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Effect of intermediate annealing on microstructure and mechanical property of a Fe–19Mn–0.6C TWIP steel

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Abstract

Annealing is an effective strategy to improve the properties of high-strength twin-induced plasticity (TWIP) steels, however, the adoption of intermediate annealing during cold rolling (CR) is scarcely studied. Here, the Fe–19Mn–0.6C TWIP steel was subjected to CR-annealing and CR-intermediate annealing-CR-annealing processes at room temperature to determine the role of intermediate annealing in the improvement of microstructure and mechanical properties. The total cold-rolled reduction in both processes is 75%. The morphological and phase characterizations of the TWIP steel annealed for 1 h showed that uneven element distribution had occurred as the annealing temperature was greater than the recrystallization start temperature, causing the presence of minor carbides. Moreover, the carbides vanished at the recrystallization end temperature and were quantitatively analyzed content via the refined XRD. Finally, the recrystallized austenite grains completely replaced the cold-deformed microstructures. At the same total CR reduction of 75%, the TWIP steel exerted intermediate annealing facilitates the formation of twins, endowing the tensile strength vast increase. This would provide a significant reference to improve the mechanical properties of steels via annealing.

1. Introduction

Lightweight steels with several advantages, saving energy and reducing emissions as well as safety, tempting increased attention paid to developing lightweight body materials [1–3]. Especially, TWIP steel with high strength and good plasticity becomes the candidate material for large-scale use in the automotive industry [4, 5]. The cold rolling (CR) process has always been employed to obtain high-strength thin plates to meet the requirements of the automotive industry. However, the cold-rolled TWIP steel exhibited poor elongation [6]. CR combined with subsequent annealing was an effective way to improve the plasticity of TWIP steel [7, 8]. The results showed that the microstructural evolution and mechanical properties of cold-rolled steel were strongly dependent on the annealing temperature. Therefore, studying the CR and annealing process (recovery, partial recrystallization, complete recrystallization, and so on) of TWIP steel featured a significant theoretical meaning and practical value.

The TWIP steel with a chemical of Fe-31Mn-3Al-3Si was studied by Dini et al [8], which mechanical properties were closely related to the CR reduction and subsequent partial recrystallization from annealing. The recovery kinetics in Fe-22Mn-0.6C steel was reported by Huang et al [9], revealing that steel with outstanding properties was obtained by CR and annealing. The influence of CR reduction on the microstructures and mechanical properties of TWIP steel was further researched by Mi et al [10]. The results showed that the best
The experimental TWIP steel in this study was self-designed and the chemical composition is shown in Table 1.

|   | C    | Mn   | Si   | S    | Cu   | Cr   | Ni   | Fe   |
|---|------|------|------|------|------|------|------|------|
|   | 0.590| 19.120| 0.124| 0.012| 0.025| 0.019| 0.006| Bal. |

Comprehensive mechanical properties can be gained from the TWIP steel with 65% CR and annealing at 1000 °C. Meanwhile, high-density fine twin structure, high strength, and elongation can be acquired via low-temperature recovery treated cold-rolled TWIP steel, which was proposed by Bouaziz et al. [11] and Diji et al. [8]. In addition, Kang et al. [12] and Schinhammer et al. [13] investigated the impact of recrystallization on microstructural evolution and mechanical properties, and the carbide precipitation during recrystallization heating was also studied. Meanwhile, several kinds of research on carbide precipitation and mechanical properties during recovery, partial recrystallization, and isothermal annealing of Fe-Mn-C TWIP had been reported [14].

In recent years, the development of TWIP steel mainly focused on cold-rolled microstructure and mechanical property variation [15–19]. How the annealing strategies affect microstructural evolution and corresponding properties remains a challenge and is urgent to study. To date, several studies about annealing during CR processes had been reported [6, 13, 20, 21]. For instance, Tewary et al. [6] used a hot rolled–annealing–cold rolled process to improve the microstructures and properties of low carbon high Mn TWIP steel, but their study focused on the influence of CR. The effect of multiple stages on how recrystallization and precipitation affected the properties of TWIP steel was reported by Schinhammer et al. [13]. Three-cycle deformation and subsequent annealing were employed to uniformly introduce more and thinner nano twins in FeMnSiC TWIP steel [20]. Outstanding mechanical properties of 1210 MPa yield strength and 18% tensile for medium-Mn TWIP steel were obtained by using cold rolling and intercritical annealing process [21]. However, these reports mainly focused on the effect of final annealing or deformation rather than intermediate annealing or various annealing strategies.

Inspired by the above analysis, this work aims to study the microstructural evolution and mechanical properties of the cold-rolled TWIP steel at various annealing strategies. The CR can endow Fe-19Mn-0.6C TWIP steel with high strength, while annealing can reduce dislocation density and improve ductility [22]. Moreover, Li et al. studied that Fe21W2C6 precipitates can improve the strength and plasticity of multi-alloying TWIP steel [23]. Yen et al. also considered that vanadium carbides can cause precipitation hardening for a Fe-21.6Mn-0.63C-0.87V TWIP steel [24]. Their study mainly focused on how precipitates affect properties, however, the study of how annealing affects carbide precipitation is quite lacking. Especially, for the TWIP steel with CR-intermediate annealing-CR-annealing process, the study of microstructural evolution during annealing as well as how this process improves microstructures and properties was scarce. Here, the processes of CR-intermediate annealing–CR-annealing and CR-annealing were carried out in the cold-rolled Fe-19Mn-0.6C TWIP, the microstructural evolution and properties in various annealing strategies were studied, which have significant guidance on developing a reasonable heat treatment process in producing automobile steel.

### 2. Experimental procedures

The experimental TWIP steel in this study was self-designed and the chemical composition is shown in Table 1. The Fe-19Mn-0.6C TWIP steel was melted in a 50 kg vacuum induction furnace, and using electromagnetic stirring refining. Finally, the ingot was hot-forged into a 20 kg weighted cylinder billet with a diameter of 100 mm. Subsequently, annealing at 1200 °C for 1 h was employed on the ingot, then the ingot was hot forged into a thickness of 20 mm for the original sheet.

The experimental sheet was cut into smaller pieces with a dimension of 120 × 30 × 20 mm³ to be suitable for CR. The total CR reduction of 75% was designed and realized by a two-high mill (200-type) at room temperature. The roll diameter is 310 mm and the CR rate is 0.4 m s⁻¹. Two types of CR schedules were used in this study and named schedule 1 and schedule 2, shown in scheme 1. In schedule 1, the samples were first cold-rolled for 75% reduction, followed by annealing at the temperatures of 400 °C, 450 °C, 485 °C, and 550 °C for 1 h. In schedule 2, the samples were initially cold-rolled for 50% reduction and then isothermally annealed at 415 °C for 1 h, following, cold-rolled 50% reduction again to acquire 75% total CR reduction, lastly, annealing treatment was conducted at the temperatures of 400 °C, 450 °C, 485 °C, and 550 °C for 1 h. In schedule 1 and schedule 2, without or with intermediate annealing, the tested TWIP steels both feature 75% total CR and a similar annealing process.

Optical microscopy (OM, Zeiss Axiovert 200-MAT) was conducted to observe the microstructural evolution of CR and annealing treatment samples. The longitudinal section and cross-section (rolling section) were
selected for non-recrystallization and recrystallization microstructures, respectively. All the samples were etched with 4 vol% alcohol nitrate solution after the specimens were polished using standard metallographic procedures. Moreover, electropolishing was carried out to eliminate the remained deformation layer before characterizations, performing for 15 s (optical and electron microscopy) or 1 min (phase analysis) at room temperature at 20 V. The electropolishing solution was a mixture of 90% acetic acid and 10% perchloric acid.

Hitachi FEM-SEM (S-3400 N) equipped with energy-dispersive x-ray spectroscopy (EDS) was used for microstructures and elements analysis. To qualitatively compare the distribution of all elements at the CR and annealing grains, the spot-line scanning analysis was measured [25, 26]. X-ray diffractometer (XRD, D/max-2500/PC), with Cu target Kα line at the voltage of 40 kV and a current of 200 mA, was used to qualitatively determine the dislocation density towards the cold-rolled and annealed TWIP steel. The XRD patterns of the samples were analyzed by the Rietveld method as implemented in the Material Analysis Using Diffraction (MAUD) software package [27]. A pseudo-Voigt peak shape function with the asymmetry correction included was employed to obtain Rietveld Quantitative Phase Analysis (RQPA) [26, 28]. The refined overall parameters were: phase scale factors, background coefficients, unit cell parameters, zero-shift error, peak shape parameters, and preferred orientation coefficient if needed. March-Dollase ellipsoidal preferred orientation correction algorithm was employed [29, 30]. The background of each experimental pattern was fitted by a polynomial of degree five. For transmission electron microscopy (TEM), typical 3 mm diameter discs were punched out and subsequently electro-polished in a 95% ethanol and 5% perchloric acid solution using a twin jet electro polisher (Tenupol-5, Struers, Denmark) at −30 °C. The thin foils were examined in a JEM-2010 high-resolution TEM for microstructure analysis. For XRD testing, the degree ranged from 20° to 100° at a constant step of 2° min−1, using a graphite monochromatic filter and Si powder as the internal standard. The tensile tests were carried out at room temperature by using an Instron 5582 testing machine with an optical extensometer. Tensile specimens, with a gauge length of 20 mm and cross-section of 1×2.2 mm², were mechanized along with the tensile axis parallel to the rolling axis. Each tensile data was determined by repeating it three times at a strain rate of 2×10⁻³ s⁻¹.

3. Results and discussions

3.1. Microstructural evolution of cold-rolled TWIP steel after annealing

The typical morphologies of 75% reduction cold-rolled Fe-19Mn-0.6C TWIP steel at various annealing temperatures were displayed in figures 1 and 2. Compared to the recovery microstructures (figures 1(b), (c), and 2(c)), the cold-rolled microstructures (figures 1(a) and 2(b)) exhibited a lot of twins. Annealing at 400 °C, the recovery microstructure without any change, still featured numerous deformation twins. The selected area electron diffraction (SAED) pattern in figure 2(b) showed twin spots along the [110] zone axis, implying the presence of the austenite phase. Extremely tiny grains were formed in elongated grains at 450 °C (figures 1(d) and (e)), suggesting the occurrence of partial recrystallization. Meanwhile, very fine-started recrystallized subgrains can be also observed (figure 2(d)). As the annealing temperature increased to 485 °C, the recrystallization degree further increased (figures 1(f) and (g)). Nucleation tended to generate at grain boundaries (GBs), which may be caused by the vast newly formed recrystallized fine grains gathered at GBs. Figure 1(h) (cross-section) and (i) (rolling section) showed the optical microstructures of cold-rolled samples at 550 °C, showing new-formed recrystallized grains. Grains in the deformed zone, with severe cold deformation and offering a large driving force for recrystallization during annealing, were relatively intensive and smaller, showing the uneven distribution of grains and their size, thus, the ending recrystallization temperature was determined to be 550 °C. The recrystallized grains would start to grow up and distribute gradually uniformly as the annealing temperature further increased.

To deeply explore the phase transformation of the TWIP steel with 75% CR reduction during the annealing process, the XRD patterns are conducted (figure 3(a)). Same to the pattern in cold-rolled TWIP steel, the XRD
Figure 1. Optical microstructures with 75% CR after annealing for 1 h at various temperatures. (a) cold-rolled; (b) 400 °C and (c) 400 °C at rolling section; (d) 450 °C and (e) 450 °C at rolling section; (f) 485 °C and (g) 485 °C at rolling section; (h) 550 °C and (i) 550 °C at rolling section.

Figure 2. Microstructural characterizations. (a) SEM image and (b) TEM bright-field micrograph of 75% cold-rolled TWIP steel, and the SAED pattern of twin and matrix is inserted. TEM bright-field micrograph of 75% CR reduction after annealing for 1 h at (c) 400 °C and (d) 450 °C.
peaks at 400 °C were also assigned to a single austenite phase rather than other phases, implying this state was a recovery phase. A few subgrains were arisen in figures 2(d) and 3(b), as the annealing temperature increased to 450 °C, suggesting the presence of partial recrystallization. Moreover, as indexed in figure 3(a), the carbides begin to appear at 450 °C. The intensity of carbide peaks was slightly increased at 485 °C. To quantitatively analyze the phase fraction of austenite and carbides, the XRD patterns for 450 °C and 485 °C annealed TWIP steel were refined via the Rietveld method (figures 3(c) and (d)). As a result, the carbide content increased from 1.5% at 450 °C to 2.4% at 485 °C. However, the peaks for the carbides disappeared as the temperature was further increased to 550 °C, implying the elimination of segregation (figure 3(a)).

The absence of additional peaks in the cold-rolled Fe-19Mn-0.6C TWIP steel demonstrated that the CR process cannot induce phase transformation. However, minor carbides appeared at the recrystallization annealing temperature of 450 °C. It can be deduced that the precipitation of carbides emerged in TWIP steel during the recrystallization process, which was caused by segregation rather than cold deformation. To further analyze the reasons for the presence of carbides during the annealing, SEM and EDS are applied to determine the element distribution in 75% cold-rolled TWIP steel and subsequently annealed at 400 °C.

As the annealing temperature increased to 450 °C, plenty of fine grains were generated (figures 3(a) and (b)), indicating the occurrence of partial recrystallization. Several carbides also can be observed, which was consistent with the results in figure 3(a). Moreover, the EDS path, which was passed through twins to the newly formed tiny grains, was screened to analyze the elemental distribution (figure 3(c)). An even distribution of Mn element, while a Fe-poor zone and a C-rich zone can be observed. The C-rich zones (figure 3(c)) and the generated few carbides (figures 3(a) and (b)) indicated that the generated minor carbides were closely related to C-elemental segregation. As shown in figures 3(d) and (e), the partial recrystallization content increased with the annealing
temperature reaching 485 °C. Mn element remained in even distribution at this temperature while the C-rich zones enlarged (figure 5(f)), which was consistent with the carbides increased trend in figure 3(a). The carbides increased with the C segregation degree increasing, suggesting that C segregation was the main reason for the generation of carbides. As a result, element content variation would induce the presence of precipitates, meanwhile, carbide content was proportional to the C segregation degree. The SEM microstructures at 550 °C exhibited completed recrystallization (figures 5(g) and (h)), implying the presence of numerous fine austenite grains with relatively C-rich content. The slight variation of Fe, Mn, and C (figure 5(i)) and the absence of carbides (figure 5(a)) indicated that the C segregation gradually disappeared with the completed recrystallization, resulting in the formed carbides was not enough.

**Figure 4.** The EDS images of rolled and recovery TWIP steel with 75% CR (the corresponding SEM images were inserted in the EDS images). (a) as-rolled; and (b) 400 °C.

**Figure 5.** SEM and corresponding EDS line profiles of 75% cold-rolled TWIP steel at various annealing temperatures. (a)–(c) 450 °C; (d)–(f) 485 °C; and (g)–(i) 550 °C.
Deformation bands also can be observed. The 50% microstructures and mechanical properties. Above analysis, it is urgent to reveal the differences between the various annealing strategies and their effects on content and thinner twin lamellae can be obtained as the CR reduction was further increased. Inspired by the thinner twins than that of 75% TWIP steel at 415 °C. In general, the recovery annealing can reduce dislocation density, thus, higher twin content and thinner twin lamellae can be obtained as the CR reduction was further increased. Inspired by the above analysis, it is urgent to reveal the differences between the various annealing strategies and their effects on microstructures and mechanical properties.

The optical microstructures of 50%–415 °C–50% TWIP steel at various annealing temperatures (as-rolled, 400 °C, 450 °C, 485 °C, and 550 °C) were shown in figure 7. At the same total CR reduction of 75%, the deformation bands also can be observed. The 50%–415 °C–50% TWIP steel (figures 8(a) and (b)) exhibited thinner twins than that of 75% TWIP steel (figures 2(a) and (b)), which exhibited straight interfaces while throughout the overall grains, suggesting they were deformation twins. Meanwhile, the increased twin content was received as the introduction of intermediate annealing at 415 °C. Compared with 75%–400 °C (figures 2(a) and (b)), however, the 50%–415 °C–50% TWIP steel featured decreased dislocation density (figures 8(a) and (b)). In 75%–400 °C TWIP steel, the absence of intermediate annealing induced work hardening and higher dislocation density. It can be concluded that the interaction existed between dislocations and twins. The moved dislocations, which were hindered by twin boundaries, can impede the twin formation of TWIP steel at the CR process.

The 50%–450 °C–50% TWIP steel exhibited tiny grains at 450 °C, generated at elongated grains and stemmed from partial recrystallization (figure 7(c)), which was similar to the microstructures in 75%–450 °C (figure 2(d)). As a result, the microstructural evolution for the total 75% CR reduction at various annealing strategies of 450 °C and 485 °C exhibited similarities (figures 7(c) and (d); figures 1(c) and (d)). The microstructures showed completed recrystallization at the intermediate annealing temperature of 550 °C (figures 7(e) and (f)), which was similar to that in 75%–550 °C (figures 1(e) and (f)). Therefore, it can be considered that intermediate annealing almost had no impact on the ultimate recrystallized grains. One reason for the formed fine grains was that larger CR reduction provided a larger recrystallization driving force. On the other hand, the presence of carbide resulted from the C segregation as the high carbon content in tested steel, which can pin the GBs and suppress grain coarsening.

In the cold-rolled TWIP steel, the presence of recrystallized grains with C-rich zones caused the presence of carbides. The C segregation induced the formation of carbides and C-poor zones. For the C element, Song et al proposed that [29] the C segregation can stabilize the austenite interface as the lower C content. Similar results also had been revealed by Karimi et al [30]. In detail, the carbides preferentially nucleated at prior austenite GBs in the Mn-poor and C-poor zones. Before the presence of carbides, Mn reduced C activity and stimulated C to disperse in Mn-rich zones of austenite. As the completion of recrystallization, however, the presence of carbides and element segregation gradually diminished or even disappeared. Hence, C segregation degree and carbide generation followed the order of increase, weaken, and disappear.

3.2. The mechanism of annealing strategies affecting microstructures and properties
In the view of production, cold-rolled TWIP steel with large reduction is always likely accompanied by intermediate stress relief annealing. In this study, we annealed the 50% cold-rolled TWIP steel at 415 °C, then exerted a further 50% CR reduction to reach 75% total CR reduction, and the sample was named 50%–415 °C–50% TWIP steel. As shown in figure 6, the phase of 50% cold-rolled TWIP steel exhibited no change after recovery annealing at 415 °C. In general, the recovery annealing can reduce dislocation density, thus, higher twin content and thinner twin lamellae can be obtained as the CR reduction was further increased. Inspired by the above analysis, it is urgent to reveal the differences between the various annealing strategies and their effects on microstructures and mechanical properties.

The XRD patterns of as-rolled and 415 °C-1 h TWIP steel.
Furthermore, we revealed the variety of phases and dislocation density at various states of TWIP steel. Here, take 50%–415 °C–50% and as-rolled 75% TWIP steel as examples. As shown in figure 8(c), the presence of carbide precipitates indicated that intermediate annealing at 415 °C did not induced phase transformation. The 50%–415 °C–50% TWIP steel exhibited more carbide precipitates than that of 75% TWIP steel, suggesting the significant effects of intermediate annealing. In 50%–415 °C–50% TWIP steel, the element diffusion-induced carbide precipitates were generated at the intermediate annealing temperature of 415 °C. Furthermore, the second 50% CR and annealed at 400 °C was exerted on the TWIP steel, and the carbides exhibited declined as the carbides may occur dissolved at 400 °C. For 75%–400 °C TWIP steel, the carbide precipitates formed at 400 °C,
and the continuous 75% CR resulted in larger precipitation kinetics, thus exhibiting higher carbide precipitates content than that of 50%–415 °C–50%–400 °C (figure 8(d)). As a result, XRD patterns at various annealing strategies just can reveal the carbide content change rather than any phase transformation.

To further verify the result of 50%–415 °C–50% TWIP steel with reduced dislocation density than that of 75% TWIP steel, the quantitative calculation of dislocation density at various states is required. Equation (1) [31] is used to compute the dislocation density in TWIP steel.

$$\rho = \frac{3\sqrt{2\pi} (\frac{a^2}{b^2})^{1/2}}{Db}$$

Wherein, $\rho$ means the density of dislocations, $D$ represents grain size, $(\frac{a^2}{b^2})^{1/2}$ displays the strain value, and $b$ means the Burgers vector ($b = a/\sqrt{2}$, $a$ is the lattice constant).

The dislocation density of TWIP steel at different states was calculated from figures 6 and 8(c)–(d) and listed in table 2. The 50% TWIP steel exhibited a higher dislocation density ($9.85 \times 10^{15} \text{m}^{-2}$) than that of 50%–415 °C TWIP steel ($5.1 \times 10^{15} \text{m}^{-2}$), implying the recovery annealing had a significant impact on dislocation density. As the CR reduction increased from 50% to 75%, the dislocation density increased from $9.85 \times 10^{15} \text{m}^{-2}$ to $10.92 \times 10^{15} \text{m}^{-2}$. Hence, a larger CR reduction exhibited an ignorable influence on dislocation density. Compared with 50%–415 °C TWIP steel ($5.1 \times 10^{15} \text{m}^{-2}$), however, the dislocation density of 50%–415 °C–50% TWIP steel ($10.15 \times 10^{15} \text{m}^{-2}$) showed a huge increase. Meanwhile, the twin refinement degree of 50%–415 °C–50% TWIP steel exceeded that of 75% TWIP steel. As shown in figure 8(b), the twin occurred through increase, thinning, and further nucleation. For the CR-annealing-CR process, as shown in table 2, the first CR and subsequent annealing offered a lower dislocation density and the second CR increased the dislocation density.

Furthermore, the 75% TWIP steels exhibited higher dislocation density as compared with 50%–415 °C–50% TWIP steel weather for as-rolled or annealing (table 2). For the tested steels with the same total CR reduction, as per the above-analyzed, the intermediate annealing moderately reduced dislocation density, hence, improving the mechanical properties of TWIP steel.

To guide the annealing process in practice, it is urgent to analyze the mechanical properties of Fe-19Mn-0.6C TWIP steel with a total 75% CR reduction at various annealing strategies. The engineering stress–strain curves of 75% cold-rolled TWIP steel annealed at different temperatures were shown in figure 9. The 75% cold-rolled TWIP steel featured a high tensile strength (1919 MPa) and yield strength (1700 MPa) but a low elongation of 3.2%. However, the minor elongation but high tensile strength and yield strength at this state cannot meet the requirements of production application. The various annealing strategies, therefore, are employed to increase elongation while remain the high strength of TWIP steel. As shown in figure 9(a), the 75%–400 °C TWIP steel exhibited improved elongation while the slightly declined tensile strength and yield strength. The strength occurred a significant decline and the elongation showed almost no change as the annealing temperature increased to 450 °C. The reasons can be summarized as the dislocation density was greatly reduced at the early stage of recrystallization, which led to decreased work hardening and sharply reduced strength.

As shown in figure 9(a), 75%–485 °C TWIP steel featured a yield strength of 1210 MPa, a tensile strength of 1460 MPa, and an elongation of 10.5%, suggesting partial recrystallization can deliver a better mechanical property. Furthermore, a larger elongation of 49% was obtained at the completed recrystallization temperature of 550 °C (figure 9(a)), meanwhile, the yield strength and ultimate tensile strength both conformed to the practical production applications. The changing trend of strength and elongation as the annealing temperature was plotted in figures 9(c) and (d). As a result, the yield strength and ultimate tensile strength declined, as the cold-rolled TWIP steels transformed from recovery to partial recrystallization, while the elongation increased.

| Processing     | Grain size (nm) | Strain ($10^{-4}$) | Dislocation density ($\text{m}^{-2}$) |
|----------------|----------------|--------------------|--------------------------------------|
| 50%            | 23.48          | 78.5               | $9.85 \times 10^{15}$                |
| 50%–415 °C     | 27.08          | 46                 | $5.1 \times 10^{15}$                 |
| 75%            | 18.82          | 69.8               | $10.92 \times 10^{15}$               |
| 75%–400 °C     | 22.18          | 46.7               | $6.2 \times 10^{15}$                 |
| 50%–415 °C–50% | 17.57          | 66.5               | $10.15 \times 10^{15}$               |
| 50%–415 °C–50%–400 °C | 23.67 | 46.8 | $6.06 \times 10^{15}$ |
For the TWIP steel with annealing treatment, the C element tended to segregate at 450 °C and formed carbides. The formed carbides may serve as a stress concentration center and further lead to the fracture. Hence, the recrystallization rather than partial recrystallization temperature facilitated improved elongation for the TWIP steel with a large CR reduction.

As discussed above, 50%–415 °C–50% TWIP steels exhibited tiny grains and decreased dislocation density, the engineering stress-strain curves of which were annealed at various temperatures were drafted in figure 9(b). The changing trend for kinds of 50%–415 °C–50% TWIP steel was similar to that of 75% rolled steel at various annealing temperatures. The C segregation feature can be observed from various degrees of partial recrystallization occurring at the annealing temperature ranging from 450 °C–550 °C. For the Fe-19Mn-0.6C TWIP steel with 75% CR reduction, annealing at partial recrystallization was detrimental to improving its mechanical property. Comparing the data in figure 9, it can be concluded that exerting intermediate annealing during CR can greatly increase ultimate tensile strength and yield strength while elongation showed a slight change. The process of CR-annealing-CR was a promising strategy to improve the strength of designed steel at a large CR reduction while offering considerable elongation.

4. Summary

Microstructure and mechanical property evolution of the cold-rolled Fe-19Mn-0.6C TWIP steel at various annealing strategies are studied. The main results can be summarized as follows:

1. During the annealing process, the C segregation induces C-poor zones and facilitates the formation of carbides. At a temperature lower than the ending recrystallization temperature but higher than the recovery temperature, the carbide content increased as the annealing temperature increased.

2. For Fe-19Mn-0.6C TWIP steel with the same total CR, exerting intermediate annealing in the CR process can reduce dislocation density, increase twin content, and refine twin lamellae, thus improving the mechanical properties of TWIP steel.

3. Compared to the CR-annealing process, the CR-intermediate annealing-CR-annealing process featured higher ultimate tensile strength and yield strength while elongation showed a slight change as the annealing temperature was lower than 450 °C, exhibiting improvement in mechanical properties of TWIP steels.
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Data availability statement

The data that support the findings of this study are available upon reasonable request from the authors.

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References

[1] Fu H, Yan Y, Zhang J L and Li J X 2022 Annealing treatment induced martensite formation and evolution in TWIP steel Mater. Lett. 308 131110

[2] Fu H, Wang W, Chen X, Pia G and Li J 2020 Grain boundary design based on fractal theory to improve intergranular corrosion resistance of TWIP steels Mater. Design 185 108253

[3] Yang Z, Yan D, Lu W and Li Z 2019 Fractal and multifractal analysis of fracture surfaces caused by hydrogen embrittlement in High-Mn twin transformation-induced plasticity steels Appl. Surf. Sci. 470 870–81

[4] Shao C W, Zhang P, Zhu Y K, Zhang Z J, Tian Y Z and Zhang Z F 2018 Simultaneous improvement of strength and plasticity: additional work-hardening from gradient microstructure Acta Mater. 145 413–28

[5] Lee T, Koyama M, Tsuzaki K, Lee Y H and Lee C S 2012 Tensile deformation behavior of Fe-Mn–C TWIP steel with ultrafine elongated grain structure Mater. Lett. 75 169–71

[6] Tewary N K, Ghosh S K, Bera S, Chakrabarti D and Chatterjee S 2014 Influence of cold rolling on microstructure, texture and mechanical properties of low carbon high Fe TWIP steel Mater. Sci. Eng. A 615 405–15

[7] Santos D B, Saleh A A, Gazder A A, Carman A, Duarte D M, Ribeiro É A S, Gonzalez B M and Pereloma E V 2011 Effect of annealing on the microstructure and mechanical properties of cold rolled Fe–24Mn–3Al–2Si–1Ni–0.06C TWIP steel Mater. Sci. Eng. A 528 S545–53

[8] Dini G, Najafizadeh A, Uejii R and Monir-Vaghefi S M 2010 Improved tensile properties of partially recrystallized submicron grained TWIP steel Mater. Lett. 64 15–8

[9] Yuan G W and Huang M X 2014 Supper strong nanostructured TWIP steels for automotive applications Prog. Nat. Sci. 24 50–5

[10] Mi Z L, Dai Y J, Wang H Q and Li S S 2007 Influence of cold rolling reduction on microstructure and mechanical properties of TWIP steel Acta Metall. Sin. 20 441–7

[11] Bouaziz O, Scott C P and Pettingad G 2009 Nanostructured steel with high work-hardening by the exploitation of the thermal stability of mechanically induced twins Scripta Mater. 60 714–6

[12] Kang S, Jung Y S, Jun J H and Lee Y K 2010 Effects of recrystallization annealing temperature on carbide precipitation, microstructure, and mechanical properties in Fe–18Mn–0.6C–1.5Al TWIP Steel Mater. Sci. Eng. A 527 745–51

[13] Schinhammer M, Pecnik CM, Rechberger F, Hänzi A C, Löffler F J F and Uggowitzer P J 2012 Recrystallization behavior, microstructure evolution and mechanical properties of biodegradable Fe–Mn–C(–Pd) TWIP Alloys Acta Mater. 60 2746–56

[14] Liu J B, Liu X H, Liu W, Zeng Y W and Shu K Y 2010 Microstructure and hardness evolution during isothermal process At 700 °C for Fe–24Mn–0.7Si–1.0Al TWIP steel Mater. Charact. 61 1356–8

[15] Xu J, Wang Z, Yan Y, Li J and Wu M 2020 Effect of hot/warm rolling on the microstructures and mechanical properties of medium-Mn steels Mater. Charact. 170 110682

[16] Salas Reyes A E, Altamirano Guerrero G, Flores Alvarez J F, Chávez Alcalá J F, Salinas A, Figueroa I A and Lara G 2020 Rodriguez, influence of the As–Cast and cold rolled microstructural conditions over corrosion resistance in an advanced TWIP steel microalloyed with boron J. Mater. Res. Technol. 9 4034–43

[17] Li D, Qian L, Wei G, Liu S, Zhang F and Meng J 2020 The role of Mn on twin behavior and tensile properties of coarse- and fine-grained Fe–Mn–C twin-induced plasticity Mater. Sci. Eng. A 789 139586

[18] Ding J, Shang Z, Li J, Wang H and Zhang X 2020 Microstructure and Tensile Behavior of Nanostructured Gradient TWIP Steel Mater. Sci. Eng. A 785 139346

[19] Yu W X, Liu B X, He J N, Chen C X, Fang W and Yin F X 2019 Microstructure characteristics, strengthening and toughening mechanism of rolled and aged multilayer TWIP maraging steels Mater. Sci. Eng. A 767 138426

[20] Jing T F, Zheng H B, Liao Q, Song I X, Peng H B and Wen Y H 2022 Homogeneously introducing more and thinner nanotwins by engineering annealing twin boundaries: a TWIP steel as an example Mater. Scic. Eng. A 840 142908

[21] Bai S, Xiao W, Niu W, Wang Y, Li D, Zhang W, Shi Q and Liang W 2021 Microstructural evolution and mechanical properties of V-containing medium-Mn steel manufactured via cold rolling and intercriticall annealing J. Mater. Res. Technol. 14 1504–17

[22] Anand K K, Mahato B, Haase C, Kumar A and Ghosh S 2018 Chowdhury, correlation of defect density with texture evolution during cold rolling of a twin-induced plasticity (TWIP) steel Mater. Sci. Eng. A 711 69–77

[23] Li S, Chen C, Chen Y and Wang X 2020 The simultaneous improvement of strength and plasticity of a novel alloying steel by precipitation recovery annealing Lett. 270 127710

[24] Yen H W, Huang M, Scott C P and Yang J R 2012 Interactions between deformation-induced defects and carbides in a vanadium-containing TWIP steel Scripta Mater. 66 1018–23

[25] Sabet Ghorabaei A and Ghasemi Banadkouki S S 2017 Abnormal mechanical behavior of a medium-carbon steel under strong ferrite-pearlitic-martensite triple-phase microstructures Mater. Sci. Eng. A 700 562–73

[26] Huang Z, Jiayang Y, Hou A, Wang P, Shi Q, Hou Q and Liu X 2017 Rietveld refinement, microstructure and high-temperature oxidation characteristics of low-density high manganese steels J. Mater. Sci. Technol. 33 1531–9

[27] Finger D E, Li W and Jephcoat A P 1994 A correction for powder diffraction peak asymmetry due to axial divergence J. Appl. Crystallogr. 27 892–900

11
[28] Dollase W A 1986 Correction of intensities for preferred orientation in powder diffractometry: application of the march model J. Appl. Crystallogr. 19 267–72
[29] Song C, Yu H, Li L and Lu J 2016 Effect of carbon at interface of austenite on manganese segregation of low carbon and manganese steel Mater. Lett. 174 75–8
[30] Karimi Y, Hossein Nedjad S, Miyamoto G, Shirazi H and Furuhasha T 2017 Banding effects on the process of grain refinement by cold deformation and recrystallization of acicular C-Mn steel Mater. Sci. Eng. A 697 1–7
[31] Dini G, Ueji R, Najafizadeh A and Monir-Vaghefi SM 2010 Flow stress analysis of TWIP steel via the XRD measurement of dislocation density Mater. Sci. Eng. A 527 2759–63