INFLUENCE OF C CONTENT AND ANNEALING TEMPERATURE ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF DOUBLE ANNEALED MEDIUM MN STEELS.

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https://doi.org/10.37904/metal.2024.4877

Abstract

The effect of holding temperature on the microstructure and mechanical properties evolution of double annealed medium Mn steels with two different C level was studied. Samples were annealed at various intercritical temperatures using gradient batch annealing furnace after preliminary austenitization and quench. Mechanical properties were measured using a standard tensile test. Microstructure observations were performed and retained austenite fraction was measured using the X-ray diffraction. Curious effect of C content on the RA fraction and strength-ductility balance was observed and explained. The influence of holding temperature on the microstructure was more classical, but the resulting RA fractions and associated mechanical behavior were appealing. The questions about these relationships were addressed and certain explanations were proposed.

Keywords: medium Mn steel, double annealing, C effect, retained austenite, mechanical properties,

1. INTRODUCTION

For many years automotive market pushed the researchers in the development of high strength steels. High Mn TWIP steels have very good mechanical behaviour, but their elaboration path is quite complex and costly. Hence, studies of intermediate solutions of strength-ductility compromise have been taken and the resulted steel products are called 3rd generation AHSS. The good strength-ductility balance of these steel grades is majorly based on the enhanced TRIP effect, originating from the significant volume fraction of retained austenite (RA). One of such concepts is the so-called “Medium Mn” steel (MMS) with the Mn content in between 4 and 12 wt.% and the resulting mechanical behavior is very attractive [1-4]. Generally, these steels have an ultra-fine microstructure with a significant amount of retained austenite which can be obtained in different ways. It can be obtained during the direct intercritical annealing of hot rolled or cold rolled steel or after an additional austenitization treatment. In the last case, the formation of austenite happens through the so-called “Austenite Reverted Transformation” (ART) mechanism. Other thermo-mechanical treatments are also possible. Lately, a lot of studies in different fields were performed on the MMS. Part of them were dedicated to the phase transformations during heat treatments [5-8] and another part to the emerging mechanical behavior [9-12]. Evolution of the retained austenite under mechanical loading (TRIP effect) has also received an important attention [2, 3, 13-15]. Some of the researchers are even investigated combination of two concepts : MMS+Q&P (Quenching & Partitioning) [16-18]. Although the studies of MMS are abundant, the effect of C content on the microstructure formation during ART treatment and associated mechanical behavior was rarely adressed [19]. As well, the influence of thermal treatment parameters can be combined with the compositional changes which opens a large field for the future research.

Therefore, the main motivation of this work was to study the influence of C content and holding temperature on the evolution of microstructure during the intercritical annealing of initial as-quenched martensite structure and associated mechanical behavior.

2. EXPERIMENTAL METHODS AND MATERIAL

The studied steels were prepared using vacuum induction melting furnace. The chemical compositions of the steels are shown in Table 1. Then, the ingots were hot rolled and cold rolled to 1.2 mm thickness. The cold rolled specimens were submitted to the double annealing. First austenitization was performed in the furnace with argon atmosphere at 750°C for 30 minutes. In the end the samples were water quenched. Obtained martensitic microstructure contained small amount of retained austenite. Then the sheets were batch annealed in the batch annealing furnace with the gradient of temperature between 600 and 700°C. Consequently, 15 samples with different holding temperature were obtained for each steel composition. Holding time was fixed to two hours. Retained austenite fraction was measured using the X-ray diffraction (XRD) with Co Kα radiation. Integrated intensities of (220)α, (211)α, (200)α and (200)γ, (220)γ, (311)γ reflections was taken for the estimation of retained austenite fraction. Microstructure of obtained samples was observed using the optical and scanning electron (SEM) microscopes after Marshall etching. Two tensile tests were performed for each holding temperature on specimens with gauge length 20 mm and width 5 mm at a constant rate of 7.5mm/min.

Table 1 – Chemical composition of studied steel (10^{-3} wt. %)

Ref.	C	Mn	P	S	N	B
0.1C	98	4740	5	1	7.2	0.6
0.15C	150	4730	5	1	6.5	0.5

3. RESULTS AND DISCUSSIONS

Results of tensile tests were plotted versus annealing temperature in **Figure 1**. For both steels ultimate tensile strength (UTS) does not vary a lot till 640°C (**Figure 1 (A)**). Passed this temperature UTS increases with increasing temperature. Strength of 0.15C steel is higher because of higher carbon content. On the other hand, the yield strength at 0.2% engineering strain (YS0.2) of both steels has close values at different holding temperatures. A rapid decrease of YS0.2 can be observed at the temperatures above the 640°C.

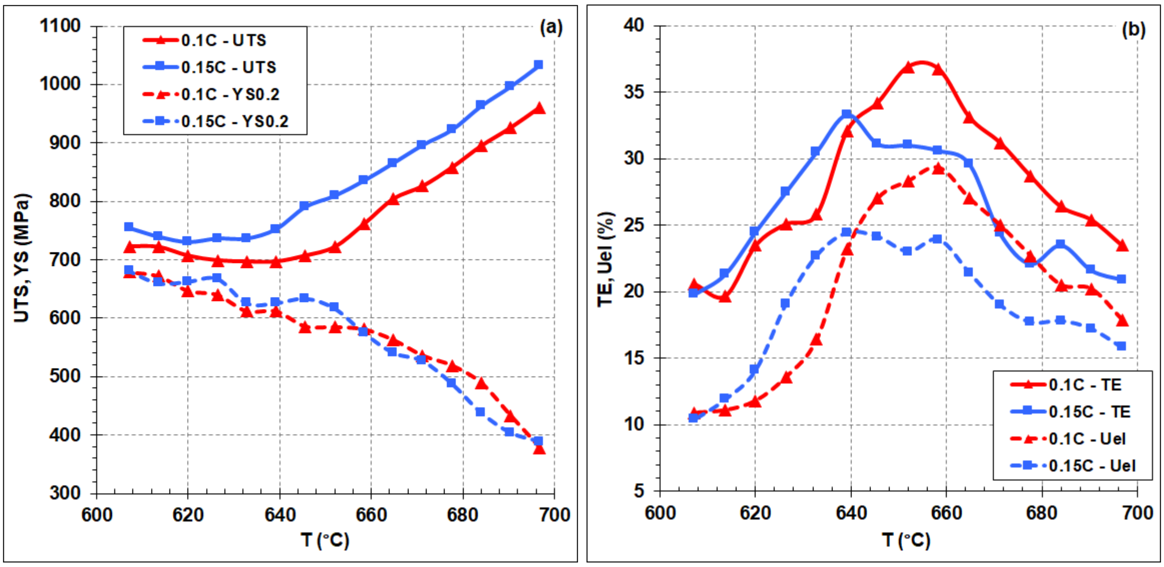

Figure 1 – Evolution of mechanical properties as a function of annealing temperature for both steels: (a) – UTS and YS; (b) – Uel and TE

The impact of temperature increase on ductility is different. Total elongation (TE) and uniform elongation (Uel) curves have a dome form ((**Figure 1 (B)**). Maximum values of Uel and TE for both grades are observed in the range between 640 and 660°C. However, the level of Uel and TE of 0.1C grade is superior to that of 0.15C grade, classically following the trend of higher elongation for lower strength.

The optical micrographs of the obtained samples at different temperatures are presented at **Figure 2 (a)**: only few micrographs were selected to represent this evolution. A classical trend of the increase of secondary phases (pearlite, fresh martensite and RA) with the increase of temperature can be observed and the fraction evolution was quantified using image analysis. The results of secondary phases fraction estimations, as well as the RA measured using XRD are presented in **Figure 2 (b)** and compared with the equilibrium fraction of austenite calculated with the CEQCSI software [20].

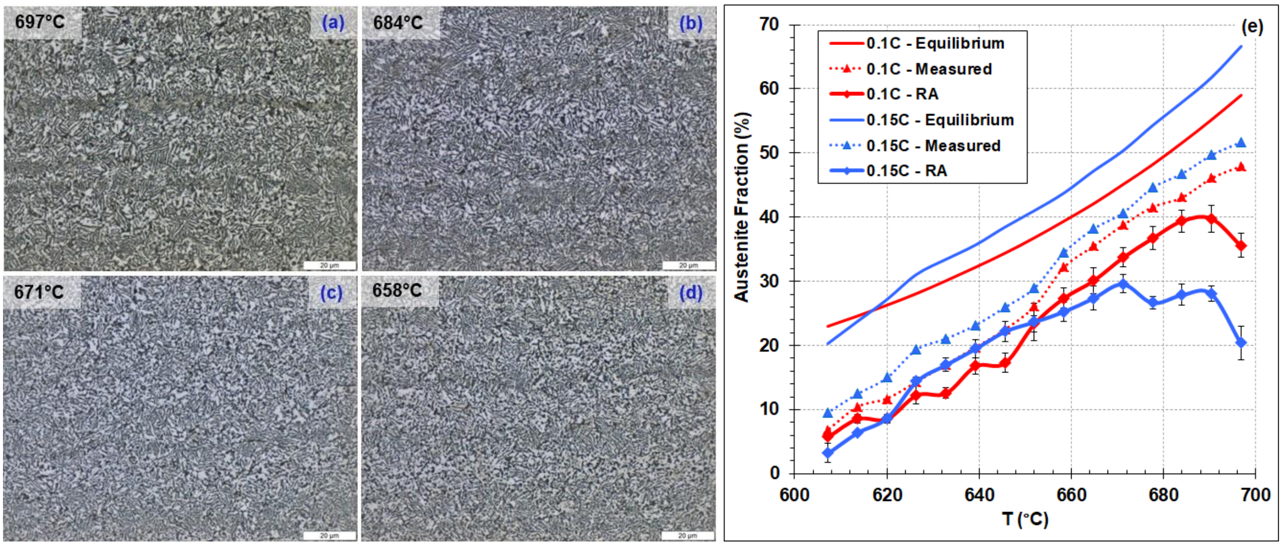

Figure 2 – (a, b, c, d) Optical micrographs of selected samples of 0.15C steel after Marshall etching; (e) Evolution of austenite fraction as a function of holding temperature: solid lines are the equilibrium austenite fraction; dotted lines with points are the measured austenite fraction at room temperature represented by all secondary phases: pearlite, fresh martensite and RA; solid lines with points are the RA measured with XRD

It can be seen in **Figure 2 (b)** that the equilibrium fraction is not reached after slow heating and 2h holding for both steels and even for higher temperature (~700°C) and the measured austenite fraction of both steels are very close. Interestingly, RA fraction at high temperatures is higher for the 0.1C steel in comparison with 0.15C and for lower temperatures (below 660°C) the fractions are very similar. This difference between 0.1C and 0.15C is contradictory to the classical thinking that higher C content provides higher RA fraction, and it will be of interest to understand why. Comparison of dotted curves with points and the RA ones also shows that there are more products of austenite decomposition during cooling (martensite and/or pearlite) in the 0.15C steel.

The strange effect of C content can be also observed in **Figure 3 (a)** which shows the compromise between strength and ductility through the parameter UTS*TE (PSE). Curiously, it can be observed that both steels show very similar trend, and the best compromise is found in the 0.1C and not in 0.15C. From the general metallurgical knowledge, the 0.15C steel is more prominent to stabilize higher RA fraction, due to higher C content, and, thus, it can be supposed to have better strength-ductility balance. But in this study an inverse trend is observed. To understand the relation between RA fraction and measured ductility of samples, Uel was plotted s function of RA fraction in **Figure 3 (b)**. For low fractions of RA a sort of linear relation with Uel can be seen. However, the tendency is much more complex for the fractions above 15% and it can be represented as a loop. It is also interesting to note that alike loop-trend is obtained for both steels, nevertheless the C content. It can be observed that similar Uel is obtained with very different RA fractions. To understand better this relation and the surprising effect of C, several samples were selected for SEM characterization. The selection was based on the holding temperature and Uel which was targeted to be similar or close to the maximum level for both steels: 697°C, ~680°C, ~660°C and 639°C. **Table 2** presents the Uel of the selected samples.

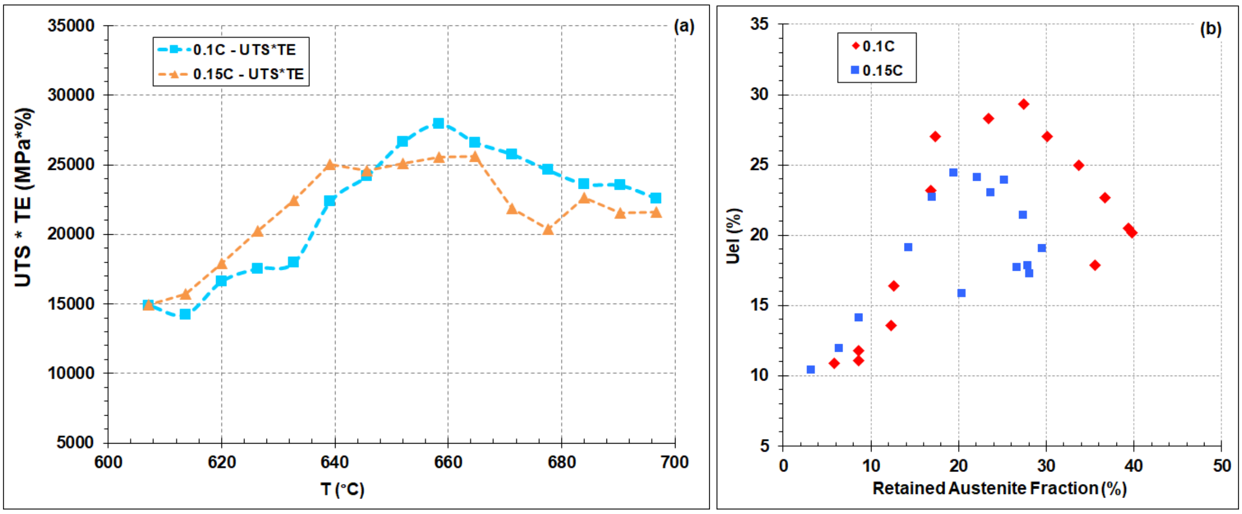

Figure 3 – (a) Evolution of PSE (UTS*TE) parameter as a function of annealing temperature for both steels; (b) Relation between Uel and RA fraction

Table 2 – Uniform elongation of selected samples (%)

Ref. \ Thold	697°C	~680°C	~660°C	639°C
0.1C	17.9	22.7	29.3	23.2
0.15C	15.8	17.8	21.4	24.4

Figure 4 shows the SEM images of the selected samples after Marshall etching. The evolution of the microstructure between different C content steels and different holding temperatures can be observed. It is coherent with the optical micrographs and estimated evolution of secondary phases: secondary phase fraction increases with the temperature increase and slightly higher fraction is observed for higher C content steel. Interestingly, in the case of 0.15C steel micrographs in **Figure 4 (e, f, g)** show the presence of small fraction of pearlite. But no pearlite was observed in 0.1C steel. This can be the reason why 0.15C has lower RA fraction than 0.1C. In fact, during slow process of Batch Annealing cooling part of the austenite is transforming to pearlite and, thus, also consuming part of the C which won't be available for austenite stabilization at room temperature. As it is known, pearlite formation is promoted with the increase of C content, hence, it is observed in 0.15C steel and not in 0.1C steel.

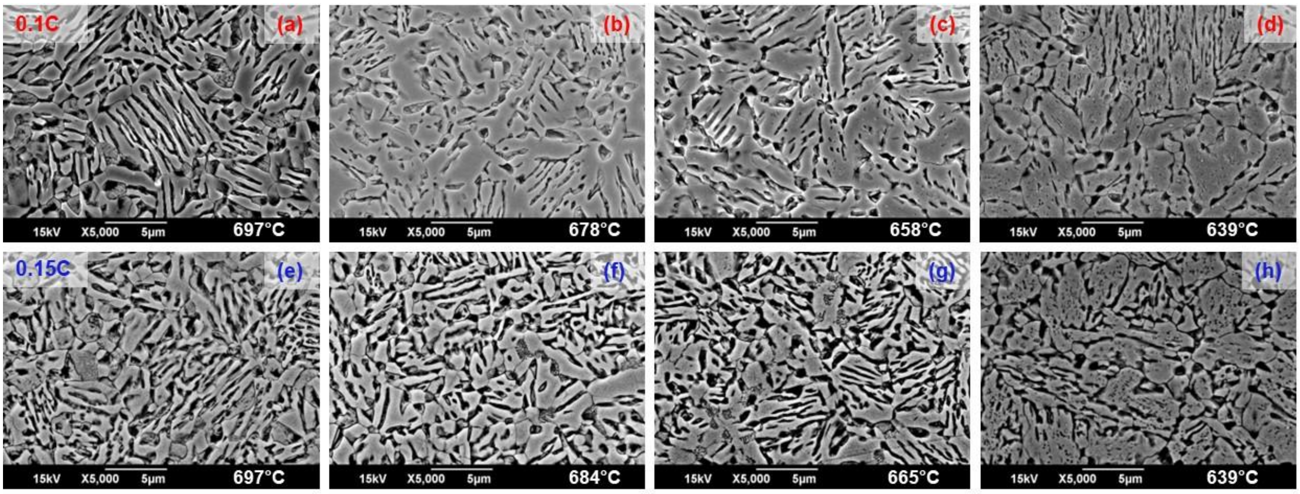

Figure 4 – SEM images of samples annealed at different holding temperatures for both steels: (a-d) 0.1C and (e-h) 0.15C

Presence of pearlite can explain the similar PSE curves of two steels and the lower RA fraction of 0.15C steel as if it contained lower C content. However, there are still unanswered questions about similar ductility but with different RA fractions and/or similar RA fraction resulting in different elongations. Of course, partial response to these questions is the RA mechanical stability and the evolution of RA during mechanical loading which was already studied [2, 3, 13-15]. Although, there are another factors that can have influence on the ductility of these steels like matrix and presence of minor phases. In case of this study both of them are quite important. The initial structure before annealing is the as-quenched martensite, hence during annealing it will undergo tempering and carbides formation. It is known that C content has an important influence on both [21], hence the final mircostructure will be also dependent on it. It is also aknowdlged that with the increase of C content the density of dislocation of the as-quenched martensite is increasing, hence one can say that the final matrix after annealing may contain higher dislocation density. Today, it is only a speculation and it is needed to be further studied and to be prooved.

4. CONCLUSION

Two medium Mn steels with different C level subjected to double annealing with different holding temperatures were studied. Microstructure evolution and associated mechanical behavior was monitored as a function of holding temperature and C content. Retained austenite fraction was measured using the X-ray diffraction for each annealing condition. The following outputs can be drawn from this work:

- Strength and elongation evolutions as a function of holding temperature are very alike for both steels with typical increase of UTS and decrease of YS at high temperatures and optimum peak of elongation.
- Steel with higher C content (0.15C) has similar or even lower strengh ductility balance at higher temperatures. This is due to the pearlite formation during slow cooling which traps part of the C, making it unavailable for RA stabilization.
- Higher RA fraction was obtained for 0.1C steel in comparison with the 0.15C steel at temperatures above 660°C. The maximum achieved RA fraction in 0.1C steel was close to 40%.
- At high annealing temperatures both steels have similar and particular relationship between Uel and RA fraction in form of loop. This means that for different RA fractions the observed level of ductility is similar and, inversely, for the same RA fraction different Uel can be obtained.

The observed trends of ductility and evolution of microstructure raise some questions about the influence of matrix and of minor phases on the mechanical behavior of double annealed medium Mn steels which should be further studied.

ACKNOWLEDGEMENTS

Authors are very grateful to Aude NADLER and Gregory LEUILLIER from ArcelorMittal Maizieres Research and Development for their help with the XRD measurements.

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