Analysis of the relationship between microstructure and mechanical properties of intercritically annealed 3.5Mn steel

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Abstract
The paper mainly studies intercritically annealed medium manganese steel, which only contains 3.5 wt% Mn. After annealing at different temperature, the microstructure of the experimental steel is composed of ferrite and austenite with different morphology. With the increase of intercritical annealing temperature, a small amount of martensite appears in the microstructure due to the decrease of austenite stability, and the proportion of retained austenite decreases. When the experimental steel is annealed at 700 °C, the best matching of strength and plasticity is obtained, which benefits from the appropriate proportion of austenite with different stability. The results show that excellent mechanical properties of 3.5 wt% Mn steel can be achieved by adjusting the intercritical annealing process.

1. Introduction

Environmental protection and light weighting have become an inevitable trend in the development of the automotive industry. A body made of advanced high-strength steel is the best way to achieve lightweight and improve crash safety at the same time. At present, the third generation of advanced high-strength steels under development includes lightweight steel, quenching and partitioning (Q&P) steel, and medium manganese steel [11] (3–12 wt% Mn). Among them, medium manganese steel with excellent comprehensive mechanical properties and low alloy cost has attracted the attention of materials scholars all over the world [2–8]. The high product of strength and elongation of medium manganese steel is closely related to the meta-stable austenite formed in austenite reverse transformation [9, 10]. Therefore, regulating the retained austenite phase is the key to achieving high strength and high plasticity for medium manganese steel. Literatures [11–16] show that the stability of the austenite is related to its size and morphology of the surrounding matrix. Small-sized retained austenite grains and thin films shaped retained austenite has relatively better stability.

However, most existing researches regarding the medium Mn focused on the steels with a Mn content of 5–12 wt%. Although high Mn content could provide better properties for the steel, also it can easily lead to chemical segregation in smelting and casting process, resulting in a banded distribution of microstructure in steel, which seriously affects the macro mechanical properties of the materials. Thus, one leading direction for medium Mn steel is low alloy composition design, while meeting the mechanical properties of the third-generation advanced high-strength steel, it will further reduce the cost of medium Mn steel and the difficulty of industrial production. De Moor et al [17] investigated the microstructure-property relationship of 3.3Mn steel after the Q&P process, and the best combinations of tensile strength and total elongation obtained in the 0.2C–3Mn–1.6Si steel after intercritical annealing was associated with strength levels in the 1000–1200 MPa range and total elongations ranging from 14 to 20%. Grajcar et al [18] investigated 3 Mn and 5 Mn steels in thermo-mechanical processing resulting in bainitic structures with retained austenite. Among them, 3Mn steel has...
achieved mechanical properties with the tensile strength of 960 MPa and elongation of 15%. Obviously, due to the lower Mn content, the mechanical properties of the steel in the above article are not outstanding. Therefore, the present work takes 3.5Mn steel as the research, and explores the quantitative relationship between the microstructure and mechanical properties at different intercritical annealing temperatures.

2. Materials and methods

The chemical composition of the experimental steel is 0.23C–0.96Si–3.51Mn–0.95Al–0.48V–0.20Mo (wt%). After reheating to 1200 °C for 2 h, a 50 kg ingot was hot-rolled to a thickness of 3.8 mm by five passes, and air-cooled to room temperature. Rectangular specimens of 30 × 100 mm were machined from the hot-rolled steel sheet parallel to the rolling direction and then prepared for the heat treatment on the electric box furnace (SX2-16-13). The variation trend of austenite alloy composition and phase fraction with temperature was calculated by Thermo-Calc software based on the TCFE10 database, as shown in figure 1. According to the phase diagram, four intercritical annealing temperatures of 675 °C, 700 °C, 725 °C and 800 °C were established for the experimental steel to obtain different phase proportions and related mechanical properties. The schematic diagram of the hot rolling and intercritical annealing process is presented in figure 2.

Rectangular specimens of 10 × 4 mm were cut from the hot-rolled plates, and the DIL805 A type dilatometer was used to simulate the austenite reversion during the intercritical annealing process. The microstructure of the experimental steel was characterized by scanning electron microscope (Zeiss ultra 55). Twin-jet polishing technique was implemented to prepared thin foil specimens for the transmission electron microscopy (G20). X-ray diffraction was used to measure the fraction of retained austenite. The sample (15 × 15 mm) was detected by the XRD at room temperature using D/MAX-RB operating at 45 kV and 150 mA. To test the mechanical properties of the experimental steel, a dog-bone tensile specimen with a gauge length of 25 mm was used for uniaxial tensile testing at a speed of 1 mm s⁻¹ at room temperature.
The *in situ* electron backscatter diffraction (EBSD) test adopts tensile specimens with a gauge distance of 10 mm, as shown in figure 3, and the tensile deformation is carried out at a speed of 0.5 mm min\(^{-1}\). The phi-710 type Auger nano-electron probe equipped with electron backscattering diffraction was used to characterize the microstructure in the observation area marked by hardness point. EDAX-OIM software is used to process the original data of EBSD.

### 3. Results and discussion

#### 3.1. Dilatometric curves and microstructure analysis

The dilatometric specimens were heated to 700 °C and 725 °C at 5 °C s\(^{-1}\) and held for 1.5 h, and then cooled to room temperature at 50 °C s\(^{-1}\), and the corresponding dilatometric curves are shown in figure 4. It can be seen that during the cooling process, there is an inflection point on the curve annealed at 725 °C, which means the occurrence of martensitic transformation. In comparison, there is no martensitic transformation in the specimen annealed at 700 °C.

The microstructure of the experimental steel after annealing at different intercritical temperatures is shown in figure 5. After hot rolling, the initial lath martensite structure of the experimental steel is obtained, without austenite and coarse precipitates, as shown in figures 5(a), and (b) is an enlarged view of the black rectangle in figure 5(a). There are many undissolved carbide particles in the matrix of the specimen annealed at 675 °C, which is consistent with the calculation result of TCFE7 in figure 1(a). With the increase of annealing temperature, the cementite disappears, and the proportion of intercritical austenite increases gradually. In the specimen annealed at 700 °C, the lamellar morphology of intercritical ferrite and austenite are alternately arranged, inheriting the hot-rolled lath martensite structure. In addition, some blocky structures appear in the specimen, as shown by the white circle in figure 5(d). As the annealing temperature rises to 725 °C, a small amount of lath martensite appears in the specimen except for ferrite and austenite. However, when the sample is annealed at 800 °C, almost all austenite is transformed into martensite after quenching to room temperature. The microstructure is composed of lath ferrite and lath martensite. Due to the higher isothermal temperature, the grains are coarsened.

XRD was used to detect retained austenite content in annealed specimens at different temperatures, and the results are shown in figure 6. Among them, the retained austenite content in the sample annealed at 675 °C is 7.1%, and that reaches the peak value of 17.9% in the sample annealed at 700 °C. Compared with the former,
The retained austenite content in the sample annealed at 725 °C is reduced to 14.7%, which is consistent with the SEM observation. When annealed at 675 °C, the proportion of austenite at this temperature is low due to the presence of partially undissolved cementite particles in the microstructure. When the annealing temperature rises to 700 °C, the higher isothermal temperature makes the cementite all dissolve and increases the diffusion rate of Mn element from ferrite to austenite, resulting in an increase in the volume fraction of austenite. When annealed at 725 °C, the proportion of intercritical austenite increases further, and the average content of C and Mn in austenite decreases continuously, as shown in figure 1(b), which leads to the decrease of the stability of austenite and the martensitic transformation during quenching [19], resulting in the reduction of the proportion of austenite at room temperature.

In order to verify the element content of austenite in the specimen of annealed at 700 °C and 725 °C, the average alloy composition of austenite in the two samples was qualitatively deduced by comparing the diffraction peaks of (200)γ and (311)γ, and according to the empirical formula of lattice constant. The lattice constant formula of austenite is as follows [20]:

$$\alpha_\gamma = 3.578 + 0.033 X_C + 0.00095 X_{Mn} + 0.0056 X_{Al} + 0.0031 X_{Mo} + 0.0018 X_V$$  (1)

Where $\alpha_\gamma$ is the lattice constant of austenite (in Å), $X_C$, $X_{Mn}$, and $X_{Al}$ are the mass percentages of C, Mn, and Al in austenite, respectively. It can be seen from figures 6(c) and (d) that the 2θ value of austenite diffraction peak in the specimen annealed at 725 °C is more significant than that of the sample annealed at 700 °C. According to the formula [21]:

![Figure 5. SEM micrographs of microstructures after different treatments, hot rolling and annealing at different intercritical temperatures (a) and (b) after hot rolling; (c) 675 °C; (d) 700 °C; (e) 725 °C; (f) 800 °C. The α, γ, α and θ represent ferrite, austenite, martensite, and cementite, respectively.](image)
Where, $\lambda$ is the wavelength, $h$, $k$, and $l$ are the crystal plane index, and $\theta$ is the diffraction peak angle. According to formula (2), the lattice constant of austenite in 725 °C annealed specimen is smaller than that in 700 °C annealed sample. Similarly, according to formula (1), it can be inferred that the content of alloying elements in austenite of the former is less than that of the latter.

Figure 7 TEM morphology of the specimen annealed at 700 °C. There are two kinds of retained austenite in the microstructure: blocky and lamellar, and the width of the lamellar austenite has an extensive distribution range, about 25 ~ 100 nm, as shown in figure 7(a). Because the morphology is one of the main factors affecting the mechanical stability of retained austenite, the TRIP effect of retained austenite with different stability occurs continuously during tensile deformation, which delays the occurrence of necking. Randomly select 3 points in the ferrite and austenite with different morphologies to analyze the energy spectrum of the Mn element and the results are shown in figure 7(a). The mass fraction of Mn in ferrite, blocky austenite and lamellar austenite are 2.7 wt%, 6.2 wt% and 7.3 wt%, respectively. The results show a strong Mn partitioning between austenite and ferrite during the intercritical annealing process, and the Mn content in lamellar austenite is higher than that in blocky austenite. This is because most of the lamellar structure is smaller than the blocky structure, which shortens the diffusion distance of Mn. From figure 7(b), it can be clearly seen that the parallel dislocation lines are arranged in the ferrite, and the dislocations are entangled at the grain boundary of ferrite and austenite. This is because when austenite grows along the adjacent lath ferrite, it causes a large local strain at the interface between ferrite and austenite. Therefore, the phase interface of the lamellar-like structure has a higher dislocation density. Figure 7(c) is a bright field image of nanoparticle precipitates in the ferrite. The size distribution range of the precipitates is between 8 and 16 nm. According to the energy spectrum analysis, these nano-scale precipitates dispersed in the ferrite are VC, as shown in figure 7(d).

### 3.2. Mechanical properties and work hardening behaviors

The engineering stress–strain curves are shown in figure 8(a). From the engineering stress–strain curve, it can be seen that a short yield plateau appears in the specimen annealed at 675 °C, and the curves of all other specimens show continuous yield characteristics, which is closely related to the microstructure of the sample at different
annealing temperatures. At a relatively high annealing temperature, the fraction of austenite in the microstructure increases, and the diffusion of solute atoms from ferrite to austenite is accelerated. As a result, the content of solid solution C atoms in ferrite is reduced, and it is difficult to interact with defects such as dislocation lines, showing continuous yield characteristics [22]. In addition, the serrated fluctuation appeared on the curves of 700 °C and 725 °C annealed specimens, which was caused by the discontinuous TRIP effect of retained austenite. The relevant research shows that [23, 24], the discontinuous TRIP effect is related to the following two reasons: (1) due to the volume expansion caused by martensite transformation during the tensile deformation process, the ferrite around the martensite is deformed, leading to local stress relaxation and transfer; (2) there are different levels of mechanical stability of the retained austenite, so the TRIP effect could only occur when the stress reaches a certain critical value.

The specimen annealed at 675 °C has the highest yield strength of 1125 MPa. The change of yield strength mainly comes from two factors. First, the high density of dislocations in the hot-rolled lath martensite matrix recovers continuously during the heating process. The higher the temperature, the fewer dislocations in the intercritical ferrite, and the more obvious the decrease in yield strength. On the other hand, in the temperature range of 675 °C to 725 °C, the microstructure of the experimental steel is mainly composed of intercritical ferrite and austenite. As the annealing temperature increases, the average Mn content in austenite decreases with the increase of the austenite ratio. At the same time, because the distribution of manganese in intercritical ferrite and austenite is not completely instantaneous, higher heat treatment temperature is conducive to the entire partition of Mn in the original martensite lath to the adjacent austenite. Therefore, the content of carbon and manganese in ferrite reduces correspondingly, weakening the solid solution strengthening effect in ferrite, which leads to the decrease of yield strength [22]. When the intercritical annealing temperature rises from 700 °C to 800 °C, the tensile strength increases with the increasing proportion of quenched martensite in the microstructure. The total elongation decreases continuously, which is consistent with the variation trend of volume fraction of retained austenite in the specimens. Finally, the experimental steel obtained excellent mechanical properties with tensile strength of 1152 MPa and total elongation of 29% when annealed at 700 °C.

The work hardening rate curves of the experimental steel at different annealing temperatures are shown in figure 8(b). It can be seen that, except for the sample annealed at 800 °C, the work hardening behavior of all samples can be divided into three stages. The first stage is the rapid decrease of the work hardening rate, which is mainly due to the dynamic recovery of dislocations in the ferrite [25]. The second stage is the obvious increase of the work hardening rate. At this stage, the retained austenite with the TRIP effect begins to transform into

![TEM micrographs of microstructures created by intercritical annealing at 700 °C](image)
martensite in a large amount, resulting in an obvious strengthening effect. In the third stage, as the strain increases, the amount of transformation of retained austenite decreases, resulting in a slow decline in the work hardening rate until the material fractures. In addition, due to the propagation of the PLC band, obvious stress serration flow appears on the work hardening curves at this stage of specimens annealed at 700 °C and 725 °C. It’s obvious that the specimens annealed at 700 °C begin to show an obvious TRIP effect at the true strain of about 0.02, and continue until the true strain is around 0.20. During the whole deformation process, the material has always maintained a work hardening value of 1000 ~ 6000 MPa, indicating that a higher proportion of retained austenite in the specimens could provide a strengthening effect continuously and prevent the appearance of necking.

3.3. Characterization of retained austenite during in situ deformation

In order to explore the differences in the stability of different morphology of austenite in the experimental steels and the evolution of intercritical ferrite during the plastic deformation process, the annealing samples at 700 °C were analyzed and characterized by discontinuous tensile test and in situ EBSD observation. According to the engineering stress-strain curve of regular tensile test, strains of 0%, 2%, 5%, and 14% were selected for in situ observation.

According to the results of in situ tensile tests in figure 9, the yield strength of the material increased significantly with the discontinuous tensile test, and was accompanied by the appearance of a yield plateau. The increase of yield strength can be explained by the Bausching effect, and the appearance of the yield plateau is related to the mean free path of dislocation motion. After the experimental steel is annealed, the ferrite retains the ultra-fine lath structure of the original hot-rolled martensite. Due to the first two tensile tests, part of the lath ferrite appears the dislocation cell structure, which further shortens the mean free path of movable dislocations. Therefore, in the early stage of the third tensile deformation, the propagation rate of dislocations is less than the extinction rate of dislocations. Similar to the traditional carbon and nitrogen interstitial atoms interacting with dislocations to form ‘Cottrell atmosphere’ and repeatedly ‘pinning and unpinning,’ the ultra-fine lath ferrite grain boundaries and finer dislocation cell structures form the repeated ‘obstruction and continued movement’ of movable dislocations, which is shown as the yield plateau period of the engineering stress-strain curve. Until the amount of plastic strain increases further, a higher density of dislocations is formed to maintain the macroscopic plastic deformation of the material. The higher density of dislocations makes the propagation rate of dislocations higher than the annihilation rate of dislocations, so greater external stress is needed to promote the further plastic deformation of the material [16].

Figure 10 shows the in situ EBSD characterization under different strains, where a1~a4 are the IQ maps of retained austenite (red code), ferrite (green code) and martensite (grayscale code). It can be seen that there are two types of retained austenite in steel. One is a slightly larger blocky retained austenite, which mainly distributes at the grain boundaries of the original austenite, and part of the austenite has a tendency to merge and grow, as shown in area 1 and area 3 in figure 10(a1). The other is a typical lamellar retained austenite, which mainly distributes between the original hot-rolled martensite, as shown in area 3.

Figures 10(b1)~(b4) and (c1)~(c4) are the KAM maps of the BCC and the FCC phases. The corresponding KAM statistics are shown in figure 11. With the increase of strain, the average KAM values of the BCC phase and FCC phase both increase continuously, indicating that the dislocation density of the specimen has been significantly increased. In addition, it can be seen from figure 10(c1) that some undeformed larger-sized austenite grains have higher fine linear KAM values. From the IQ map (a1) of the microstructure, it can be seen
that this is mainly due to the presence of some sub-grain boundaries in the undeformed larger-sized austenite grains. Combined with the annealing process of the experimental steel, it is believed that the formation of this sub-grain boundary originates from the remaining part of the grain boundary where austenite merges and grows at the interface during the intercritical annealing. According to the statistical results in figure 11(b), for the BCC phase, the total fraction of KAM in the range of $0 \sim 1$ decreases as the strain increases, while the total fraction of KAM in the range of $1 \sim 2$ and $2 \sim 3$ is increasing. Among them, after the second discontinuous tensile test, the KAM value changes in the three ranges are the most significant, which is related to the formation of a large amount ($10.2\%$) of strain induced martensite in this process. First, the high-density dislocations of martensite itself greatly increase the KAM value of the material. Secondly, the martensite transformation is accompanied by volume expansion, which leads to an increase in geometrically necessary dislocations in the surrounding ferrite. The combined effect of these two aspects leads to a substantial increase in the KAM value of the material at this stage. In the FCC phase, the total fraction of low-index KAM in the range of $0 \sim 1$ also decreases with the increase of strain, while the total fraction of KAM in the range of $1 \sim 2$ shows a trend of first increasing and then decreasing, and the ratio of KAM in the range of $2 \sim 3$ is basically constant. This indicates that the retained austenite of the FCC phase cannot exist stably with the increase of strain like the BCC phase. When the KAM value in the grain reaches a certain level, the retained austenite will be transformed into martensite under the action of stress.

Relevant studies have shown that [11–16] large-size blocky retained austenite has lower stability than lamellar retained austenite. The results of this experiment prove that after the first constant deformation tensile, the large-sized blocky austenite in area 1 begins to undergo martensitic transformation, while lamellar austenite hardly undergoes a transformation. At this time, the retained austenite content in the specimen dropped from the initial 22.9\% to 18.4\%. After the second constant deformation tensile, almost all the blocky austenite, including area 2 is transformed into martensite. At the same time, the lamellar austenite between the martensite laths also undergoes partial phase transformation, which further cuts the lamellar austenite into extremely small austenite particles, thus exhibiting extremely stable characteristics. Combining the normal orientation distribution map of the rolling surface of the FCC phase in figures 10$(d_1)$ and $(d_3)$, it can be seen that the grain orientation of the untransformed lamellar austenite in area 2 has changed, and the KAM value has increased significantly compared with the unaltered form.

4. Conclusions

In this research, the effect of annealing temperature on microstructure and mechanical properties of 3.5 wt\% Mn hot-rolled medium manganese steel was studied. The main conclusions are as follows:

(1) The microstructure of the experimental steel is mainly composed of ferrite and austenite with different morphologies. With the increase of annealing temperature, the thermal stability of austenite decreases, resulting in the appearance of quenched martensite in the specimens annealed at 725 ℃. When annealed at 700 ℃, the proportion of retained austenite reaches the peak value of 17.6\%.

Figure 9. Regular and in situ tensile stress-strain curves of specimens.
**Figure 10.** In situ EBSD characterization of specimens at different deformations of (a1, b1, c1, d1) 0%; (a2, b2, c2, d2) 2%; (a3, b3, c3, d3) 7% and (a4, b4, c4, d4) 21%. Retained austenite red code, ferrite green code and martensite grayscale code IQ maps (a1, a2, a3, a4). The KAM maps of BCC structures (b1, b2, b3, b4). The KAM maps of FCC structures (c1, c2, c3, c4). IPF maps (d1, d2, d3, d4) of the retained austenite rolling surface normal.

**Figure 11.** (a) RA fraction, average KAM value of FCC and BCC; (b) and (c) total fraction of KAM of BCC and FCC under different deformations.
(2) The tensile strength of 1152 MPa and the total elongation of 29% were obtained when the experimental steel was annealed at 700 °C. In addition, the retained austenite with different stability provides a continuous TRIP effect during the tensile process, which improves the work hardening value of the material and effectively inhibits the occurrence of necking.

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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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