MICRO STRUCTURAL EVOLUTION IN LOW CARBON MEDIUM MANGANESE STEEL DURING INTERCRITICAL ANNEALING

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ABSTRACT

The present paper investigates over the influence of annealing temperature and holding time on the micro structural evolution. It was found that the microstructure evolved from martensite structure into a structure consists of austenite and ferrite during intercritical annealing process. The improved ductility may be attributed to large volume fraction of austenite retained after intercritical annealing. A thermodynamic model for the prediction of optimal annealing temperature and maximum retained austenite content has been thoroughly evaluated. For further characterization, scanning electron microscopy, EBSD, micro-hardness testing and X-ray diffraction were carried out. The investigations manifested a pronounced influence of both annealing temperature and holding time, on the phase fractions of ferrite, austenite and martensite, which must be taken into account by designing batch annealing route for Medium-Mn TRIP (Transformation induced plasticity) steels in order to obtain superior combination of strength and ductility.

Key words: Intercritical Annealing, Annealing Temperature, TRIP Steel, Microstructure Evolution, Austenite Volume Fraction.


1. INTRODUCTION

Recently, urbanisation and mechanisation of life style of the people lead to global warming which is a major environmental issue. Keeping in view of the above fact, research is in progress in automobile industries to reduce the vehicle weight thereby increasing the fuel efficiency which in turn reduces CO₂ emission. Competition is going on by automobile sectors to develop and use high strength or even ultrahigh strength steels to achieve the benefit of reduced weight and increased fuel efficiency [1]. However, increase in strength means decrease in ductility
which limits the use of above steels due to their worse formability and squeeze properties. This situation has triggered the research community to develop new grades of steel with better combination of both strength and ductility [2, 3]. It has been observed that by increasing the austenite percentage of steel can strongly enhance the ductility due to transformation induced plasticity (TRIP) of austenite.

The existence of metastable retained austenite improves the ductility of steels by transformation induced plasticity (TRIP) or twinning induced plasticity (TWIP) effects. Besides excellent combination of strength and ductility of TWIP steels, use of costly alloying elements and complicated fabrication process restricts its application for modern vehicles [3]. To overcome such problems, a series of TRIP steels have been developed and used in automobile industries which are relatively cost effective as well as results in higher amount of retained austenite. However, the commercially available TRIP steels could not be able to meet the strength and ductility required by modern automobiles. It has been observed that new types of medium manganese steels with 3-9 wt% of Mn possess high strength up to 1-1.5 GPa with total elongation up to 25-30% which are significantly higher than that of traditional TRIP steels.

Researchers have focussed attention to develop low-carbon medium manganese TRIP steels which contains significant fractions of retained austenite. In these steels sufficient manganese enrichment occurs which provides ultrafine grain microstructure contributing to mechanical stabilization of retained austenite. This retained austenite is believed to be crucial to obtain tensile ductility levels of interest. To ensure sufficient manganese enrichment, prolonged annealing time is required which can be possible by batch annealing. Further, selection of optimal annealing temperature is also a crucial factor in order to adjust an appropriate amount of retained austenite. Too low annealing temperature leads to less amount of retained austenite resulting in thermal martensite formation upon final cooling. It is apparent that there is optimal too high annealing temperatures are responsible for its insufficient chemical and mechanical stabilization, which in turn annealing temperature where a maximum of austenite can be retained at room temperature. One of the promising candidates to fulfill these requirements is the group of TRIP Steels having Medium-Mn steels with Mn contents between 4-10 Ma.-%. Using this approach, ≥ 30 vol.-% of retained austenite can be chemically stabilized by C and substantially by Mn enrichment during intercritical annealing [4, 5]. Present paper represents the transformation behavior of a steel composition with 0.09 Ma.-% C and 4.7 Ma.-% Mn, in order to determine the microstructural evolution during thermal processing of low carbon medium-Mn steel, focusing on the conditions, most applicable for batch annealing cycles. Various intercritical annealing temperatures along with the holding time were investigated where a maximum amount of retained austenite will be obtained at room temperature [6], results in evolution of the respective phases.

2. EXPERIMENTAL WORK

The investigated material was melted in a high frequency induction furnace under the vacuum atmosphere and cast to 30 kg of ingot; then the ingot was homogenized at 1200°C for 30 minutes before hot forging. Final thickness of the specimen after forging was about 30 mm. The hot forged specimen was hot rolled at 1100°C in two high rolling mills to the thickness of about 3 mm. The obtained thin sheet was kept at 1000°C for soaking time 30 minutes in trolley furnace and water quenched. This thin sheet of material was tested for XRD and confirmed to be fully martensite in nature. The water quenched thin sheet was then intercritically annealed at 620, 640, 660°C temperature in the portable furnace for soaking time 1 and 40 hours and finally water quenched to room temperature. The phase diagram of designed steel was calculated by Thermo-Calc software as shown in diagram 1.
Micro Structural Evolution In Low Carbon Medium Manganese Steel During Intercritical Annealing

Figure 1 Phase diagram of designed steel calculated by Thermo-Calc software.

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>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.09</td>
<td>4.72</td>
<td>0.006</td>
<td>0.001</td>
<td>0.005</td>
<td>0.001</td>
</tr>
</tbody>
</table>

For micro structural investigations the samples were etched for SEM and EBSD which was used to clearly distinguish the location of the retained austenite observations. The phase characterization was additionally supported by XRD using (Cu Kα radiation) using step scanning 20 from 40° to 100° with a step size of 0.02 at standard current and voltage of 40 mA and 40 kV for the purpose and ASTM E-975-03 to quantify the retained austenite content. The Vickers Hardness testing (HV1) was performed on polished samples. The data for thermodynamic modeling was calculated with the software package of Thermo-Calc (database TCFE6). The approach to predict the optimal annealing temperature was adopted from [4] and [7] consisting of following steps:

- Calculations of equilibrium amounts of austenite as function of temperature.
- Calculation of austenite composition as function of temperature assuming equilibrium conditions
- Incorporation of martensite formation during cooling assuming that diffusional transformations are concealed.

The amount of martensite formation was estimated using equation (1) proposed by [8] to estimate the $M_S$ temperature which was subsequently implemented in equation (2) [9]:

$$Ms(n^0C) = 539 - 423Ma.\%C - 30.4Ma.\%Mn - 7.5Ma.\%Si + 30Ma.\%Al$$  \hspace{1cm} (1)

$$f_{\alpha'} = 1 - \exp[-\alpha(Ms - RT)\beta]$$  \hspace{1cm} (2)

Where, $f_{\alpha'}$, is the fraction of transformed martensite, $M_s$ is the calculated $M_S$ temperature and $\alpha$ and $\beta$ are constant parameters. While in [4] $\alpha$ and $\beta$ are suggested to be constants, the approach of [7] for elevated Mn contents propose both parameters to be dependent on C and Mn following equations (3) and (4):

$$\alpha = 0.0076 - 0.0182C + 0.00014Mn$$  \hspace{1cm} (3)

$$\beta = 1.4609 + 0.4483C - 0.0545Mn$$  \hspace{1cm} (4)

C and Mn contents are given in weight percent.
The microstructure (examined by SEM) for 0.1 C-4.70 Mn steel which was austenised at 1000 °C at time of holding for 1 hour and water quenched was shown in Figure 2, as:-

**Figure 2** Microstructure of 0.1C- 4.7 Mn Steel austenising at 1000° C for 1 hour with water quenching.

As shown in Figure 2, it can be found that the microstructure was fully martensitic lath structure and Figure 3 shows the SEM micrographs representing the different annealing temperatures at the certain holding times. It was found that, martensite has apparently occurred.
during final cooling due to the high $M_s$ temperature. For the annealing temperature of 660 °C the austenite formation started along with ferrite whereas in the case of decreasing annealed temperature the $M_s$ temperature was declining. The austenite formation continued isothermally at the respective intercritical annealed temperature. The effect of reducing $M_s$ temperature with decreasing annealing temperature was also reflected in a decrease of hardness from 271 HV to 220 HV annealed at 600 °C for holding time of 40 hours. It is however noticeable that while there was a substantial reduction of the $M_s$ temperature between annealing at 640 °C to 600 °C, no significant decrease in hardness was observed for holding time of 1 hour. After annealing at 660 °C and 1 hour of holding time the microstructure exhibited cementite precipitates with an oblivious triaxial alignment which indicates the presence of tempered martensite. Self tempering of fresh martensite has apparently occurred during final cooling due to the high $M_s$ temperature.

![Figure 4](image)

*Figure 4* The value of Hardness and $M_s$ temperature with respect to Annealing temperature.

Figure 4 indicates a clear explanation of decrease in hardness in long holding time, and a slight variation of hardness for short holding time of 1 hr for 660 °C to 600 °C.

3. RESULTS

3.1 Influence of Annealing Temperature

The Intercritical annealing temperature strongly affects the phase fractions at room temperature. When the annealing temperature is low (620 °C) the amount of retained austenite at room temperature increased solely with time while at (640 °C) the amount of retained austenite increased and then decreased at 660 °C. The maximum volume fractions of retained austenite obtained under these heat treatments were 17% (620 °C , 40 hr), 23% (640 °C , 40 hr) and 19% (660 °C , 40 hr) respectively and also the hardness variation with the change of annealing temperatures and holding time. At 600 °C, the hardness increased solely with annealing time and then decreases with increase in holding time. At 640 °C the hardness reduced at first and then rises with increasing with annealing time of 40 hours. From 640 °C to 660 °C the hardness increases slightly at first and then goes on increasing with increase of holding time of 1 hour. For the annealing temperature of 660 °C the austenite formation started along with ferrite whereas in the case of decreasing annealing temperature the $M_s$ temperature was declining.

The austenite formation continued isothermally at the respective intercritical annealing temperature. The effect of reducing $M_s$ Temperature with decreasing annealing temperature was also reflected in a decrease of hardness from 271 HV to 220 HV annealed at 600 °C for
holding time of 40 hours. It is however conspicuous that while there was a substantial reduction of the $M_S$ temperature between annealing at 640 °C to 600 °C, a significant decrease in hardness was observed for holding time of 40 hours.

After annealing at 660 °C for 1 hour of holding time, the microstructure exhibited cementite precipitates with an obvious triaxial alignment which indicates the presence of tempered martensite, as shown in Figure 5. Self tempering of fresh martensite has apparently occurred during final cooling due to the high $M_S$ temperature. This indicates a clear explanation of variation of hardness in long holding time, and a slight variation of hardness for short holding time of 1 hr for 660 - 600 °C.

![Figure 5 EBSD blue: bcc and green: fcc (660 °C 1 h).](image)

The intercritical annealed specimens were characterized by lath like microstructure, as already observed by [10]. For identification of the respective phases in the steel microstructure, EBSD-measurements were performed. As demonstrated in Figure 4, the specimen annealed at 660 °C contained predominantly ferrite (blue). The second phase was identified as austenite (green). Considerable amounts of the microstructure were not detectable because of the pronounced etching attack. These areas were merged with the sections characterized as face-centered cubic and therefore can also be referred to austenite. The differences are most likely due to the Mn enrichment of the austenite, with a significantly lower electrochemical potential compared to the adjacent ferrite. The amount of retained austenite was quantified by means of XRD. According to the preceding investigations, the evaluation of the diffraction patterns indicate that with a lower annealing temperature the retained austenite content was constitutively increased up to a maximum amount of 23 vol.-% at 640 °C. It shows that by further decreasing the intercritical annealing temperature the retained austenite content continuously reduced. Two effects dominantly influence the amount of retained austenite. One is the C and Mn enrichment of austenite with the decreasing intercritical annealing temperature and therefore the chemically stabilization of the austenite. The $M_S$ temperature is thereby decreasing and a martensitic transformation during cooling is prevented. The second factor is the amount of intercritically formed austenite, which is decreasing with lower annealing temperatures and thus being a limiting factor. Consequently an optimal annealing temperature where maximum austenite content can be stabilized to room temperature is existing.
3.2. Modeling Approach

![Figure 6](image1.png)

Figure 6 Mn and C content in Austenite.

![Figure 7](image2.png)

Figure 7 A plot showing the variation of calculated values of intercritical austenite percent, Fresh martensite and retained austenite.

The modeling approach was applied for the present alloy to predict the optimal annealing temperature reaching the maximum retained austenite content. The calculations of the respective phase fractions with the instantaneous austenite composition are plotted as a function of annealing temperature in Figure 6, respectively. The comparison of the experimental data with the model calculation clearly indicates that approach by [4] deliver quite a satisfactory correlation regarding the predicted amount of maximum retained austenite (Figure 7). As to the optimal annealing temperature, there is quite a significant difference comparing the model to the experimental data.
4. DISCUSSIONS ON RESULTS AND MODELING APPROACH USED

The influence of intercritical annealing temperature on the microstructure of the steel containing 0.1 Ma.-% C 4.7 Ma.-% Mn was investigated by means of SEM, XRD, EBSD and hardness measurements. The decrease of annealing temperature resulted in a substantial decline of the $M_S$ temperature as a result of Mn and C enrichment of the remaining austenite. For annealing temperatures of less than 620 °C, the martensite formation was completely suppressed during cooling to room temperature. Parameter annealing temperature has to be considered in order to stabilize the maximum amount of the retained austenite at room temperature. Moreover, it is clear that the austenite in the case of fully austenised samples contained a lower C and Mn content compared to the austenite after intercritical annealing, which resulted in the higher $M_S$ temperature. The comparison of thermodynamic model with experimental data showed that while there was a satisfactory correlation concerning the amount of maximum retained austenite and the prediction of the optimal annealing temperature. Nevertheless, there are several possible explanations for the differences between predicted and measured optimal annealing temperature. One of them is certainly the limited holding time of 1 hr, which could be insufficient to provide full equilibrium conditions during intercritical annealing. A second possible reason could be the fact that austenite stability does not only depend on chemical enrichment but also on mechanical stabilization via grain size [11, 12]. As the austenite grain size is considered to be less than 1 μm, this could affect the martensitic transformation and therefore the optimal annealing temperature. However, it does not explain the shift at the lower temperature range, where the martensitic transformation has no influence on the retained austenite content. One further reason for the difference may results from the fact, that model include several empirical formulas. Therefore slight differences of, for example, chemical composition or initial microstructure, could lead to inaccuracies. Furthermore, in [4] there is also a considerable difference between experimental data and modeling prediction. Even so, the modeling approach is a useful tool for alloying design or estimation of annealing temperatures.

5. CONCLUSIONS

On the basis of discussions on results and modeling approach used the following conclusions have been obtained:

1. The microstructure after the intercritical annealing had a lath like morphology.
2. Dependent on annealing temperature and time the final microstructure consisted of a certain amount of ferrite, martensite, tempered martensite and retained austenite, respectively.
3. Fresh martensite, formed at higher $M_S$ temperature.
4. Lower intercritical annealing temperatures resulted in a decrease of the $M_S$ temperature stabilizing a higher amount of the retained austenite content with its maximum of 23 vol.-percent at an annealing temperature of 640 °C.
5. Based on the comparison with the experimental data the thermo dynamical modeling approach by [4] showed very good correlation concerning the prediction of maximal retained austenite content.

REFERENCES


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