Original Article

Adjusting the microstructure evolution, mechanical properties and deformation behaviors of Fe-5.95Mn-1.55Si-1.03Al-0.055C medium Mn steel by cold-rolling reduction ratio

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\textbf{A B S T R A C T}

For a representative medium Mn steel with the actual chemical composition of Fe-5.95Mn-1.55Si-1.03Al-0.055C (wt. %), the effect of cold-rolling deformation on microstructural evolution and mechanical properties was investigated systematically. The thickness of coarse $\delta$-ferrite grains decreases with the increase of cold-rolling reduction, and when the cold-rolling reduction ratio reaches up to a certain value, these $\delta$-ferrite grains can be broken into small pieces due to the severe plastic deformation. Additionally, a critical cold-rolling reduction ratio for recrystallization exists. Below this critical reduction value, the medium Mn steel after austenite reverted transformation (ART) annealing remains lath-shaped structure originating from the initial martensitic morphology, and when recrystallization occurs, however, submicron equiaxed grains dominate. The initial microstructure before ART annealing, which is usually determined by cold-rolling reduction, strongly influences not only the martensitic/ferritic matrix, but also reverted austenite grains. Non-recrystallization matrix promotes the formation of acicular reverted austenite, whereas recrystallization forces the austenite grains spherical and promotes the grain size of austenite homogenizing. Under the situation of non-recrystallization, the cold-rolling reduction prior to ART annealing only has a negligible effect on the final mechanical properties. However, the occurrence of recrystallization results in not only the yielding plateau, i.e., discontinuous yielding, but also the remarkable increase of yield strength.

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1. Introduction

In recent years, automobile lightening is bringing higher demands for the development of advanced high strength steels (AHSS) [1–4]. However, due to the insufficient mechanical properties for 1st generation AHSS and high cost of alloying elements for 2nd generation AHSS [5–7], the development of 3rd generation AHSS with both low-cost and superior mechanical properties is one of the urgent requirements. Since medium Mn steels with 3–10 wt.% Mn content show an excellent combination of strength and ductility, it is being considered as one of the most promising candidates of AHSS to meet the increasing requirement for automobile lightening. The reason why medium Mn steels often have a high product of tensile strength and total elongation (PSE) is that metastable austenite included in the microstructure of medium Mn steels can transform into martensite during deformation, i.e., transformation induced plasticity (TRIP) effect [8,9], which can retard necking due to a localized work hardening.

A lot of research works on medium Mn steels have been carried out in the past decade. Gibbs et al. [10] reported that tensile strength and total elongation of a Fe-7.1Mn-0.1C steel annealed at 600 °C for 1 week were 876 MPa and 42 %, respectively. Shi et al. [11] investigated four steels with nominal chemical composition of Fe-5–7Mn-0.2–0.4C, and it was found that after an annealing at 650 °C for 6 h, these steels can obtain the tensile strength of 950–1420 MPa and total elongation of 31–44.5 %. Suh et al. [12] first proposed to add Al element in medium Mn steels in order to raise the ART-annealing temperature above 700 °C and shorten annealing time to 2–3 min so that it can adapt to continuous annealing (CA) process. Based on this, Cai et al. [2] developed an Al-added medium Mn steel, i.e., the Fe-11Mn-4Al-0.2C alloy, which exhibited surprisingly good tensile strength and total elongation as 1007 MPa and 65 %, respectively. In terms of microalloying design, Hu et al. [13] reported that a V-microalloyed medium Mn steel enabled the fact that VC particles precipitated in δ-ferrite and contributed to enhance yield strength. Song et al. [14] newly developed the Cu-containing medium Mn duplex lightweight steels, and later some other research works were performed on the Cu-containing medium Mn steels as well [15,16]. Cai et al. [17,18] tried to add microalloying elements, Mo and Nb, in medium Mn steels in order to improve yield strength. But to sum up, most previous works like these particularly stressed on the effect of either heating treatment parameters [19–23] or the chemical composition design [24,25] on microstructure and properties.

In the previous research, it has been found that the microstructure and mechanical properties of medium Mn steels are sensitive to the initial microstructure before ART annealing [26,27], which usually depends on the rolling process. However, much less attention has been placed on the influence of cold-rolling parameters on microstructure and performance of medium Mn steels so far [28,29]. Thus, in this work, we intend to investigate the effect of cold-rolling deformation on microstructural evolution, mechanical properties and deformation behavior of medium Mn steel systematically.

**Table 1** Chemical compositions of the medium Mn steel in experiments (wt.%).

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Al</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.055</td>
<td>1.55</td>
<td>5.95</td>
<td>1.03</td>
<td>0.005</td>
<td>0.001</td>
</tr>
</tbody>
</table>

![Fig. 1 – Schematic illustration of the thermomechanical process used in the present study.](image)

2. Experimental procedure

The chemical composition of the investigated medium Mn steel is shown in Table 1. Based on the calculation using Thermo-Calc software with the TCFE6 database, the AE1 and AE3 temperatures of the investigated steel are about 431 °C and 889 °C, respectively. This alloy was smelted in a vacuum furnace, and the ingot was hot-forged into a slab with a cross-section dimension of 60 × 100 mm. The slabs were hot-rolled into 2.5 mm in thickness after reheating to 1200 °C for 2 h, and then held at 600 °C for 1 h followed by furnace-cooled to room temperature for the coiling simulation. By cold rolling, the hot-rolled sheets were further thinned to a final thickness of 2.25 mm and 0.75 mm with a thickness reduction of 10 % and 70 %. The tensile specimens with a 25 mm gauge length (according to the ASTM-E8 sub-size standard) parallel to the rolling direction were then machined from the cold-rolled plates. These tensile specimens were directly annealed under a fixed condition of temperature and time, that is, at 700 °C for 3 min, for continuous annealing simulation. The full thermomechanical process is illustrated in Fig. 1. For brevity, each specimen was given with a sample ID, for example, the specimen cold-rolled with 50 % reduction and then annealed at 700 °C for 3 min was named as CR50-700-3, and so on.

The microstructure of the samples was etched using a 4 % nitral etch after mechanical polishing. The optical microscope (OM) photographs were obtained using Leica DMRM optical microscope. The secondary electron (SE) micrographs were obtained using Zeiss Ultra 55 scanning electron microscopy (SEM), and reverted austenite grains were characterized by electron backscatter diffraction (EBSD) techniques on this machine at 15 kV by a spatial step size of 0.05 μm. Channel 5 software was used to collect and index the Kikuchi band pat-
terns. The microscopic distribution of chemical elements in the representative sample was characterized by a JXA-8530F electron probe micro-analyzer (EPMA). In order to determine the amount of reverted austenite in each sample, X-ray diffraction (XRD) analysis was performed on a D/Max2400 analyzer using Cu Kα radiation operating at 50 kV and 150 mA. Patterns were taken in the 2θ range from 40° to 100° with a (2θ) scanning speed of 4°/min. The integrated intensities of the (200) γ, (220) γ, (311) γ, (200) α, and (211) α peaks were calculated to quantify the amount of austenite by the following method [30]:

\[ V_γ = \frac{1}{1 + C(I_α/I_γ)} \]

where \( V_γ \) is the volume fraction of retained austenite for each peak, \( I_α \) and \( I_γ \) is the integrated intensity of ferrite and austenite peaks, G-value for each peak was used as follow, 2.5 for \( I_α \) (200)/ \( I_γ \) (200), 1.38 for \( I_α \) (200)/ \( I_γ \) (220), 2.02 for \( I_α \) (200)/ \( I_γ \) (311), 1.19 for \( I_α \) (211)/ \( I_γ \) (200), 0.06 for \( I_α \) (211)/ \( I_γ \) (220), 0.96 for \( I_α \) (211)/ \( I_γ \) (311). As for the estimation of dislocation density, the XRD profiles were taken using XPert Pro MRD diffractometer with Cu Kα radiation operating at 40 kV and 40 mA. The diffractograms were recorded from 40° to 100° with a (2θ) scanning speed of 1.5 °/min. The modified Williamson-Hall equation (MWH) [31,32] was used to estimate the dislocation density of several samples, which will be detailed in Section 3.4. The samples for EBSD and XRD measurement were first mechanically ground and then electro-polished using 700 mL CH₃OOH +200 mL HClO₄ solution.

Mechanical performance testing was carried out on a CMT-5105 tensile machine with a crosshead displacement rate of 3 mm-min⁻¹ (i.e., corresponding to a nominal strain rate of 2.0 × 10⁻³ s⁻¹). The gauge length of the extensometer was 25 mm.

3. Results and discussion

3.1. Microstructural characterization of cold-rolling plates

Fig. 2 shows the microstructure of the cold-rolled steel with different rolling reduction in thickness through optical microscope. There are numerous δ-ferrite grains which are coarse and fibrous along the rolling direction (RD), as shown in Fig. 2a-c. The formation of coarse δ-ferrite grains in medium Mn steels is usually attributed to inheritance from as-cast microstructure due to non-equilibrium cooling during solidification. In present study, it is worth noting that the cold-rolling reduction affects the δ-ferrite morphology. When the cold-rolling reduction ratio is less than 50 %, the average thickness of δ-ferrite grain should decease with the cold-rolling reduction increasing. However, when the cold-rolling reduction ratio reaches up to 70 %, these coarse δ-ferrite grains are broken into small pieces due to the severe plastic deformation, as indicated in Fig. 2d. In addition to δ-ferrite, martensite (or containing small amount of bainite) is the principal part of the microstructure because of the good hardenability. With the cold-rolling reduction increasing, martensitic matrix is twisted and deformed more and more seriously.

Fig. 3 shows SEM micrographs and the corresponding kernel average misorientation (KAM) maps by EBSD examination.
for the cold-rolled samples with different rolling reductions in thickness. The coarse δ-ferrite grain was followed with interest as indicated by the dotted lines. Since KAM map is usually considered to highlight regions of higher microstrain caused by crystal defects, such as dislocations [33,34], it can be seen that under the cold-rolling reduction of 10%, regions with relatively higher KAM values are mainly martensitic matrix as shown in Fig. 3b. Within the fibrous δ-ferrite region, local high microstrain concentrates around grain boundaries (indicated by the black arrows). Moreover, with the cold-rolling reduction increasing, microstrain gradually accumulates in δ-ferrite grains. Since the δ-ferrite grain is soft phase, its deformation feature in medium Mn steels during cold-rolling process can be attributed to the good deformation compatibility.

XRD examination was carried out on both hot-rolled and cold-rolled samples, and in Fig. 4 XRD spectrums of these samples are displayed. Only the XRD spectrum of the hot-rolled sample shows obvious austenitic peaks, and the amount of retained austenite in hot-rolled steel is about 15.1 vol.% through quantitative calculation based on the above-mentioned method. Due to lack of austenitic peaks in other XRD spectrums, the content of retained austenite cannot be determined. However, it can be confirmed that in spite of the merely 10% cold-rolling reduction, the content of retained austenite can be reduced to a quite low level, which is below the limitation of XRD measurement.

3.2. Microstructural characterization after ART-annealing

Fig. 5 shows the microstructure of ART-annealed samples with different cold-rolling reductions prior to annealing through SEM. We were not surprised to find that the morphology of δ-ferrite grains remains the same with that before ART annealing in different samples. As for other microstructure, however, there is remarkable difference among these samples. The CR10-700-3 and CR20-700-3 samples, of which the cold-rolling reduction prior to ART annealing is merely 10% and 20%
respectively, present lath-shaped structure originating from the initial martensite morphology due to only slight deformation of cold rolling. When the cold-rolling reduction prior to ART annealing reaches up to 30%, the submicron equiaxed grains dominate in the CR30-700-3 sample instead of lath-shaped structure. With the further increase of cold-rolling reduction prior to ART annealing, the CR50-700-3 and CR70-700-3 samples are mainly composed of submicron equiaxed grains besides δ-ferrite grains. Apparently, these submicron equiaxed grains form from recrystallization of deformed matrix. Thus, according to the observation of microstructure, there is a critical reduction ratio of cold rolling for recrystallization, above which recrystallization occurs. In the case of the present studied steel, the critical reduction ratio of cold rolling for recrystallization is about 30%. In addition, as indicated by the yellow arrows in Fig. 5c, several granular grains are observed within δ-ferrite grains, for which it is impossible to occur solid phase transformation during annealing. These granular grains are a little weird; especially in the SEM micrograph each submicron grains cannot be determined as a certain phase for the studied steel. Thus, another characterization method was employed to identify them, which will be specified in Section 3.3. Moreover, as shown in Fig. 6, the distribution of elements is obtained through EPMA. The mapping result indicates that δ-ferrite grain is the region which is rich in Al element but depleted in C and Mn elements.

XRD technique was also used to quantify the amount of reverted austenite in these annealed samples. Fig. 7a shows the XRD spectra, in which austenitic diffraction peaks are captured clearly for each sample. The exact volume fractions of reverted austenite in each sample are summarized in Fig. 7b according to the calculating method mentioned above. The amount of reverted austenite in annealed samples are about 21.0% except for the CR10-700-3 sample, in which the volume fraction of reverted austenite is estimated as 23.2%. It can be assumed that the highest content of reverted austenite among these annealed samples should be attributed to the higher amount of retained austenite nucleus surviving from the minimum cold-rolling reduction (10%) though they cannot be detected through XRD examination as shown in Fig. 4.

3.3. Features of metastable austenite resulting from the initial microstructure prior to ART annealing

As shown in Fig. 8, EBSD technique was used to characterize the reverted austenite grains, and then the effect of the initial microstructure on characteristics of reverted austenite grains will be summarized and discussed. In the case of the CR10-700-3 and CR20-700-3 samples, since the matrix remains lath-like martensitic morphology due to the relatively small cold-rolling reduction, the reverted austenite grains which are mainly distributed between martensitic laths are acicular as marked by the black arrows. Besides, the reverted austenite grains with blocky shape are mainly located along martensitic packet boundaries or prior austenite grain boundaries, as indicated by the white arrows. As for the CR50-700-3 and
CR70-700-3 samples, however, almost all the reverted austenite grains are near-spherical. This should be attributed to the full recrystallization of martensitic matrix originating from the severe plastic deformation prior to ART annealing. Accordingly, the initial microstructure before ART annealing, which is determined by cold-rolling reduction, strongly influences not only the martensitic/ferritic matrix, but also the condition of reverted austenite grains. The CR30-700-3 sample, however, contains reverted austenite grains with all kinds of abovementioned features due to partial recrystallization of deformed martensitic matrix.

Table 2 summarizes the grain size (GS) and average aspect ratio of grains in these ART-annealed samples subjected to different cold-rolling reductions prior to annealing. The grain size is evaluated using the equivalent grain diameter in the present work. Statistically, the average GS of reverted austenite in all the annealed samples is below 300 nm, and these values for the CR50-700-3 and CR70-700-3 samples are larger than that of other samples. Nevertheless, the maximum GS of reverted austenite in CR10-700-3 and CR20-700-3 samples reaches up to 1076 nm and 720 nm respectively, which is significantly larger than that of other samples. In addition, average aspect ratio is used to evaluate the change of reverted austenite morphology with processing technology. It can be seen that with the increase of cold-rolling reduction prior to ART annealing, the average aspect ratio of reverted austenite in the annealed samples is decreased from 2.32 to 1.60. And it is quite worth noting that the variations of the average aspect ratio between reverted austenite and martensite/ferrite with cold-rolling reduction prior to ART annealing accord with each other very well, as displayed in Table 2. The statistics above reflect that whether the recrystallization of deformed matrix occurs or not and the degree of recrystallization influence both of the GS and morphology of reverted austenite grains. So to
Fig. 8 – Combination of IQ map and austenitic phase (red) map of ART-annealed samples with different cold-rolling reductions prior to annealing by EBSD analysis: (a) 10 %, (b) 20 %, (c) 30 %, (d) 50 % and (e) 70 % (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

### Table 2 – Grain features of ferrite and austenite in the ART-annealed samples with different cold-rolling reductions prior to annealing based on EBSD detection.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Martensite/ferrite</th>
<th>Reverted austenite</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Average GS, nm</td>
<td>Average aspect ratio</td>
</tr>
<tr>
<td>CR10-700-3</td>
<td>441</td>
<td>2.31</td>
</tr>
<tr>
<td>CR20-700-3</td>
<td>471</td>
<td>1.93</td>
</tr>
<tr>
<td>CR30-700-3</td>
<td>415</td>
<td>1.85</td>
</tr>
<tr>
<td>CR50-700-3</td>
<td>335</td>
<td>1.80</td>
</tr>
<tr>
<td>CR70-700-3</td>
<td>303</td>
<td>1.71</td>
</tr>
</tbody>
</table>

summarize, non-recrystallization matrix due to small cold-rolling reduction promotes the formation of acicular reverted austenite besides the blocky austenite with large size, whereas recrystallization due to heavy cold-rolling reduction forces the austenite grains spherical and promotes the GS of austenite homogenizing.

The IPF map of reverted austenite grains superimposed on the IQ map demonstrates the crystal orientation of each grain. In the CR10-700-3 and CR20-700-3 samples, the local consistency of crystal orientation of reverted austenite grains is easily noticed as indicated by the white circles in Fig. 9a and b. This should be attributed to the fact that the orientation of reverted austenite grain inherits from the prior austenite grains in the absence of recrystallization [26]. Thus, as for the CR30-700-3 sample, there are also some reverted austenite grains sharing the same or similar crystallographic orientation in certain non-recrystallization region (see the white circle). In the case of the CR50-700-3 and CR70-700-3 samples, full recrystallization enables more random distribution of austenite orientation, the same as within the recrystallization regions in the CR30-700-3 sample. Since whether recrystallization occurs or not depends on the initial microstructure to some extent, the cold-rolling reduction prior to ART annealing affects the crystal orientation of reverted austenite grains as well.

3.4. Estimation of dislocation density in the cold-rolled and ART-annealed samples

Dislocation densities of several samples were estimated using modified Williamson-Hall (MWH) method [31,32]. The MWH equation is written as follows:

\[ \Delta K = \frac{0.9}{D} + bM \sqrt{\frac{\pi}{2P}} \left( K\tilde{c}^{1/2} \right) \]  

(2)

where \( K = 2\sin\theta/\lambda \), \( \Delta K = \cos\theta(2\theta)/\lambda \). Here, \( \Delta 2\theta \), \( \theta \) and \( \lambda \) represent the full width half maximum (FWHM) of the diffraction peak, diffraction angle and wavelength of the X-ray, respectively. For Cu radiation, the value of \( \lambda \) is 0.154059 nm. \( D \) and \( b \) are the average grain size, dislocation density and the magnitude of the Burgers vector of dislocations, respectively. \( M \) is a dislocation distribution parameter depending on the effective outer cut-off radius of dislocation, and in the present investigation, the value of \( M = 2 \) is used. The average contrast factor of the dislocations, \( \tilde{c} \), is calculated by

\[ \tilde{c} = \tilde{c}_{h00}(1 - qH^2) \]  

(3)

where \( \tilde{c}_{h00} \) is the average contrast factor corresponding to h00 reflection, and the value of \( \tilde{c}_{h00} = 0.266 \) was used assuming...
that edge and screw dislocations are present in equal proportion [35,36]. \( H^2 \) is represented as:

\[
H^2 = \frac{h^2k^2 + h^2l^2 + k^2l^2}{(h^2 + k^2 + l^2)^2}
\]  

(4)

where \( h, k, l \) are the Miller’s indices of each peak. In the present work, the XRD diffraction peaks used for this estimation were \((110)\), \((200)\), \((211)\) peaks. Eq. (2) can be converted into the following form:

\[
\Delta K \equiv \alpha + \beta K^{1/2}
\]  

(5)

In order to obtain the value of \( \alpha \), the \( \Delta K \) for each \([hkl]\) peak was plotted as a function of \( K \), and the linear fitting \((\Delta K \text{ versus } K)\) gives \( \alpha \) as the intercept on the \( \Delta K \) axis, as shown in Fig. 10a. Eq. (5) can be transformed into the quadratic form and Eq. (3) is substituted, thus the following equation is obtained:

\[
(\Delta K - \alpha)^2/K^2 \equiv \beta^2 C_{600}(1 - qH^2)
\]  

(6)

On the basis of Eq. (6), from a linear relation between \((\Delta K - \alpha)^2/K^2\) and \( H^2 \), the parameter \( q \) can be derived as an inverse of the intercept on the \( H^2 \) axis, as shown in Fig. 10b. Then, based on Eq. (5), fitting \( \Delta K \) against \( K^{1/2} \) as a linear relation (see Fig. 10c), the slope of the fitted curve is used as the value of \( \beta \). Finally, the dislocation density is estimated from the following equation, assuming the value of \( b \) is 0.252 nm in ferrite:

\[
\rho = \frac{2\beta^2}{nM^2b^2}
\]  

(7)

Table 3 gives the estimation of dislocation density of several representative samples. It can be seen that as for the cold-deformed samples, dislocation density increases with cold-rolling reduction. After the same annealing treatment, the dislocation density in both the CR20-700-3 and CR70-700-3 samples decreases when compared with that of cold-deformed state. However, the dislocation density of the CR70-700-3 sample is still higher than that of the CR20-700-3 sample though the former experienced recrystallization, which is usually considered to lead to a dramatic annihilation of dislocation.

\# \( \rho_D \) represents the dislocation density of the cold-rolled sample; \( \rho_A \) represents the dislocation density of the ART-annealed sample.

### 3.5. Mechanical properties after ART annealing

After the cold-rolled samples with the rolling reduction ranging from 10 % to 70 % were intercritically annealed at 700 °C for 3 min, the mechanical properties of these annealed samples were examined by uniaxial tensile test at room temperature. Fig. 11 shows the engineering stress–strain curves of the ART-annealed samples, and the local details of the initial stage of plastic deformation are amplified in the inset. When the cold-rolling reduction ratio prior to ART annealing is not above 20 %, the samples show continuous yielding because there is no phenomenon of yield point elongation. However, when the cold-rolling reduction ratio prior to ART annealing ranges from 30 % to 70 %, the samples have a remarkable yielding plateau, i.e., Lüders strain, which indicates a discontinuous yielding behavior. Moreover, it is worth noting that with the increase of cold-rolling reduction prior to ART annealing, the Lüders strain increases from 0.38 % to 3.69 %, as shown in Table 4. It was reported that martensite has an inherently high ratio (18 %) of mobile screw dislocations from displacive shear transformation [37,38]. The CR10-700-3 and CR20-700-3 samples without recrystallization may inherit the high ratio of mobile dislocations, which can promote continuous yielding. Other

![Fig. 9 – Combination of IQ map and inverse pole figure (IPF) of the reverted austenite of ART-annealed samples with different cold-rolling reductions prior to annealing by EBSD analysis: (a) 10 %, (b) 20 %, (c) 30 %, (d) 50 % and (e) 70 %.

![Table 3 – Dislocation density of the representative cold-rolled and ART-annealed samples.](image)
samples due to recrystallization may have quite low ratio of mobile dislocations, which results in discontinuous yielding. The mechanical properties of these samples are summarized in Table 5. The yield strength ranges from 715 MPa to 883 MPa, and the tensile strength ranges from 895 MPa to 1002 MPa. The total elongations of the samples are all above 25 %, and the maximum elongation and PSE are respectively 29.1 % and 26.7 GPa% for the CR50-700-3 sample. Moreover, the yield-to-tensile ratio of the CR50-700-3 sample is 0.91, which is the highest value among these samples. For the automotive structural components, high yield strength benefits to the improvement of intrusion resistance during crash of the vehicles.

The mechanical properties of the annealed samples with cold-rolling reduction prior to ART annealing are plotted in Fig. 12a and b. According to the microstructural characterization mentioned above, Fig. 12a and b are divided into three zones marked by different colors. Yellow represents the non-recrystallization zone, blue represents the recrystallization zone, and grey represents the transition zone from non-recrystallization to recrystallization. Under the situation of non-recrystallization, cold-rolling reduction prior to ART annealing only has little effect upon the mechanical performance of the investigated steel. It can be seen that the increase of cold-rolling reduction prior to ART annealing from 10 % to 20 % just results in the fact that the yield strength and tensile strength decrease slightly and the total elongation increases a little. Under the situation of recrystallization, however, the mechanical properties vary more dramatically. Both yield strength and tensile strength increase with the cold-rolling reduction prior to ART annealing remarkably. This can be explained by the fact that the average grain size of ferrite in the ART-annealed samples decreases with the cold-rolling reduction prior to ART annealing when recrystallization occurs, as displayed in Table 2. With the increase of cold-rolling reduction, the stronger plastic deformation can provide more nucleation sites for recrystallization, which accounts for the finer ferrite grains. In addition, relatively high dislocation density, as mentioned above, may contribute to the increase of strength as well [38]. However, the total elongation is not monotone with the cold-rolling reduction prior to ART annealing, and the peak of elongation is obtained when the cold-rolling reduction ratio is at 50 %. For the transition from non-recrystallization to recrystallization, yield strength, which is more sensitive than tensile strength, increases significantly. This can also be attributed to the grain refinement due to recrystallization based on the statistical data of grain size as well, as shown in Table 2. In other word, recrystallization results in not only the above-mentioned yielding plateau but also the remarkable increase of yield strength. According to the estimation of dislocation density in Section 3.4, however, Lüders strain should not be mainly attributed to the low dislocation density due to the occurrence of recrystallization. More direct and important reason for Lüders strain in medium Mn steels should be explored thoroughly in the future research.

Table 4 – The Lüders strain of the ART-annealed samples with different cold-rolling reduction ratios prior to ART annealing.

<table>
<thead>
<tr>
<th>Cold-rolling reduction ratio, %</th>
<th>10</th>
<th>20</th>
<th>30</th>
<th>50</th>
<th>70</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lüders strain, %</td>
<td>0</td>
<td>0</td>
<td>0.38</td>
<td>0.58</td>
<td>3.69</td>
</tr>
</tbody>
</table>

4. Conclusion

(1) The thickness of coarse δ-ferrite grains decreases with the increase of cold-rolling reduction, and when the cold-rolling reduction ratio reaches up to a certain value (70 % for the steel in the present work), these δ-ferrite grains can be broken in to small pieces due to the severe plastic deformation.
Table 5 – Mechanical properties of the ART-annealed samples with different cold-rolling reduction ratios prior to ART annealing.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Rp0.2 or Rm (MPa)</th>
<th>Rm (MPa)</th>
<th>Yield-to-tensile ratio</th>
<th>A (%)</th>
<th>PSE (GPa%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CR10-700-3</td>
<td>724</td>
<td>909</td>
<td>0.80</td>
<td>25.5</td>
<td>23.1</td>
</tr>
<tr>
<td>CR20-700-3</td>
<td>715</td>
<td>895</td>
<td>0.80</td>
<td>25.9</td>
<td>23.2</td>
</tr>
<tr>
<td>CR30-700-3</td>
<td>768</td>
<td>897</td>
<td>0.86</td>
<td>27.3</td>
<td>24.5</td>
</tr>
<tr>
<td>CR50-700-3</td>
<td>836</td>
<td>916</td>
<td>0.91</td>
<td>29.1</td>
<td>26.7</td>
</tr>
<tr>
<td>CR70-700-3</td>
<td>883</td>
<td>1002</td>
<td>0.88</td>
<td>25.2</td>
<td>25.3</td>
</tr>
</tbody>
</table>

(2) According to the observation of microstructure, there is a critical cold-rolling reduction ratio (20–30 % for the investigated steel) for recrystallization, above which recrystallization occurs. Under the situation of non-recrystallization, the samples will remain lath-shaped structure originating from the initial martensite morphology. When recrystallization occurs, however, the samples will mainly composed of submicron equiaxed grains besides δ-ferrite grains.

(3) The initial microstructure before ART annealing, which is determined by cold-rolling reduction, strongly influences not only the martensitic/ferritic matrix, but also reverted austenite grains. Based on the statistics of the average grain size and aspect ratio of reverted austenite, non-recrystallization matrix due to small cold-rolling reduction promotes the formation of acicular reverted austenite besides the blocky austenite with large size, whereas recrystallization due to heavy cold-rolling reduction forces the austenite grains spherical and promotes the grain size of austenite homogenizing.

(4) Below the critical reduction ratio for recrystallization, the cold-rolling reduction prior to ART annealing only has a negligible effect on the final mechanical properties. However, the occurrence of recrystallization results in not only the yielding plateau, i.e., discontinuous yielding, but also the remarkable increase of yield strength, which can be attributed to the grain refinement due to recrystallization. Based on the estimation of dislocation density, however, Lüders strain should not be mainly attributed to the low dislocation density due to the occurrence of recrystallization.

Fig. 12 – Variations in mechanical properties of the ART-annealed samples with different cold-rolling reductions prior to annealing: (a) tensile strength, yield strength and yield ratio, and (b) total elongation and product of tensile strength and total elongation (PSE).

Conflict of interest

The authors declare no conflicts of interest.

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