MICROSTRUCTURE EVOLUTION IN COLD ROLLED MEDIUM MANAGANESE STEEL DURING TWO STEP INTERCRITICAL ANNEALING

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ABSTRACT

Microstructure evolution during two step intercritical annealing of 0.1C-5Mn cold rolled Steel processed by austenisation at 650°C with different holding time was examined by SEM, TEM and XRD. The experimental results indicate that the TRIP steel with Mn of 5% could form a considerable amount of retained austenite with good TRIP effect. The evolution of microstructure during the second annealing was analyzed. Austenite reverted transformation (ART) was observed during Intercritical annealing. It was shown that a complex matrix microstructure composed of three phases (retained austenite/martensite/ferrite) was formed and two types of morphologies were detected (lath like and polygonal). Furthermore, a high volume fraction of retained austenite (22%), which was stabilized at room temperature, was the origin of a TRIP effect. A good balance between strength and ductility can be achieved by optimizing the heat treatment.

Keywords: retained austenite, Transformation induced plasticity (TRIP), intercritical annealing and microstructure evolution.

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1. INTRODUCTION
During last few years, the automobile industry has shifted its focus to the design of light, fuel efficient and environmentally friendly vehicles, which lead to multi-phase steels with the right balance of strength and ductility [1-4]. Transformation induced plasticity (TRIP) steel have small amount of retained austenite into martensite during plastic deformation, having excellent combination of strength and elongation and retained austenite plays an important role in controlling the mechanical properties of TRIP steel [5-8]. Mn, being an austenite stabilizer, expanded the austenite area and lowers the temperature of (Martensite start temperature) which could decrease about 30°C adding Mn of 1% [9]. Mn plays an active role in increasing the volume fraction of retained austenite and also induce significant solid solution hardening [10]. Thus a new type C-Mn steels with medium manganese content (from 3wt% to 9wt%) were reported to be capable of producing substantially improved mechanical properties with strength high up to 1 GPa and total elongation high up to 30%. This excellent combination of strength and ductility is remarkably superior to that of the conventional TRIP steels and is comparable with that of the TWIP steels. In that case, the designed steels were first austenized at relative high temperature (above Ac1) and then followed by long time intercritical annealing (between Ac1 and Ac3) to develop ultrafine austenite and ferrite duplex structure with austenite fraction about 30%–40% [11]. The second step of intercritical annealing at 650°C process is called austenite reverted transformation annealing (ART-annealing), which can obtain large amount of metastable austenite to produce high strength and ductility [12-14]. It was well known that the formation and development of metastable austenite were strongly dependent on the heat treatment temperature and time. A two-step annealing was applied to forged steel with Mn content of 5–7wt%. First the steel was soaked in the austenitic domain and quenched, thus a fully martensitic structure was obtained at room temperature. Then a second intercritical annealing followed by water cooling permitted to stabilize fractions of retained austenite. In this work the second annealing concept was applied to a 0.092%C–4.7%Mnwt%,cold rolled steel. Cold rolling reduction will decrease the austenite grain size in the following austenitization annealing, thus it should probably affect austenite reverse transformation in the final treatment. The objective was to study the evolution of microstructure, especially retained austenite fraction, as a function of intercritical annealing time.

Table 1 Composition of investigated steel in wt%.

<table>
<thead>
<tr>
<th>X10 Mn 4.7</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Al</th>
<th>P</th>
<th>S</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>0.09</td>
<td>4.7</td>
<td>0.006</td>
<td>0.001</td>
<td>0.005</td>
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2. EXPERIMENTAL PROCEDURE
Vacuum induction melting was used to prepare the steel. The chemical composition of the studied steel is shown in Table 1. Then, the ingot was reheated to 1200 °C and hot rolled with the finishing temperature around 930 °C. Coiling was simulated by a slow cooling in the furnace from 625 °C. Afterward, 70% of reduction was done to get the final thickness of cold rolled steel at about 1 mm. Then, the specimens were submitted to the second annealing schematized in Fig. 1.
Microstructure Evolution in Cold Rolled Medium Managanese Steel during Two Step Intercritical Annealing

![Diagram of thermal cycle](image)

**Figure 1** Scheme of the thermal cycle used in the present study.

It consists of two thermal cycles. First, a complete austenitization at 750°C for 30 minutes followed by water quench. Second, an intercritical annealing at 650°C with different holding times (3min, 10min, 30min, 1h, 2h, 3h, 7h, 10h, 20h and 30h) followed by water quench in the end. All double annealing treatments were performed in furnace under Ar atmosphere to avoid any decarburization. The mean heating rate in the furnace was about 5°C/s.

The microstructure of resulting samples was observed using the Optical Microscope (OM) and Field Emission Gun Scanning Electron Microscope (FEG-SEM) after revealing it with different etchings (Dino, Marshall, Picral, 2%Nital). Characterization of different phases and Energy Dispersive X-ray spectroscopy (EDX) analysis were also done in the transmission electron microscope (TEM) using thin foils. In order to prepare a thin foil the sheet was ground mechanically to approximately 80 µm then twin-jet polished in a solution of 5% perchloric and 95% acetic acids at about15°C. The X-ray diffraction (XRD) with CoKα radiation was applied in order to estimate the fraction of retained austenite. The calculation was done using the integrated intensities of (2 2 0)\(\alpha\), (2 1 1)\(\alpha\), (2 0 0)\(\alpha\) and (2 0 0)\(\gamma\), (2 2 0)\(\gamma\), (3 1 1)\(\gamma\) reflections. Electron Probe Micro Analyser (EPMA) was used in order to build the quantitative chemical maps of Mn after different steps of steel processing: cold rolling, first annealing and second annealing.
3. RESULTS

3.1. MICROSTRUCTURE EVOLUTION
The microstructure after first annealing cycle (austenitization followed by quench) was characterized using different techniques: optical microscope (OM), scanning electron microscope (SEM), transmission electron microscope (TEM) and X-ray diffraction (XRD). It can be seen that the resulting microstructure is composed of lath martensite. The figure 2 shows the microstructure after first annealing cycle with the help of optical microscope (OM), SEM image and TEM image on thin foils.

Figure 2 Characterization of the martensite present in the microstructure after first annealing cycle: (a) OM image after Dino etching (b) SEM image after Metabisulfite etching; (c) TEM image obtained on thin foil. (d) TEM image of retained austenite.

It was found that it consists of martensite matrix with some small quantity of retained austenite.

3.2. MICROSTRUCTURE AFTER SECOND ANNEALING TREATMENT
Figure 3 shows the microstructures obtained after different intercritical holding times. Using OM (Figure 3A and C) it is possible to get macro vision in order to define the global scenario of the microstructure evolution during second annealing. As it can be seen the microstructural constituents have ultra-fine size even after 30 h holding. The detailed analysis was done using the SEM-FEG (Fig 3B and D). These examinations suggested that the microstructure consists of three components: ferrite, martensite and retained austenite. From the deeper observations using OM and SEM-FEG it could be seen that these components have two types of morphology: polygonal and agglomerates of lath-like elements. The lath like agglomerates appers as bands in the microstructure having higher value of Mn content in the region of micro segregations. It can be also noticed that with the increase of holding time the fraction of lath like agglomerates decreases and the polygonal features became coarser.
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**Figure 3** Microstructure of samples annealed at 650°C for 7 hr (A and B) and 30 hr (C and D) revealed with Dino etching: (A and C) OM Images (white colour corresponds to ferrite and black and brown – to martensite and/or retained austenite): (B and D) SEM Images (uniform grey colour corresponds to ferrite and rough (heterogeneous) grey areas are martensite and/or retained austenite).

The presence of three constituents in the microstructure and their two types of morphology were also confirmed by TEM observations. Figure 4 shows the microstructure of the sample annealed for 7h at 650°C. Figure 4A and B reveals clearly the existence of ultrafine features (less than 1µm) and coarser components with the size of about several microns. Figure and D identifies the phases present in the microstructure as a result of selected area diffraction (SAD).

**Figure 4** Microstructure of the sample annealed for 7 h at 650 °C: (A) backscattered electron SEM-FEG image after OPU polishing and picral etching; (B) zoom on the zone selected with red dotted line in image (A); (C) and (D) TEM images.
An EDX analysis was also performed on different phases; Figure 5 shows the TEM image and EDX spectrums of ferrite and retained austenite. The Mn content in ferrite was evaluated to be around 2.2 wt. % and rather homogeneous from one grain to another. In counterpart different values were found for the retained austenite, which Mn content varies from 8wt.% to almost 12wt.%. Even so, it was possible to estimate the mean value of Mn content from different observations which is accessed to be around 8.5wt. %.

**Figure 5** EDX analysis of ferrite and austenite in the TEM for the sample annealed at 650°C for 7 h.

The presence of some laths in the retained austenite was observed by TEM (Figure.6A). Also an ultrafine grain with the band- like interior structure was found (Figure.6B). Based on previous studies [15,16], these features are supposed to be thermally activated ε - martensite laths and ε - martensite grain, respectively. Retained austenite present in the final microstructure after double annealing has a high Mn content. The mean value of Mn content is about 8.5wt.%, however in a number of cases the Mn content is so high (∼12wt.%) that the formation of thermally activated Retained austenite present in the final microstructure after two step annealing has a high Mn content. The mean value of Mn content is about 8.5wt. %, however in a number of cases the Mn content is so high (∼12wt.%) that the formation of thermally activated ε - martensite laths can be possible. According to the Schumann’s phase stability diagram of Fe–Mn–C system at room temperature [17], the presence of ε -martensite is possible with the Mn content over the 10wt. %.

**Figure 6** TEM images of the sample annealed for 7 h at 650 °C: (A) supposed ε-Martensite laths inside the retained austenite; (B) supposed ε-martensite grain respectively.
Figure 7 Histogram of retained austenite as a function of holding time at the intercritical temperature.

Figure 7 demonstrates the progress of austenite fraction retained at room temperature as a function of holding time at 650°C. It can be seen that the fraction of retained austenite is quite important already after 1h of holding (∼22%) and that it continues to increase till 7h, for which the maximum value of ∼23% is attained. Longer holding times provoke a fall of austenite fraction. Thus after a very long holding for 30h, nearly (∼20%) of retained austenite were determined. Huang et al. [18] already observed this type of evolution in the steel with similar composition, therefore the optimum time (7 h) determined in the present work seems to be coherent with previous results.

3.3. Quantitative Manganese mapping using EPMA

Quantitative Mn mapping were performed for different annealing process i.e. after cold rolling, after first annealing cycle at 750°C for 30min and after second annealing cycle at 650°C for 7h. The results of Mn mapping were shown in figure 8. It can be seen that the distribution of Mn changes along the heat treatment, long range Mn segregation being more accentuated in the cold rolled sample in comparison with the two other samples. Actually, first annealing at relatively high temperature (750°C-30min) aggravates some redistribution of Mn, but diffusion of Mn is much more evident after second intercritical annealing for a long time(7h).

Figure 8 EPMA quantitative Mn mapping of different samples:(A) cold rolled sample; (B) sample after 750°C holding for 30min; (C) sample after second annealing at 650°C for 7h.
Comparison of EPMA Mn map with the real microstructure observed in OM was done at higher magnification. Figure 9 presents both images taken on same area. Deeper investigation of these images demonstrated that the zones with low Mn content (∼3wt.%) correspond to ferrite whereas the zones rich in Mn (from 6 to 8wt.%) match with martensite and/or retained austenite. The levels of Mn in ferrite and austenite found using EPMA are coherent with the values previously measured by EDX in TEM. Macro observation of EPMA Mn map and corresponding microstructure also permits to confirm the previously stated hypothesis that the lath-like agglomerates are formed preferentially in the Mn rich bands.

![Figure 9](image-url) Comparison of EPMA Mn map (A) with the microstructure after Dino etching observed in OM (B) for the sample after second annealing at 650 °C for 7 h.

4. DISCUSSION

4.1. Microstructure evolution during two step annealing

The analysis of the microstructure after two step annealing shows the presence of three phases: ferrite, retained austenite and martensite (Figures 3-5). Two types of morphologies were observed for both ferrite and retained austenite: polygonal and lath like ones. The formation of austenite from a fully martensitic steel without any deformation proceeds into different steps. The first is that martensite is simply restored for the main reason that the driving force for recrystallization is not high enough to lead to ferrite recrystallization. Indeed, without any deformation, the stored energy is not sufficient nevertheless the martensitic structure has a high dislocation density [19]. The second step is nucleation of austenite at lath boundaries, packet boundaries and prior austenite grain size [20-21]. The third is the growth of austenite at a rate controlled primarily by carbon diffusion in austenite. This result in austenite morphology seems to be lath type as the diffusion path is lying along the preferential austenite nucleation sites mentioned previously. As a conclusion, non-polygonal ferrite and lath-like austenite coexists in the final microstructure [22]. This shows the presence of polygonal ferrite may suppose that recrystallization of martensite occurs during annealing regardless of the fact that no cold deformation was applied before thermal treatment. This shows that the dislocation density in martensite with high Mn is higher and may induce the recrystallization process. Such an assumption is supported by the works done by [23–25] that highlight recrystallization of martensite without any initial deformation.
To analyze the presence of two types of morphology in such steel, it is necessity to take into account the interaction between austenite formation and ferrite recrystallization in the presence of Mn heterogeneity. Based on EPMA analysis shown in figure 8 and figure 9 it can be assumed that microstructure can be represented as alternating bands with high and low Mn content. It can be supposed that C is uniformly distributed at the end of soaking time. By neglecting the thermodynamic interaction between C and Mn, it is possible to calculate the evolution of equilibrium volume fraction of austenite as a function of temperature in both Mn-rich and Mn-poor regions. In the Mn-rich regions (red colour bands) the austenite transformation starts at lower temperature. So it is quite possible that, austenite formation precedes ferrite recrystallization. It can be reasonably supposed that the dislocation density will decrease in the matrix due to the phase transformation. This effect is more pronounced when the austenite volume fraction is higher. This can affect both recovery and recrystallization process. As the driving force and stored energy may decreases for recrystallization prevents further formation of ferrite grains. In this situation, fibrous austenite may grow mainly along lath boundaries(lath-like morphology). Obviously, ferrite appears as lath-like because it is located between two adjacent austenite laths. This type of microstructure was already reported by Koo and Thomas [22]. In the Mn-poor regions (blue colour) the austenite formation starts at higher temperature. In that case, the recovery process is less affected by phase transformation and ferrite recrystallization may start before austenite formation or at the same time. As a result ferrite grains may appear in the Mn poor region. This indicates the interaction between austenite formation and ferrite recrystallization during heating and soaking play a key role in microstructure development.

5. CONCLUSION
The two step annealing treatment was applied to cold rolled medium Mn steel from where the following outcomes are taken from the microstructural evolution.

1. Complex ultrafine mixture of ferrite, retained austenite and martensite was obtained after two step intercritical annealing.

2. Two morphologies of micro structural features were observed: lath-like and Polygonal. This double morphology can be explained by the Mn microsegregation and the different phenomena that happen in Mn-rich and Mn-depleted bands.
4. The martensite recrystallization takes place in the Mn-depleted zones as the austenite transformation does not interfere with the recrystallization.

5. EPMA analysis demonstrates a massive Mn diffusion during the temperature of 650°C intercritical annealing. The high redistribution of Mn is enhanced when the microsegregation band spacing decreases.

6. The important fraction of retained austenite was measured in the final microstructure which results in a good TRIP effect in mechanical tests of strength and ductility.

REFERENCES


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