

Kinetics of the austenite formation during intercritical annealing

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ABSTRACT

Purpose: of this paper is the effect of the microstructure of the 6Mn16 steel after soft annealing on the kinetics of the austenite formation during next intercritical annealing.

Design/methodology/approach: Analytical TEM point analysis with EDAX system attached to Philips CM20 was used to evaluate the concentration of Mn in the microstructure constituents of the multiphase steel,

Findings: The increase in soft annealing time from 1-60 hours at 625 °C increases Mn partitioning between ferrite and cementite and new formed austenite and decreases the rate of the austenite formation during next intercritical annealing in the ($\alpha+\gamma$) temperature range at 700 and 750 °C.

Research limitations/implications: The amount of the austenite and final multiphase microstructure can be optimised by changing the time / temperature parameters of the intercritical heating in the ($\alpha+\gamma$) temperature range.

Originality/value: The knowledge of partitioning of alloying elements mainly Mn during soft annealing is very important to optimise the processing technology of intercritical annealing for a given amount of the austenite.

Keywords: Heat treatment; Soft and intercritical annealing; Mn partitioning; Austenite formation

1. Introduction

Problem of the austenite formation during intercritical annealing of low carbon medium manganese steel has been less studied than mechanisms and kinetics of the austenite phase transformations on cooling from full austenitization temperature or the ($\alpha+\gamma$) temperature range and after controlled deformations [1-9]. In the current research of the intercritical heating of 6Mn16 steel in the ($\alpha+\gamma$) temperature range, it has been assumed that Mn diffusion in ferrite is much faster than in austenite. However for equilibrium partitioning of Mn between ferrite and austenite in low carbon manganese steel very long time of heating is needed. It was mentioned in paper [10] that for steel having 0.11 wt.% C and 2.67 wt.% Mn which was annealed in the ($\alpha+\gamma$) temperature range at temperature 680 °C after 10⁸ s the equilibrium balance was achieved for Mn partitioning between ferrite and austenite. Thus to decrease that time markedly soft annealing below A_{C1} temperature at 625 °C for 1-60 hours was applied to the

investigated steel after controlled rolling [11-15]. Purpose of that treatment was to activate Mn diffusion in the ferrite matrix and Mn enrichment process in the cementite and bainitic carbides.

2. Material and investigation methods

The chemical composition of the investigated 6Mn16 steel was 0.047 wt.% C, 3.97 wt.% Mn, 0.37 wt.% Si, 0.003 wt.% P, 0.009 wt.% S, 0.01 wt.% Cr, 0.17 wt.% Ni, 0.02 wt.% Mo, 0.02 wt.% Cu, 0.02 wt.% Nb, 0.017 wt.% Ti. The steel, after heating at 1172 °C for two hours was controlled rolled into plates of 12 mm in thickness. The critical temperatures of diffusional phase transformations on heating were $A_{C1}= 647$ °C and $A_{C3}= 850$ °C.

The dilatometric investigations were performed with dilatometer Bähr DIL 805 and DO 105 with transducer for expansion/contraction measurements of the sample length. Electronic signal was calibrated with computer, so amount of newly formed austenite was measured during continuous intercritical annealing at 700 °C and

750 °C for 1800 s. Also some amount of austenite was detected during soft annealing at temperature 625 °C for duration 10 and 60 hours.

The chemical composition of the microstructural constituents of the multiphase 6Mn16 steel were determined by analytical STEM point analysis with EDAX system attached to Philips CM20 transmission electron microscope.

3. Description of achieved results and discussion

The results of the effect of the soft annealing time at 625 °C on microsegregation of Mn and Si between ferrite matrix and carbides and locally austenite islands are presented in Table 1.

The partitioning effect of Mn between carbides and retained austenite increased with the increase in duration time of soft annealing from 3 to 60 hours, what is presented in Fig.1.

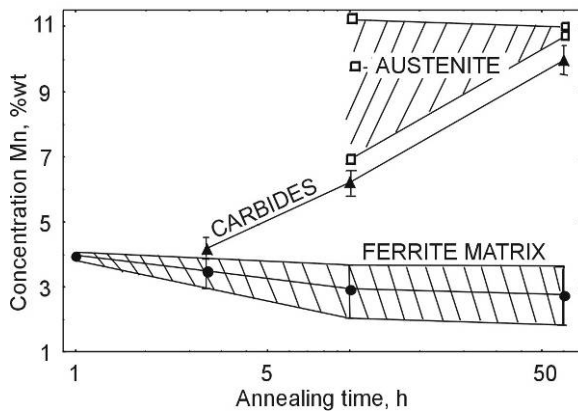


Fig. 1. Manganese concentration changes in structural constituents of 6Mn16 steel with annealing at 625°C

Table 1.

Effect of annealing time at 625°C on alloying elements concentration in structural components of the 6Mn16 steel

Alloying element	Annealing time, hours							
	3	10	60	3	10	60	10	60
	Concentration of element in % wt.							
	Ferrite matrix			Carbides			Austenite	
Si	0.44	0.4-0.57	0.36-0.61	0.36	0.15-0.33	0.15-0.31	-	-
Mn	3.5	2.06-3.81	1.9-3.5	4.0-4.35	5.73-6.69	9.69-10.2	7.0 – 11.2	10.7 – 11.3
Average Mn	3.5	2.93	2.70	4.18	6.21	9.95	9.1	11.0

In ferrite matrix continuous decrease in Mn concentration was observed with increase in time of soft annealing process while in carbides and residual austenite the concentration was increased.

The effect of Mn partitioning between ferrite and austenite was calculated for 6Mn16 steel with Thermo-Calc program for different temperatures of heat treatment in the ($\alpha+\gamma$) range. The results are presented in Table 2. They are compared to experimental data of Mn segregations between ferrite and austenite determined by microsegregation studies on thin foils with analytical STEM point analysis in Fig.2.

By simulation of soft annealing of 6Mn16 steel at 625 °C with program Thermo-Calc for conditions of thermodynamic equilibrium it was possible to foresee that microstructure will consist of 86.3% of ferrite with 2.86 wt.% Mn and 13.4 % of austenite with 11.2 wt.% Mn and small amounts of MC and M₃C carbides, i.e. (FeMn)₃C with 1.60 wt.% Mn. This results allows to foresee the direction of the changes in microstructure for given temperature, time and alloying concentrations in the steel. Thus the detected highly alloyed with Mn carbides will dissolve in the surrounding austenite increasing its concentration to 11.2 wt.% Mn while alloyed cementite will stay at 1.6 wt.% Mn. That dissolution process of alloyed carbides in newly formed austenite probably had an effect on discrepancies between calculated and measured experimental values of Mn concentrations shown in Fig.2. The increased concentrations of Mn at 625 °C for 10 and 60 hours will affect local decrease of A_{C1} temperature thus austenite islands may rise. The formation of residual austenite was confirmed with X-ray analysis and by observations of bainite-martensite islands on thin foils of rapidly cooled samples from 625 °C as an effect of phase transformations of the new austenite to martensite. It was confirmed that soft annealing at 625 °C activates Mn diffusion in the ferrite matrix and Mn enrichment process in cementite. On the other hand saturation of carbides with Mn will change their dissolution process during next intercritical annealing heating and may also change kinetics of austenite formation in the ($\alpha+\gamma$) range.

Table 2. Thermo-Calc calculations of Mn partitioning between ferrite and austenite of 6Mn16 steel for given temperature

Temperature, °C	α - Fe, wt.% Mn	γ - Fe, wt.% Mn
625	2.86	11.20
700	2.413	6.925
750	1.967	4.617
780	1.80	4.0
Ac ₃	-	3.97

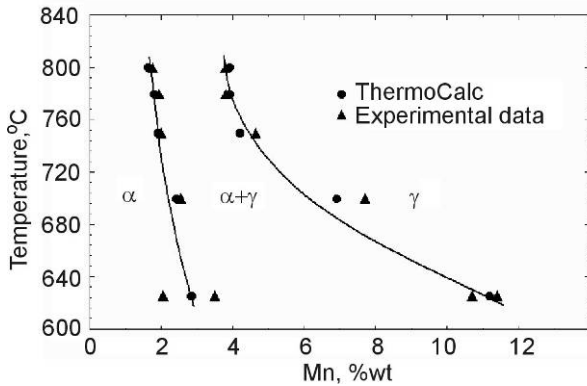


Fig. 2. Partition of Mn between ferrite and austenite of 6Mn16 steel

Final TRIP assisted multiphase microstructure of 6Mn15 steel can be optimised by changing time/ temperature parameters of the intercritical heating. Thus progress of austenite formation was studied to establish conditions of heat treatment for 20- 35% of austenite in the structure

This amount of austenite is needed for creation of the multiphase ferrite-martensite and retained austenite microstructure after fast cooling from ($\alpha+\gamma$) temperature range. Temperatures 700 and 750 °C and time 1800 s were selected for austenite progress studies.

In Fig.3 the effect of soft annealing pre-treatment on the amount of austenite formed after 1800 s at 700 °C is presented and compared to the kinetics of austenite formation during immediate annealing after controlled rolling of the steel. The fastest progress of austenite formation was observed at immediate annealing. The longer time of the soft annealing the slower is kinetics of the austenite creation during intercritical annealing at 700 °C. During immediate annealing at 700 °C 20% of austenite was achieved after 90 s while 25% after 112 s and 30% after 150 s. Phase transformation of bainitic microstructure into austenite is quite fast during continuous heating at rate 0.6 °C/s up to temperature 700 °C. 35% of new austenite was determined from dilatometric studies after 200 s.

However soft annealing at 625 °C intensified Mn partitioning. Saturation of carbides with Mn caused their slower dissolution and reduced the progress of $\alpha\rightarrow\gamma$ transformation as well as grain growth. It was found that for 20% of new austenite formation which was rich in C and Mn the increase in time from 90 to 708 s was observed after soft annealing 3600s at 625 °C and intercritical treatment 700 °C/1800 s.

These long times of intercritical annealing allows for easy control of the intercritical heat treatment at 700 °C. For the

increased duration of the soft annealing at 625 °C to 36 ks the delay in $\alpha\rightarrow\gamma$ transformation progress at the level of 20% of austenite was 1260 s and for 216 ks was 1780 s.

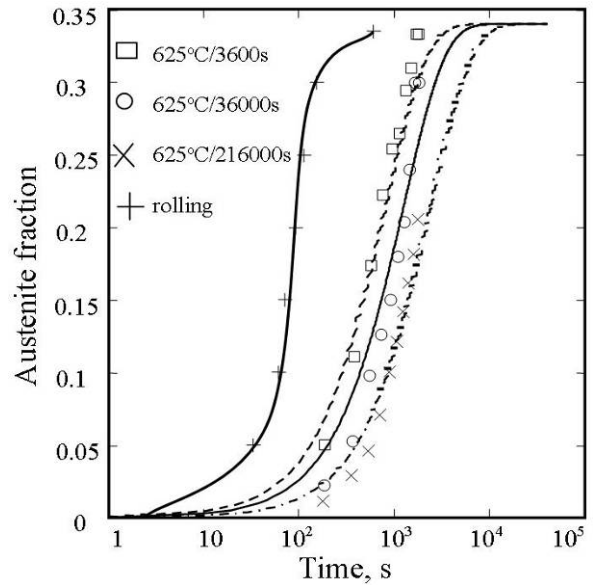


Fig. 3. Effect of annealing time at 625°C on the increase austenite fraction in 6Mn16 steel after annealing at 700°C for 1800 s compared to immediate annealing after rolling

These kinetics of $\alpha\rightarrow\gamma$ phase transformation were described with Avrami, Johnson-Mehl equations [13-15]. From experimental data which are presented in Fig.3 the value $n=1$ in J-M equations was established. Thus from mathematical simulations of the progress of phase transformation curves the constant parameter “k” values, which represented the rate of transformation were determined as: $k_{3600}=12\cdot 10^{-4}$ and $k_{36ks}= 8\cdot 10^{-4}$.

Progress of the diffusional phase transformation is determined by the movement of interphase boundaries; so $k=1/t$ where

$$t=t_{X_v}=t_{0.215} \tag{1}$$

at condition $n=1$ in equation J-M and standardisation that 0.34 relates to 100% of the total progress of $\alpha\rightarrow\gamma$ transformation at 700 °C one achieves

$$Y= 1- \exp(-kt)^n \text{ and } Y=X_v \tag{2}$$

$$X_v=1- \exp(-kt)^n =0.34 \tag{3}$$

t_{X_v} – time for fraction of transformation X_v
 k – constant for transformation rate

In Fig.4 there are presented the progresses of $\alpha\rightarrow\gamma$ transformation at 750 °C for immediate annealing the bainite microstructure of 6Mn16 steel after controlled rolling and after pre-treatment of soft annealing at 625 °C for 3600 s and 36ks. Time for formation of 20% and 50% of austenite was studied. For bainite microstructure time for reaching 20% of austenite and 80% of ferrite was 100 s, while after soft annealing 625°C for 3600 s was 280 s and that for 625°C/36ks was 500 s.

The increased temperature 750 °C makes easier Mn diffusion partitioning between ferrite and austenite. For formation of duplex structure 50% α +50% γ only 189 s is needed during immediate annealing after controlled rolling and after soft annealing 700 s or 1400 s – Fig.4. Assuming that phase transformation mechanism of $\alpha \rightarrow \gamma$ at temperature 750 °C is analogous to that at 700 °C and value $n=1$ the values of k in equations of J-M were calculated to be $k_{3600}=15 \cdot 10^{-4}$ and $k_{36ks}=9 \cdot 10^{-4}$.

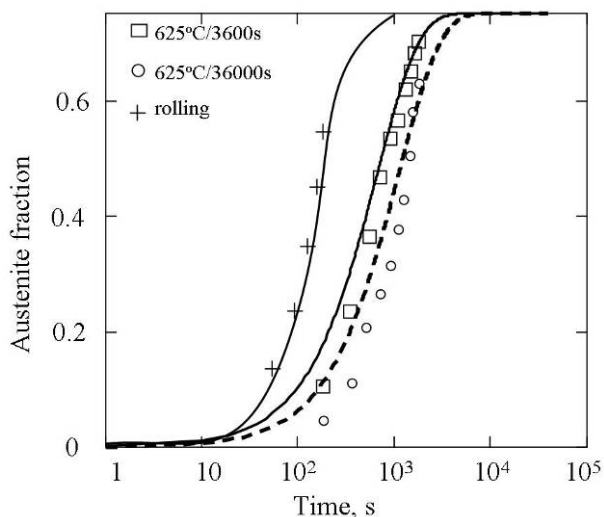


Fig. 4. Effect of annealing time at 625 °C on the fraction of austenite in 6Mn16 steel after annealing at 750 °C for 1800 s compared to immediate annealing after rolling

For thermodynamic equilibrium at temperature 750 °C, 75% of austenite may be formed with concentration of 4.62 wt.% Mn. This is slightly higher than average concentration of Mn in the steel. So that is the reason why small amount of retained austenite is observed in duplex microstructures after quenching the steel and why small TRIP effect is observable for the final product.

4. Conclusions

Mn partitioning between ferrite and cementite during soft annealing at temperature 625 °C before intercritical heat treatment has the influence on the kinetic of austenite formation during the course of phase transformation $\alpha \rightarrow \gamma$.

It has been shown that soft annealing before intercritical heat treatment in the ($\alpha+\gamma$) temperature range affected value of the parameter “ k ” which is responsible for advancement of the phase transformation.

At the first stage of diffusion controlled phase transformation, the growth of the isolated austenite islands was observed for those which met conditions for the nucleation at the beginning of transformation.

At further stage of transformation the continuous nucleation at the grain boundaries and interfaces of α/α and $\alpha/\text{cementite}$ takes place as at the preferential sites for heterogeneous nucleation and growth of γ phase in α ferrite matrix. This situation correspond to $n=1$ in J-M equation.

The increase in soft annealing time from 1-60 hours at 625 °C increases Mn partitioning between ferrite and cementite and new formed austenite and decreases the rate of the austenite formation during next intercritical annealing in the ($\alpha+\gamma$) temperature range at 700 and 750 °C.

References

- [1] A.K. Lis, J. Lis, Effect of hot deformation and cooling rate on phase transformations in low carbon HN5MVNb bainitic steel, Trans Tech Publications, Materials Science Forum 539-543 (2007) 4620-4625.
- [2] J. Lis, A.K. Lis, C. Kolan, Processing and properties of C-Mn steel with dual-phase microstructure, Journal of Materials Processing Technology 162-163 (2005) 350-354.
- [3] B. Gajda, A.K. Lis, Intercritical annealing with isothermal holding of TRIP CMnAlSi steel, Journal of Achievements in Materials and Manufacturing Engineering 20 (2007) 439-442.
- [4] N. Wolańska, A.K. Lis, J. Lis, Investigation of C-Mn-B steel after hot deformation, Archives of Materials Science and Engineering 28/2 (2007) 119-125.
- [5] A.K. Lis, B. Gajda, J. DeArdo, New steel chemistry design for TRIP and Dual Phase structures, Materials Science and Technology 2005, Pittsburgh, USA.
- [6] S.K. Chang, J.H. Kwak, Effect of Manganese on Aging in Low Carbon Sheet Steels, ISIJ International 37 (1997) 74-79.
- [7] K. Eberle, P. Cantinieux, P. Harlet, New thermomechanical strategies for the production of high strength low alloyed multiphase steel showing a TRIP effect, Steel Research 70/6 (1999) 233-238.
- [8] G. Michta, J. Pietrzyk, W. Osuch, A. Kruk, Stability of retained austenite at low temperature in low carbon cooper bearing steels with TRIP assisted effect, Materials Engineering (2003) 339-342 (in Polish).
- [9] G. Michta, W. Osuch, A. Kruk, Testing of isothermal transformation of undercooled austenite within 380-650 °C temperature range in low carbon steels with containing manganese and silicon, Metallurgist, 4 (2003) 171-176 (in Polish).
- [10] S. Sun, M. Pugh, Manganese partitioning in dual phase steel during annealing, Materials Science and Engineering A276 (2000) 187-174.
- [11] J. Lis, J. Morgiel, A. Lis, The effect of Mn partitioning in Fe-Mn-Si alloy investigated with STEM-EDS techniques, Materials Chemistry and Physics 81 (2003) 466-468.
- [12] J. Lis, Microsegregation of manganese in low carbon steels during intercritical heat treatments, Publisher WIPM and FS, Czestochowa University of Technology, Seria: Materials Engineering 7, Czestochowa, 2005 (in Polish).
- [13] Z. Kędzierski, Phase transformations in condensed systems, Academia Publisher AGH, Krakow, 2003 (in Polish).
- [14] G. Guy, Introduction to Materials Science, PWN, Warsaw, 1970 (in Polish).
- [15] J. Pietrzyk, G. Michta, A. Kruk, Kinetics of bainite transformation of austenite formed after annealing in temperature range $A_1 - A_3$ in steel 0,2 %C, 1,5 %Mn, 1,5 %Si, Metallurgist 5 (1997) 218-222 (in Polish).