm thickness and ~1.7-2.0% retained austenite with ~50 nm thickness. Intercritical annealing led to a microstructure consisting of alternate sub-micron laminated structure of tempered martensite and reversed austenite. With increase in annealing temperature from 630°C to 650°C, the volume fraction of austenite was increased from 12.6% to 19.1% at 1/4 thickness ($t/4$), and from 8.4% to 12.7% at 1/2 thickness ($t/2$). The yield strength, tensile strength, and elongation of 728 MPa, 826 MPa, and 25.5%, were obtained at $t/4$ of the plate, and 714 MPa, 814 MPa, and 24.2% at $t/2$, on annealing at 630°C. The impact energy obtained at -60°C was greater than 80 J. When the annealing temperature was increased to 650°C, strength was marginally decreased, but toughness was significantly increased to more than 110 J. Extremely small variation in microstructure and mechanical properties at $t/4$ and $t/2$ plate thickness were observed. Energy dispersive x-ray spectroscopy (EDS) confirmed ~7.2-10.1 wt.% Mn enrichment in reversed austenite during annealing, which is of significance in stabilization of reversed austenite. Work hardening behavior and tensile experiments were conducted to study the effect of reversed austenite during plastic deformation. With increased annealing temperature, the stability of reversed austenite was decreased because of less enrichment of C and Mn in reversed austenite and grain growth. At 0.05 tensile strain, reversed austenite

transformed to twin-type martensite, implying occurrence of transformation induced plasticity (TRIP) effect.

Keywords: Medium-Mn steel; Homogeneity; Reversed austenite; Mn partitioning; Work hardening

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1. Introduction

Heavy plates of advanced high-strength steels are known for high ductility, good low-temperature impact toughness, ease of weldability and safety. They are widely considered for buildings, pressure vessels, bridges, ship hull, and offshore structures [1-5]. The principal application of high-strength offshore steels with nominal yield strength in the range of ~500-800 MPa is in the fabrication of jack-up legs, rack, and pinions [6]. With increased demand for energy, oil and gas exploration is moving into deeper sea areas and polar regions, such that the usage of heavy plates for offshore structures is increasing. To meet the challenging requirements, there is a need for low carbon design for reducing carbide precipitation and consequently high low-temperature toughness. Ultra-low carbon can contribute to improved weldability [5, 7]. Currently used low alloy heavy plate steels for high-strength structural applications are essentially quenched and tempered steels with low- to medium- carbon content [8]. Generally expensive alloying elements, such as Ni, Cr, Cu, and Mo, are added to obtain required high strength in the mid-thickness of the plate to improve the microstructure, mechanical properties and homogeneity. Furthermore, the traditional high-strength structural steels adopt multi-stage heat treatment process, which consumes significant energy and is against the concept of green economy.

Mn is an important alloying element in the design of advanced high-strength steels, because it increases the stability of austenite and also impacts the kinetics of transformation [9-10]. In recent years, a number of studies have been carried out on low-carbon medium-manganese steels to explore new possibilities in the development of advanced high-strength steels [11-15]. It is known that BCC bainite/martensite transforms to FCC austenite during intercritical annealing in the two-phase region, referred as austenite reverted transformation (ART) [16]. It was demonstrated that medium-Mn steel

processed by intercritical annealing and involving ART can provide excellent combination of strength and ductility [17-18]. Fine martensitic plates containing high dislocation density contributed to strength. High strain hardenability of reversed austenite provided ductility and toughness [19-21]. Merwin [22] studied hot-rolled 0.1C medium-Mn steel annealed at 650°C. High tensile strength of ~800-1000 MPa and excellent elongation of 30-40% was obtained. Metastable austenite enhances work hardening rate to offset inadequate ductility in traditional ultra-fine grained high-strength steel. Cai et al. [23] elucidated the structure and properties of 0.18C-10.62Mn-4.06Al TRIP steel subjected to intercritical annealing, and excellent tensile elongation of 48%, ultimate tensile strength of 1012 MPa, and yield ratio of 0.58 was obtained. Luo [18] and Kamoutsi [24] carried out numerical simulations on nucleation and growth kinetics of reversed austenite in medium-Mn steel to elucidate the effect of carbon and manganese partitioning on austenite stability during intercritical annealing. However, the ART process and the beneficial effect of metastable austenite are applicable during cold rolling of thin plates for automotive applications. Only a few studies focused on the control of microstructural homogeneity and stability of austenite in ultra-low C medium-Mn and low-Al TRIP-assisted heavy plates [25].

The objective of this study is to study the microstructural evolution with respect to homogeneity in mechanical properties in an ultra-low carbon medium-manganese heavy plate (100 mm). Microstructural evolution, tensile behavior, and Charpy impact properties were studied after annealing at 630°C and 650°C in the $\alpha+\gamma$ two-phase region. Furthermore, the mechanism of enhanced ductility and toughness is discussed in terms of work hardening behavior and mechanical stability of austenite. An understanding of stability of metastable reversed austenite in terms of manganese partitioning, volume fraction, morphology, and size is elucidated.

2. Experimental material and procedure

The chemical composition of experimental steel is listed in Table 1. The ultra-low carbon and medium-manganese steels were prepared in a high frequency induction furnace in a vacuum atmosphere. Cr was added to increase both strength and corrosion resistance. The increase in strength by Cr can be attributed to solid solution strengthening and enhancement of hardenability, while corrosion resistance is related to the formation of chromium oxide film. Mo inhibits segregation of P to grain boundaries and suppresses intergranular fracture, which improves low-temperature toughness of tempered martensite [26].

150 kg ingot was forged into rectangular billet of thickness ~140 mm. Hot rolling and heat treatment process is illustrated in Figure 1. The 140 mm thick slab was heated to 1200°C for 2 h and then hot rolled to 100 mm with start and finish temperature of 980°C and 935°C, respectively, involving 5 passes. Total rolling reduction ratio (initial thickness/final thickness) was 1.4 and is lower than the conventional ultra-heavy plate that exceeds 3.0, because deformation was introduced into the core of plate to refine the prior austenite grain size [27]. Subsequently, the heavy plate was directly water-quenched to room temperature, using an accelerated cooling system to obtain martensitic microstructure. The cooling rates at $t/4$ and $t/2$ during direct quenching process were ~1.5 °C/s and ~0.84 °C/s, respectively, and were measured by thermocouple. After water quenching, the heavy plates were intercritically annealed for 30 min at 630°C and 650°C, between the two phase region ($\alpha+\gamma$). It took 80 min to attain the isothermal temperature. Finally, the heavy plates were air cooled to room temperature.

The phase transformation temperatures during continuous heating and quenching to room temperature were measured using a Formastor-FII dilatometer. The test specimens were cut from the

directly quenched steel plate, and machined to cylindrical specimens of dimensions 10 mm long and 3 mm diameter. The specimens were heated at a heating rate of 0.2 °C/s to 1000°C to simulate the slow heating process of heavy plate steel and then quenched to 20°C at 60°C/s. The critical temperatures A_{c3} , A_{c1} , M_s , and M_f were measured by dilatometer and were 758°C, 583°C, 432°C, and 197°C, respectively, as shown in Figure. 2a.

Moreover, the specimens were heated at a heating rate of 0.2 °C/s to 630°C and 650°C, respectively, to simulate the slow heating process of heavy plate steel. After holding for 30 min, the specimens were quenched to 20°C at 60 °C/s. The M_s and M_f for specimen annealed at 630°C were 199°C and 72°C, which were increased to 251°C and 116°C at 650°C, respectively. Dilatometric curves are presented in Figures 2b and c. The M_s temperature was decreased because of partitioning of alloying elements to austenite and coarse austenite grain size [28]. The phase transformation temperature of quenched steel after complete austenitization was higher than the intercritical annealed steel for 30 min. Moreover, the transformation temperatures of reversed austenite to martensite were increased with increase in intercritical annealing temperature.

Tensile and Charpy impact tests were carried out parallel to the rolling direction of plates. Tensile tests were performed using Shimadzu AG-X universal testing machine at room temperature at a crosshead speed of 3 mm/min according to ISO 6892-1:2009[29], using tensile specimens of length 30 mm and diameter 6 mm. Standard Charpy v-notch specimens ($10 \times 10 \times 55 \text{ mm}^3$) were impact tested at 20°C, 0°C, -20°C, -40°C, -60°C, respectively, using an Instron Dynatup 9200 series drop weight impact tester, consistent with ASTM E23 specification. Three tests were conducted for each condition and the tensile and impact toughness data reported was obtained using an average of 3 tests. The Charpy impact fracture

specimens were observed by FEI Quanta 600 scanning electron microscope (SEM).

The microstructure of specimens was observed by using a Leica DMIRM optical microscope (OM). Polished specimens along the rolling direction were examined by etching in 4 vol pct nital solution. TEM studies were conducted using 3 mm diameter thin foils, ground to a thickness of 40 μm , and twin-jet electropolished using a solution of 8% perchloric acid and 92% alcohol at -20°C . The XRD measurements were carried out to determine the volume fraction of austenite using D/max2400 X-ray diffractometer with Cu K α at room temperature. The integrated intensities of (200) γ , (220) γ , (311) γ , (200) α , and (211) α peaks were used to quantify the amount of retained austenite using equation (1) [30]:

$$V_{\gamma} = 1.4I_{\gamma} / (I_{\alpha} + 1.4I_{\gamma}) \quad (1)$$

where V_{γ} is the volume fraction of retained austenite, I_{γ} is the integrated intensity of γ peaks, and I_{α} is the integrated intensity of α peaks.

3. Results

3.1 Microstructural evolution

Thermodynamic calculations were carried out using Thermo-Calc combined with TCFE7 database to obtain the phase diagram, and is presented in Figure 3a. Moreover, C and Mn content in austenite at different temperatures was determined (Figure 3b). The relationship between enrichment of austenite by C and Mn and austenite fraction was studied to determine the appropriate intercritical annealing process. The volume fraction of FCC austenite was 32.8% at 630°C , and increased to 39.8% at 650°C . Cementite was completely dissolved at 573.2°C . The Mn-content in FCC decreased the increase in temperature, while C-content was first increased and then decreased. The Mn-content in wt. % in FCC austenite was 10.5 at 630°C and 9.2 at 650°C . A peak was observed in the carbon-content curve in FCC austenite, which

resulted from the dissolution of cementite [31].

Microstructural evolution across the thickness as imaged via optical microscope is presented in Figure 4. The optical micrographs of directly quenched specimens indicated fine lath martensite at $t/4$ and $t/2$ (Figures 4a and 4b). After intercritical annealing at 630°C , the optical micrographs at $t/4$ and $t/2$ consisted coarse lath martensite (Figures 4c and 4d). When annealing temperature was increased to 650°C , lath martensite continued to coarsen (Figures 4e and 4f). Similar microstructure of tempered martensite were observed at $t/4$ and $t/2$ locations.

TEM micrographs in Figure 5 show microstructural evolution at $t/4$ in directly quenched and annealed plates. It can be seen that the microstructure of directly quenched samples comprised of lath martensite with high dislocation density and a small amount of retained austenite (Figures 5a and 5b). There were $\sim 3\text{-}6$ martensite laths within each martensite packet. The width of lath martensite and retained austenite were $\sim 0.2\text{-}0.9\text{ }\mu\text{m}$ and $\sim 50\text{ nm}$, respectively. Figures 5c and 5d are bright field and dark field micrographs showing austenite film, when the specimens were annealed at 630°C , which was confirmed by selected area diffraction (SAED). The martensite structure was partially recovered through annihilation of dislocations. Luo et al. [18] studied the formation of stable austenite during intercritical annealing of 0.2C-5Mn steel. It was concluded that the nucleation of austenite took place at ferrite lath boundaries, instead of ferrite-cementite interface. This played a major role in increasing the austenite content during annealing, which was controlled by diffusion of Mn in both austenite and ferrite phases. However, retained austenite in the directly quenched specimen may also grow. The film-like reversed austenite of $\sim 20\text{-}200\text{ nm}$ width formed primarily along the lath martensite boundaries, and was present as a parallel layered structure. Figure 5e shows the microstructure of sample annealed at 650°C , which had

higher fraction of film-like austenite along with some blocky austenite. With increase in annealing temperature to 650°C, there was a small growth of austenite and the width of austenite lath was increased to ~100-300 nm. Figure 5f is the dark field micrograph of specimen annealed at 650°C with SAED pattern of austenite. Finally, laminated structure with sub-micron dimension and consisting of austenite-tempered martensite was obtained.

Figure 6 shows the XRD spectra of experimental steels. In the directly quenched spectra, peaks corresponding to α' martensite were observed, which implied near absence or insignificant content of retained austenite in the directly-quenched plates. The volume content of retained austenite at t/4 and t/2 was calculated to be 1.7% and 2.0%, respectively, using equation (1). Diffraction peaks of austenite phase were clearly visible in specimens annealed at 630°C and 650°C for 30 min, and the intensity of austenite peaks increased with increase in temperature from 630°C to 650°C with volume content of 12.6% and 19.1% at t/4, respectively. At t/2 location, steel annealed at 630°C and 650°C, the austenite content was 8.4% and 12.7%, respectively. Thus, austenite reversion (α' phase converted to γ phase) occurred during intercritical annealing.

3.2 Mechanical properties

Table 2 presents the tensile properties after intercritical annealing at 630°C and 650°C in experimental steels, and in Figure 6 engineering stress-strain plots are presented. When the directly quenched plate was annealed at 630°C, the yield strength, tensile strength, and elongation corresponding to t/4 were 728 MPa, 826 MPa, and 25.5%, respectively, and were 714 MPa, 814 MPa, and 24.2% for t/2. The yield ratio for both t/4 and t/2 was ~0.88. With increase in annealing temperature to 650°C, yield strength of t/4 was decreased from 728 MPa to 670 MPa, tensile strength remained unchanged at 825 MPa, and elongation

was significantly increased from 25.5% to 34.1%. For t/2, yield strength, tensile strength, and elongation were 638 MPa, 805 MPa, and 32.7%, respectively. Yield ratio was slightly decreased to 0.81 for t/4 and 0.79 for t/2. The tensile curves exhibited continuous yielding without any observable yield point, indicative of high density of mobile dislocations. In the experimental steel with tempered martensite and reversed austenite microstructure, an apparent work hardening was observed in the engineering stress-strain curve, as indicated by the arrow in Figure 7.

Table 3 presents the Charpy absorbed energy values with standard deviation in the test temperature range of -60°C to 20°C after intercritical annealing at 630°C and 650°C and the data is plotted in Figure 8. When the directly quenched plate was annealed at 630°C for 30 min, the impact energy at -60°C was 95.6 J and 81.2 J for t/4 and t/2, respectively. The impact energy at -60°C for both t/4 and t/2 was significantly increased to 139.4 J and 114.2 J, respectively, when the intercritical annealing was carried out at 650°C.

To study the fracture mode, the fracture surface of Charpy impact specimens was observed via SEM. SEM fractographs of annealed specimens tested at -60°C are presented in Figure 9. Figure 9a corresponds to t/4 specimens annealed at 630°C, and shows dimple mode of fracture with small and shallow dimples. However, the fracture surface of t/2 specimens annealed at 630°C was characterized by large dimples with some tearing in Figure 9b. Figures 9c and 9d show ductile-dimple fracture surface of specimens annealed at 650°C, and implied an improvement in fracture resistance based on small-size dimples. Moreover, the dimples were more homogeneously distributed at t/4. The spherical particles in different heat treatments were duplex inclusions consisting of MnS/Al₂O₃, as interpreted from EDS analysis (Figure 9c). Consequently, the impact toughness was significantly improved with increased annealing temperature. Compared to tempered martensite matrix, the reversed austenite is a relatively soft and ductile.

4. Discussion

4.1 Effect of Mn partitioning on the stability of austenite

EDS analysis was used across the martensite-austenite boundary to determine Mn-content in the sample annealed at 650°C (t/4). The Mn-content in tempered martensite matrix was in the range of ~1.7-3.6% (representative value = 1.95%) (Figure 10b) corresponding to circle 1 (Figure 10a). While the Mn-content in the reversed austenite was ~7.2-10.1% (representative value = 9.75%) (Figure 10d) corresponding to circle 3 (Figure 10a), which was similar to the value estimated by Thermo-Calc. The Mn-content at the interface was between tempered matrix and reversed austenite with a representative value of 5.98% (Figure 10c) corresponding to circle 2 (Figure 10a). The Mn-content in austenite on tempering at 650°C was ~7.2-10.1 wt.%, which exhibited considerable fluctuation. Whereas the equilibrium content of Mn in FCC phase was 9.25 wt.%, as estimated by Thermo-calc. Thus, the distribution of Mn in martensite and austenite was non-partitioning local equilibrium [32]. Diffusion rate of Mn in steel is generally low, and the resulting kinetics of Mn enrichment in austenite is therefore expected to be slow [33]. However, because of sub-micron lamellar microstructure, the diffusion distance of Mn for the applied heat treatment was probably adequate to significantly enrich Mn in austenite [34-35]. Thus, Mn was clearly partitioned to austenite phase during intercritical annealing. It was also proposed that the partitioning of C, Mn, Ni, and Cu occurs during intercritical annealing, which is helpful to stabilize austenite [31, 36-37].

The stability of reversed austenite is increased with increase in Mn-content [35, 38]. Grain refinement is also well-known to increase the stability of austenite by suppressing the martensite transformation [39, 40]. The calculated value of Mn-content continuously decreased with increase in

annealing temperature because of the dilution effect associated with increased austenite fraction [41]. When the experimental steel was intercritically annealed at a lower temperature of 630°C, a small amount of reversed austenite with a high content of austenite stabilizing element, Mn, was obtained. When the annealing temperature was increased to 650°C, the volume fraction of reversed austenite was increased, and the enrichment of Mn in reversed austenite was relatively less. Hence, the fine film-like reversed austenite formed at 630°C had higher stability in comparison to austenite formed at 650°C. This was further confirmed from the work hardening behavior (discussed below).

4.2 Effect of austenite stability on the work hardening behavior

To further study the mechanism of TRIP-assisted effect, the work hardening behavior of tensile specimens annealed at 630°C and 650°C was studied. Figure 11 shows work hardening rate as a function of true strain. The work hardening rate was rapidly decreased in the initial stage, and then increased, followed by a gradual decrease with increasing true strain. Reversed austenite with fine film-like morphology has higher stability compared to a coarse blocky morphology, which transforms to martensite at small strain [42]. The size of reversed austenite formed at 630°C in Figure 5c was finer than 650°C in Figure 5e. The extremely fine film-like microstructure formed at 630°C had higher stability in comparison to austenite formed at 650°C. At a strain of 0.01, the work hardening rate of specimens annealed at 630°C were 2170 at $t/4$ and 2799 at $t/2$, respectively. Corresponding to specimens annealed at 650°C, the work hardening rate was increased to 3589 at $t/4$ and 4252 at $t/2$, respectively. The reversed austenite in specimens annealed at 650°C transformed to martensite (TRIP effect), which led to significant increase in work hardening rate. Because of weak mechanical stability of austenite, the specimens annealed at 650°C had higher initial work hardening rate. The volume fraction of austenite formed at 650°C (19.1%)

exceeded 6.5% as compared to austenite at 630°C (12.6%) determined by XRD. The high volume fraction of austenite can sustain long-range work hardening behavior at 650°C. More metastable austenite prevented the occurrence of necking, and the ductility was enhanced.

In the initial stage, the work hardening rate is associated with high density of dislocations in the martensite matrix, as a result, the work hardening rate at $t/4$ was lower than $t/2$ because of greater extent of annealing. In the intermediate stage, with increasing strain, a large amount of metastable austenite transformed to martensite because of TRIP effect, and work hardening rate was increased. In the final stage, there was a small decrease because of competing effect between TRIP effect and movement of dislocations [43]. The work-hardening rate at $t/4$ had a tendency to exceed $t/2$ in the later period, because the high degree of annealing led to higher volume fraction of austenite at $t/4$. In summary, the work hardening behavior at $t/4$ and $t/2$ locations exhibited similar variation at identical annealing temperature, which confirmed the excellent homogeneity of microstructure and mechanical behavior.

Tensile flow curves, in particular, for 630°C specimens exhibited fluctuation during the early stage of deformation, indicating the possibility of discontinuous yielding. Large variation in strain hardening rate in Figure 11 during the initial stage of deformation supports this hypothesis. Meanwhile, this fluctuation was less for 650°C specimens with increased annealing temperature (presumably because of larger grain size). In the previous study, 6-10% medium-manganese steels with 0.13-0.3% carbon provided excellent properties resulting from the combination of twinning induced plasticity (TWIP) effect and TRIP effect operating together, which resulted in a high rate of work hardening and provide a distinct improvement in strength and ductility [28, 44]. In experimental steels, the enrichment of elements in austenite was insufficient because of lower alloying content (0.04C-5Mn). Although, higher austenite fraction was

obtained, the austenite with lower stability was more susceptible to deformation and transformed to martensite. Hence, the mechanical properties of 0.04C-5Mn steel is attributed to TRIP effect. However, there may be a weak TWIP effect because of small content of reversed austenite with higher stability.

4.3 TRIP effect and the mechanical stability of austenite

The relationship between the volume fraction of retained austenite and tensile strain at t/4 in specimens annealed at 650°C was studied. Figure 12a shows the XRD spectra of tensile specimens with increasing strain. Figure 12b presents the volume fraction of retained austenite phase as a function of tensile strain. The data indicates that with increasing strain, the volume fraction of austenite was decreased. The data was fitted to equation (2) [45].

$$f_r = f_{r_0} \exp(-k\varepsilon) \quad (2)$$

Where, f_{r_0} is the initial volume fraction of austenite phase, f_r is the volume fraction of austenite phase at a strain ε , and k is the constant.

The pre-exponential factor (f_{r_0}) of 19.1 was assigned as the initial austenite fraction, whereas the exponential factor (k) of 15.6 is related to characteristics that influence austenite stability [46]. The higher value of k represents lower stability of austenite, which indicates a higher driving force for strain-induced transformation. In the range where the strain is less than 0.1, the retained austenite fraction decreased dramatically from 19.1% to 4.1%, indicating rapid strain-induced transformation, followed by a slow decrease to 0.9% at a strain of 0.2. The initial amount of austenite and the rate of phase transformation determines the ductility and toughness of heavy plates.

The microstructure of tensile specimen deformed to 0.05 strain was characterized by TEM and is presented in Figure 13. A sub-micron lamellar martensite-austenite microstructure can be seen in Figure

13a. Figure 13b is bright field image of twin-type martensitic plates and corresponds to the magnified area in Figure 13a. The SAED pattern is presented in Figure 13d. Twin-type martensitic plates is a consequence of TRIP effect, and is the reason why the volume fraction of reversed austenite continuously decreased during tensile deformation [47].

The stability of austenite is affected by carbon and manganese-content of individual austenite grains, grain size and morphology, and the constraining effect of phases surrounding the austenite [42]. The ultra-low carbon microalloyed steel can not obtain high volume fraction of stable austenite, but medium-manganese content and partitioning provides a significant improvement in austenite stability. The TRIP effect of reversed austenite releases local stress, and hence delays necking and hinders the propagation of crack. Thus, excellent ductility and toughness were obtained.

5. Conclusions

In order to obtain good combination of strength and ductility/toughness in medium Mn steels, an ultra-low carbon medium manganese steel plate was designed. Intercritical annealing treatments were applied to heavy plate, and the microstructural evolution, mechanical properties, work hardening behavior, and the stability of austenite were studied. The conclusions are as follows:

- (1) The directly quenched steel consisted of thin lath-martensite of $\sim 0.2\text{-}0.9\ \mu\text{m}$ thickness and some retained austenite. On intercritical annealing, the lath martensite was coarsened, and reversed austenite was obtained, whose width increased from $\sim 20\text{-}100\ \text{nm}$ in sample annealed at 630°C to $\sim 100\text{-}300\ \text{nm}$ in sample annealed at 650°C . Cementite was dissolved in the matrix. Finally, a sub-micron laminated structure consisted of austenite-tempered martensite was obtained.
- (2) When the experimental steel was intercritically annealed at 630°C , the yield strength, elongation, and

impact energy at -60°C for $t/4$ was 728 MPa, 25.5%, and 95.6 J, respectively, and 714 MPa, 24.2%, and 81.2 J at $t/2$. When the annealing temperature was increased to 650°C , the strength was marginally decreased, and the toughness at low temperature was significantly increased. Alloying with 5 wt. % Mn significantly enhanced the homogeneity of the microstructure and mechanical properties across the thickness of plate because of high hardenability.

- (3) The enrichment of Mn to $\sim 7.2\text{-}10.1$ wt. % increased the tendency to revert to austenite during annealing. It is implied that Mn partitioning from martensite to austenite, and the stability of reversed austenite was significantly increased.
- (4) With increase in annealing temperature from 630°C to 650°C , the volume fraction of austenite was increased from 12.6% to 19.1% at $t/4$, and from 8.4% to 12.7% at $t/2$. The thickness of reversed austenite increased from $\sim 20\text{-}200$ nm to $\sim 100\text{-}300$ nm. The stability of reversed austenite was decreased because of reduced C and Mn enrichment in reversed austenite and grain growth.
- (5) The mechanical stability of austenite phase was explained in terms of work hardening behavior and TRIP effect of reversed austenite. The volume fraction of austenite decreased dramatically from 19.1% to 4.1% with increase in strain from 0 to 0.1, followed by a slow decrease of retained austenite to 0.9% at a strain of 0.2. Moreover, twin-type martensitic plates were obtained at 0.05 strain. The enhanced ductility and toughness is primarily attributed to TRIP effect.

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Table 1. Chemical composition of experimental steel (wt %).

Elements	C	Mn	Si	P	S	Cr	Ni	Cu	Mo	Al	Fe
Content	0.04	5.0	0.2	0.005	0.002	0.4	0.3	0.3	0.2	0.03	Bal.

Table 2. Tensile properties of experimental steels

	Yield strength /MPa	Tensile strength /MPa	Elongation /%	Reduction of area /%	Yield ratio
t/4 annealed at 630°C	728	826	25.5	68.1	0.88
t/2 annealed at 630°C	714	814	24.2	65.2	0.88
t/4 annealed at 650°C	670	825	34.1	69.4	0.81
t/2 annealed at 650°C	638	805	32.7	69.0	0.79

Table 3. Charpy impact energy with standard deviation of experimental steels as a function of test temperature

Steel number	20°C	0°C	-20°C	-40°C	-60°C
t/4 annealed at 630°C	184.5(9.5)	177.6(9.2)	164.7(10.8)	116.5(12.4)	95.6(15.7)
t/2 annealed at 630°C	200.1(9.7)	180.2(10.3)	152.1(9.8)	93.6(14.7)	81.2(17.5)
t/4 annealed at 650°C	227.1(7.9)	217.7(8.7)	206.5(9.6)	152.1(10.6)	139.4(10.9)
t/2 annealed at 650°C	206.9(8.3)	187.7(9.5)	176.4(8.4)	148.1(11.2)	114.2(13.8)

Figure 1. Schematic illustration of hot rolling and heat treatment process.

Figure 2. Dilatometric curves of specimens for different conditions: (a) continuous quenching after complete austenitization; (b) intercritically annealed at 630°C for 30 min; and (c) intercritically annealed at 650 °C for 30 min.

Figure 3. Calculated volume fraction of phase for experimental steel and weight percent of C and Mn in FCC: (a) volume fraction of phase and (b) weight percent of C and Mn in FCC.

Figure 4. Optical micrographs of experimental steels: (a) directly quenched specimen at $t/4$; (b) directly quenched specimen at $t/2$; (c) $t/4$ specimen annealed at 630°C ; (d) $t/2$ specimen annealed at 630°C ; (e) $t/4$ specimen annealed at 650°C ; and (f) $t/2$ specimen annealed at 650°C .

Figure 5. TEM micrographs illustrating microstructural evolution at $t/4$: (a) directly quenched specimen; (b) retained austenite in directly quenched specimen with SAED pattern of austenite; (c) bright field image of specimen annealed at 630°C; (d) dark field image of specimen annealed at 630°C with SAED pattern of austenite; (e) bright field image of specimen annealed at 650°C; and (f) dark field image of specimen annealed at 650°C with SAED pattern of austenite.

Figure 6. X-ray diffraction spectra of experimental steels: (a) t/4 specimen; and (b) t/2 specimen.

Figure 7. Engineering stress-strain curves of experimental steel.

Figure 8. Charpy impact energy plots with standard deviation as a function of test temperature.

Figure 9. SEM fractographs of Charpy impact specimens tested at -60°C : (a) t/4 specimens annealed at 630°C ; (b) t/2 specimens annealed at 630°C ; (c) t/4 specimens annealed at 650°C with EDS analysis; and (d) t/2 specimens annealed at 650°C .

Figure 10. (a) TEM micrograph at t/4 location after intercritical annealing at 650°C; (b) EDS analysis of tempered martensite matrix (Circle 1); (c) EDS analysis of tempered martensite and reversed austenite interface (Circle 2); and (d) EDS analysis of reversed austenite (Circle 3).

Figure 11. Work hardening rate curves as a function of true strain.

Figure 12. (a) XRD spectra of tensile specimens with increasing strain; and (b) the volume fraction of austenite phase.

Figure 13. (a) TEM micrographs of t/4 specimen annealed at 650 °C deformed at 0.05 strain; (b) bright field image of twin-type martensite plates at high magnification; (c) dark field image of twin-type martensite plates; and (d) SAED pattern of twin-type martensite plates.