Heat treatment strategies for hot-rolled medium-Mn sheet steels

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Abstract. Heat treatment strategies for advanced high-strength medium-Mn sheet steels are addressed. A review of the available thermal cycles for cold-rolled and hot-rolled sheets are presented. The benefits from the intercritical annealing following hot-rolling are emphasized. The examples of intercritical annealing for the hot-rolled sheets of initial martensitic microstructure are provided. The heat treatment was performed in a temperature range of 680-720°C for various times from 1 to 5 hours. The material used in the experiment was a medium-Mn steel containing 0.16% C, 4.7% Mn, 1.6% Al, 0.2% Mo, 0.2% Si. The scope of the research included dilatometer tests, hardness tests and microstructural characterization. The effects of temperature and time on a stabilization of retained austenite are assessed. Intercritical annealing performed at 700 and 680°C allowed forming some stable retained austenite in the microstructure. Moreover, some coiling simulations for hot-rolled strips are described.

1. State of the art
Medium-Mn sheet steels are a newest answer of the scientists for growing demands of the automotive industry regarding high-strength, formable, cost effective steel sheets [1]. They offer not only a very satisfactory combination of strength and ductility but meet other important technological requirements concerning formability (stamping, bending, etc.) zinc galvanizing, weldability, etc. [2]. Their typical microstructure consists of ferrite and retained austenite. However, the formation of ferrite takes place under conditions of ultrafine-grained microstructures, which makes the polygonal ferrite very similar in its microstructural features to bainitic ferrite. This means a dislocation density, internal stresses, etc. The crucial structural constituent is retained austenite in the amount of 20-40%. Due to its metastability this phase upon cold straining (during forming) transforms into strain-induced martensite providing the TRansformation Induced Plasticity (TRIP effect) phenomenon. This effect is very well known for first generation AHSS (Advanced High Strength Steels) [3]. Final mechanical properties depend significantly on deformation temperature, strain rate and chemical composition [4]. The latter parameter controls to a great extent the mechanical stability of retained austenite and resulting mechanical properties. In practice, C and Mn contents in retained austenite and its grain size affect a kinetics of strain-induced martensitic transformation. Due to weldability reasons a carbon content is limited below 0.2% (more often to 0.1%) [5]. This makes difficult to obtain retained austenite at room temperature because of high martensite start temperature (Ms) of steel. Typical manganese contents from 5 to 12% allow to reduce the Ms, [4]. However, the redistribution of C and Mn is beneficial during heat treatment schedules to effectively maintain the austenitic phase after final cooling to room temperature. Thus, different heat treatment strategies are needed for cold-rolled and hot-rolled steel sheets.
Figure 1. Heat treatment strategies for medium-Mn sheet steels; a) intercritical annealing (IA) or batch annealing (BA) following cold rolling, b) IA or BA following hot rolling, c) air cooling following hot rolling and sheet coiling.

1.1. Annealing of cold-rolled sheets

Typical heat treatment following cold-rolled sheets is shown in figure 1a. The conventional step requires hot rolling and cooling to room temperature. Medium-Mn steels have a martensitic structure after this step due to their high hardenability being a result of high Mn contents. The martensite is formed even at very slow cooling rates. Such a microstructure is very beneficial before an annealing step. This intercritical annealing (between $A_{c1}$ and $A_{c3}$) is crucial in forming a desired microstructure. The low-C martensite is recovered and next recrystallized very fast forming polygonal ferrite. The ferrite is very fine-grained because it nucleates at martensite laths. That is why the medium-Mn steels show typical layered microstructures. Moreover, a remaining part of the martensite is subjected to an austenite reverted transformation (ART) forming again layered thin austenitic grains along the prior martensite laths. The fast formation of the ferrite decreases the $M_s$ of the austenite due to C diffusion from the ferrite into austenite. In modern continuous annealing lines this short intercritical annealing (IA) step must be enough to decrease the $M_s$ temperature below room temperature (RT). In batch annealing (BA) schedules the time available for diffusion is much longer. Hence, the C and Mn diffusion can take place during the annealing step, which enables to decrease the $M_s$ more effectively. In other words, more cost effective lean medium-Mn steels (4-6% Mn) can be produced in this route.
1.2. Annealing of hot-rolled sheets
Typical heat treatment following hot-rolled sheets is shown in figure 1b. In this strategy a main difference is a dislocation density in the martensite before an annealing step. A lack of cold rolling is a reason of the smaller density of structural defects. Therefore, a presumable kinetics of ferrite formation and ART should be slower. Moreover, the formed ferrite-austenite mixture is more layered compared to cold-rolled sheets because of a lack of ferrite recrystallization step (or its limited extent). This affects a final morphology of structural products and time needed for efficient C and Mn redistribution. A lack of systematic studies is this field for hot-rolled sheet steels is a motivation of the research part of this study (next chapters).

1.3. Sheet coiling and air cooling
From an economical point of view it would be beneficial to produce ferrite / carbide-free bainite / retained austenite mixtures directly after hot rolling in just one technological step. Such a cheap heat treatment involving hot band coiling and subsequent air cooling of the coil is shown in figure 1c. This is a typical production strategy for hot-rolled strips used in the industry. Since the time of cooling is very long there is an opportunity to form some ferrite and carbide-free bainite fractions. If this happens the C and Mn redistributions to the remaining austenite are possible. Some laboratory trials are in progress. Further intense research is needed.

2. Material and experimental procedure

The chemical composition of the analyzed medium-Mn steel is as follows: 0.16% C, 4.7% Mn, 1.6% Al, 0.2% Mo, 0.2% Si. The steel was hot-forged (to a thickness of 20 mm) and next hot rolled and air cooled with a finishing rolling temperature of 850°C and a final sample thickness of ca. 5 mm [4]. The initial microstructure is purely martensitic (hardness ca. 525 HV1) due to the strong hardenability effect of manganese. The intercritical annealing was applied for hot rolled and machined samples. The heat treatment was performed by the means of dilatometry, which at the same time gives the information on a phase transformation kinetics upon heating and subsequent cooling. The samples in a shape of 5 mm diameter and 10 mm length were heated up to the desired temperatures in a range from 680 to 720°C (A<sub>c1</sub> is 680°C and A<sub>c3</sub> is 936°C) at a heating rate of 3°C/s, held for duration of 1, 2 and 5 hours to stimulate carbon and manganese diffusion. Finally, the samples were cooled to room temperature at the rate of 60°C/s. This heat treatment approach is presented in figure 2.

The whole process was carried out in vacuum. After the heat treatment the samples were cut in the middle part of the rod for the preparation for light and scanning electron microscopy. The samples were grinded with the use of 220, 500, 800 and 1200 grinding papers and polished with the use of 3 and next 1 µm diamond solution. Next, the samples were etched using 5% Nital. The Vickers hardness was measured with the load of 9.81 N.

![Figure 2. Heat treatment performed in the dilatometer for the hot-rolled medium-Mn steel.](image-url)
3. Results and discussion

3.1. Dilatometric analysis
The first study was a dilatometric analysis of the samples subjected to different heat treatments. These results are presented in figure 3. According to the results it can be seen that temperature of 720°C does not ensure a full austenite stability. This leads to some martensite formation upon cooling. However, if the annealing temperature drops to 700 and 680°C the stability of austenite is enough to retain it at room temperature. This effect corresponded to the amount of austenite formed during the intercritical annealing.

When the amount of austenite increases during isothermal holding the amount of polygonal ferrite decreases at the same time. Because the ferrite dissolves a very small content of carbon the excess goes to austenite increasing its stability. If the amount of ferrite is small then the amount of available carbon is small too. This situation prevents the thermal stabilisation of austenite to room temperature. The results presented in figure 3 proves this theory. When the temperature of annealing is lowered the amount of ferrite increases together with excess carbon leading to a higher concentration of carbon in the austenite [6]. The stability of austenite and related carbon content influence martensitic transformation start and finish temperatures. The higher the amount of carbon in the austenite, the lower is the martensite start temperature. The idea is to increase the carbon content in the austenite to decrease the M₁ below RT. In this situation it is possible to obtain a fraction of retained austenite in a final microstructure. The time increase in this case does not have a negative effect on a thermal stability of retained austenite. Even after 5 hours, the martensite start temperature is below room temperature. In this case not only carbon but also manganese favours the stability of austenite. Longer holding time gives more time for manganese atoms to diffuse into the austenite increasing its stability (manganese is an austenite stabilizer and it strongly reduces the M₁) [7-9].

![Dilatometric curves](image)

**Figure 3.** Dilatometric curves presenting the relative change in length during cooling the samples to room temperature following annealing at different intercritical temperatures and times.

3.2. Microstructure investigation
After the dilatometry study the light and electron scanning microscope investigations were performed. The microstructural results are presented in figure 4. The microstructure registered after the annealing at 720°C is composed of fresh martensite created during cooling, ferrite laths and a small fraction of retained austenite. The austenite is present because according to the dilatometry analysis the martensite finish temperature is below RT. Hence, the martensitic transformation is not finished. The presence of retained austenite was confirmed using XRD results (the results will be presented elsewhere).
Figure 4. Microstructure of 5Mn steel subjected to intercritical annealing (for 1 hour) between 720°C and 680°C (magnification 1kx and 20kx); a-b) 720, c-d) 700 and e-f) 680°C; F-ferrite, FM-fresh martensite, RA-retained austenite.

Figure 5. Microstructure of 5Mn steel subjected to intercritical annealing at 700°C (magnification 1kx and 10kx) for: a-b) 2 hours and c-d) 5 hours; F-ferrite, RA-retained austenite.

At 700 and 680°C temperatures the microstructure is composed of ferritic and austenitic laths without martensite. The lath-like morphology is present because the phases inherit the initial martensite morphology. During heating the ART (austenite reverse transformation) occurs leading to lath-shaped
ferrite and austenite [6, 10]. The samples annealed at 680°C are characterized by a wavy microstructure (figure 4f). The nature of such a behaviour requires further research. Moreover, the micrographs show an increase in the ferrite phase fraction together with lowering the intercritical annealing temperature from 720 to 680°C (the difference is not high). The increase of holding time affects an amount and morphology of the ferrite laths (figure 5). The polygonal ferrite formed in the intercritical region grows together with prolonging holding time from 1 to 5 hours due to longer diffusion processes. The preliminary retained austenite fractions measured using XRD at 700 and 680°C are between 20 and 30% (further research is in progress).

However, even 5 hours annealing is not enough for the microstructures to undergo a morphological transformation from a lath-like to globular morphology. Similar results were obtained by Yunbo et al. [6]. This kind of lath morphology positively influences the thermal stability of retained austenite [11,12].

3.3. Hardness results
The last step of the analysis was hardness measurement. These results are presented in figure 6. The results are in accordance with dilatometry and microstructure investigations. The lowest hardness was obtained at 680°C, which has the highest ferrite fraction. When the temperature of intercritical annealing is increased the amount of ferrite decreases resulting in an increase of the steel hardness. The hardness is increased not only because of lower ferrite fraction but by the presence of fresh martensite formed after annealing at 720°C.

Figure 6. Hardness results of 5Mn steel intercritically annealed at different temperatures and time.

4. Conclusions
The strategies for cold-rolled and hot-rolled medium-Mn sheet steels were reviewed. The differences in processing routes and microstructural features were indicated. The research part of the study dedicated to the intercritical annealing of hot-rolled 5Mn steel revealed that the temperature and time strongly affect microstructure-properties characteristics. At 720°C the microstructure was composed of ferritic and fresh martensite laths. At 700 and 680°C the austenite was stable to room temperature. It means that 700°C annealing is enough to increase the carbon content of the austenite and subsequent decrease of the martensite start temperature below room temperature. The lath-like morphology is the result of austenite reverse transformation from the initial martensite structure. The 5 hour annealing is not enough for the microstructure to change from a lath-type to more granular morphology. It is enough to increase the ferrite lath size a little bit. The decrease of annealing temperature from 720 to 680°C leads to some higher amount of ferrite phase and a resulting hardness decrease.
Acknowledgements
The financial support of the National Science Center, Poland, is gratefully acknowledged, grant no. 2017/27/B/ST8/02864.

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