

## INFLUENCE OF INTERCRITICAL ANNEALING PARAMETERS ON THE KINETICS OF THE AUSTENITE FORMATION IN 41MnSi6-5 TRIP STEEL

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### Abstract

The results of studies of the influence of intercritical annealing parameters on the austenite formation and its stability in TRIP steel (0.41 % C, 1.52 % Mn and 1.22 % Si) were presented in the paper. Based on the results of a dilatometric analysis and metallographic investigation it was noted that the pearlite to austenite transformation does not occur at a constant temperature, which is referred to as  $A_{c1}$ , but rather within certain - possible to determine - temperature range which is enclosed within  $A_{c1s}$  and  $A_{c1f}$  values.

It was shown, that in order to achieve austenite with a high carbon content, it is required to perform its annealing at a temperature slightly higher than the temperature at which the pearlite to austenite transformation begins ( $A_{c1s}$ ).

Dilatometric analyses of cooling curves confirmed these observations. It was noted that the austenite formed at a temperature of  $A_{c1s}+10$  °C transforms into martensite at a much lower temperature ( $M_s$ ) than austenite formed during annealing in two-phase ( $\alpha+\gamma$ ) temperature range, i.e. between  $A_{c1f}$  and  $A_{c3}$  temperatures. It was also found that the formed austenite was the more stable the shorter annealing time was applied.

Based on the obtained results the isothermal time-temperature-austenitisation (IHT) diagram was developed.

**Keywords:** TRIP steels, phase transformations, critical temperatures, IHT diagram

### 1. INTRODUCTION

The presence of mechanically unstable retained austenite in the microstructure provides an easy way forming elements made of TRIP steels. On the other hand, the mechanical destabilization of this phase, which occurs during such forming, contributes to the strong strengthening after the manufacturing process of already molded components [1-5].

Due to the fact that usually TRIP steels are low and less likely - medium carbon, to remain in their microstructure significant volume fraction of the retained austenite can not be easy. This problem contributed to the development of the research on the increasing volume fraction of this phase in the microstructure of TRIP steels by optimizing their heat treatment technology [6-10].

One of the main stages of the heat treatment of TRIP steels is annealing in the critical temperature range. There are as yet no clear guidelines in what temperature and for how long should be the TRIP steel - with a certain carbon content - annealed. On the one hand, technologists should be interested in obtaining the highest possible saturation of austenite with carbon, which is formed in this temperature range. On the other hand, the volume fraction of this phase should be as large as possible.

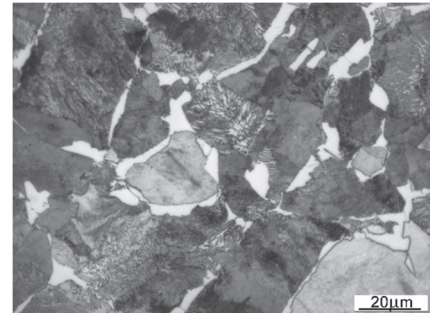
Looking for a compromise between the volume fraction of the forming austenite and the carbon saturation of this phase, the question arises: how the holding temperature and time can affect these two opposing factors? Analyse of the influence of these parameters on the kinetics of austenite formation in 41MnSi6-5 TRIP steel, which were described in this paper constitute an attempt to answer this question.

## 2. MATERIAL AND METHODOLOGY

The chemical composition of the 41MnSi6-5 steel studied in the hereby work was Fe-0.41 %C-1.52 %Mn-1.22 %Si (in mass %). The microstructure of the investigated steel in as-delivered state is presented in **Fig. 1**. It is easy to notice that is hypoeutectoid steel with a significant, nearly 85 vol. % pearlite fraction.

Samples ( $\phi$  3x10 mm) were heat treated in the L78 R.I.T.A. dilatometer. Each of the applied heat treatment variants consisted of:

- stage I** - heating with a rate of 100 °C/s to temperatures: 700, 730, 740, 750, 800, 850 and 900 °C,
- stage II** - isothermal holding at the set temperature for 7200 s (and for 750 °C - 1, 10, 100 and 1000 s additionally)
- stage III** - cooling to ambient temperature with a rate of 100 °C/s.



**Fig. 1** The microstructure of investigated 41MnSi6-5 steel in as-delivered state

For each heat treatment stage the relative elongation changes ( $\Delta L$ ) as a temperature (T) or time (t) function were determined and numerically recorded. Differentials for these dependencies i.e.  $d(\Delta L)/dT$  and  $d(\Delta L)/dt$  allowed for the accurate determination of temperatures and times at which the heat-treated samples were subtly changing their elongation.

Analysis of data recorded during the heat treatment of 41MnSi6-5 steel enabled the determination of:

- the critical temperatures at heating with the rate of 100 °C/s,
- the start (and/or finish) time of transformations during isothermal holding (when it was possible),
- the influence of holding parameters on  $M_s$  temperature.

After the dilatometric investigations metallographic cross-sections were made on the samples. After etching in 2 % nital, microstructures were observed in the light microscope Axiovert 200 MAT. These observations allowed to verify the dilatometric investigations and enabled to construct the original isothermal time-temperature-austenitisation (IHT) diagram for the investigated steel.

## 3. RESULTS AND DISCUSSION

### 3.1. Microstructure after isothermal holding

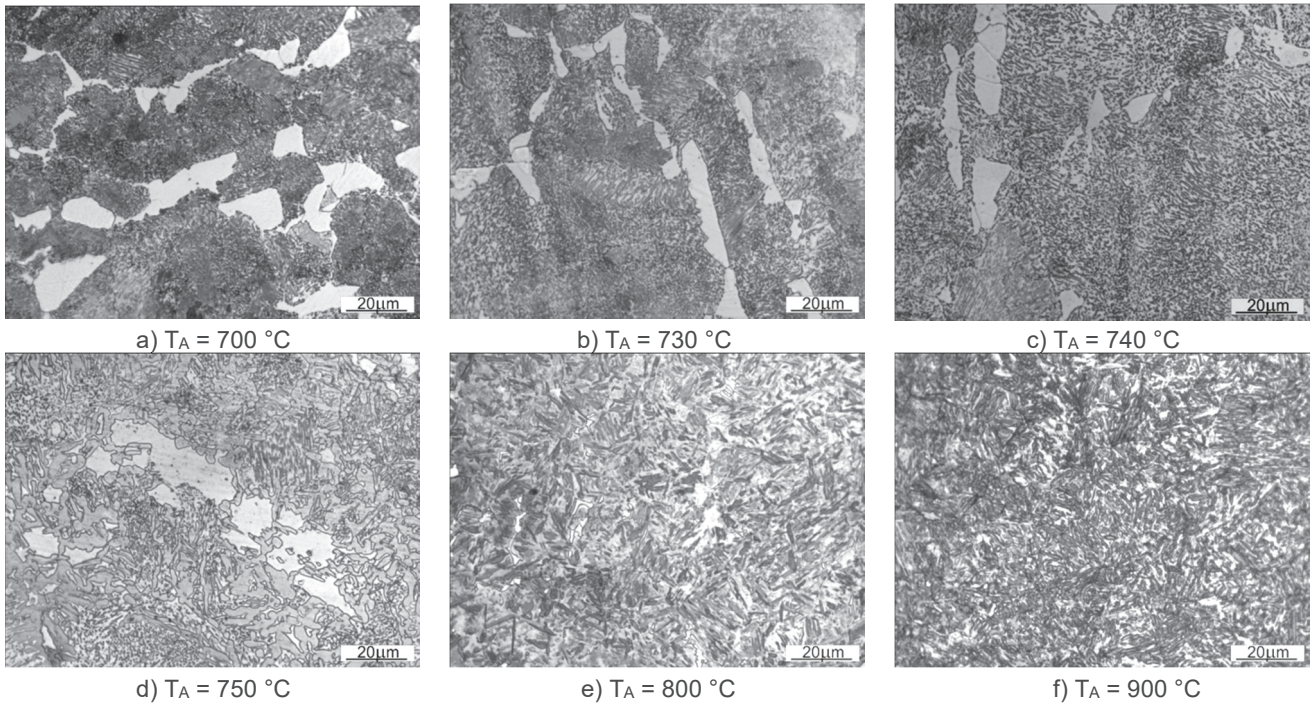
**Fig. 2** shows the microstructure of 41MnSi6-5 steel samples after isothermal holding in the range 700 -900 °C. Progressive severity of the coagulation of pearlitic cementite is well illustrated in **Figs. 2a and 2c**. For sample which was held at 740 °C (**Fig. 2c**), the coagulation is already so significantly advanced that the pearlitic cementite remains in the form of classical lamellas only in a few areas.

The microstructures shown in **Figs. 2 a ÷ c** prove that it is difficult to see other than ferrite and pearlite microstructural components. This may mean that austenite formation can not be started generally, despite the holding in the temperature range 700 ÷ 740 °C, ongoing for 2 hours. However, it is conceivable that it could occur, but only in a very limited range and austenite formed in this way, which during a rapid cooling should transform into martensite, is not possible to be seen at the magnification used for the light microscopy.

Most likely grey areas in pearlite and at the boundary between the pearlite and ferrite, clearly visible in **Fig. 2d**, are products of such martensitic transformation and suggest that during isothermal annealing at 750 °C certainly the austenite formation occurred.

After isothermal annealing in range of 800 ÷ 900 °C in the microstructure of quenched samples almost only martensite is visible (**Fig. 2e and 2f**). That means that austenite is formed even faster after annealing in such

temperature range. A lack of bright areas in the microstructure of samples which were annealed at 850 °C and 900 °C (**Fig. 2f**) can indicate that the ferrite to austenite transformation was completed.



**Fig. 2** The microstructures of the investigated 41MnSi6-5 steel, after isothermal annealing in the range 700 ÷ 900 °C for 7200 and subsequently cooling with the rate of 100 °C/s - to a room temperature

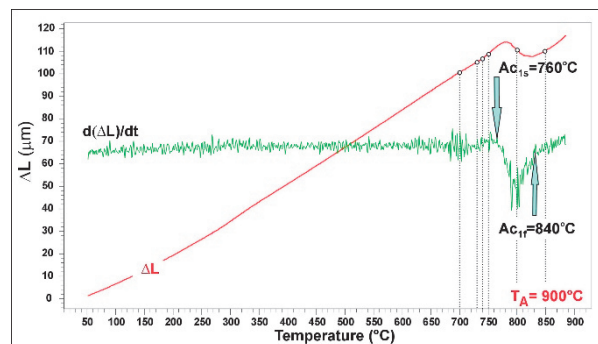
Metallographic analysis of the 41MnSi6-5 steel microstructure provided useful information on the thermally activated processes which occurred during isothermal annealing. However these studies, only with the results of dilatometric analysis, may be used for the evaluation of the influence of isothermal annealing parameters on the kinetics of phase transformations - on the austenite formation especially.

### 3.2. Dilatometric analysis of heating curves

**Fig. 3** shows an example dilatometric diagram, which in detail illustrates the elongation changes of heating sample to the highest temperature as a function  $\Delta L = f(T)$  and corresponding to such dependencies a differential curve  $d(\Delta L)/dT = f(T)$ .

It is easy to see that no dilatation effects induced phase transformations during the heating of steel samples to the 750 °C. This can mean that the observed constant increasing of samples elongation was caused only by thermal expansion and the potential transformations can start only after the beginning of the isothermal holding at a given temperature.

Above the 750 °C a significant negative effects of elongation on the dilatometric curves of samples heated to range of 800 ÷ 900 °C which is the result of the pearlite to austenite transformation.

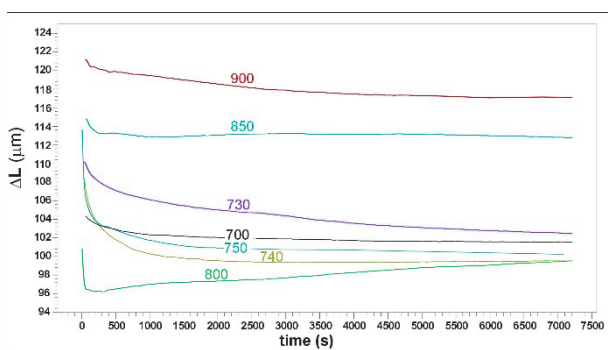


**Fig. 3** Dilatometric curve  $\Delta L = f(T)$  and differential curve  $d(\Delta L)/dT = f(T)$  recorded during heating of sample to 900 °C with the rate of 100 °C/s

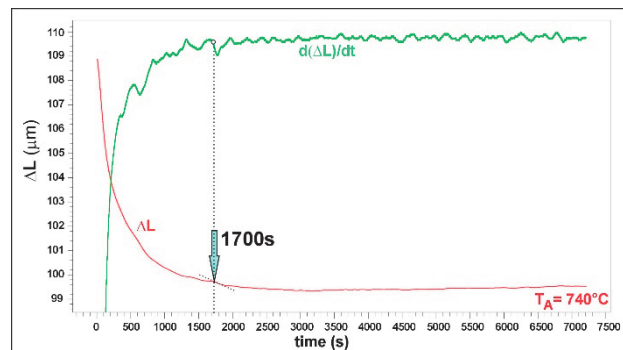
The determination both the start ( $Ac_{1s}$ ) and finish ( $Ac_{1f}$ ) temperatures of pearlite to austenite transformation was possible on the dilatogram diagram recorded for the sample heated up to 900 °C (**Fig. 3**). They were respectively: 760 and 840 °C. It should be noted that even after heating to such high temperature it is still not possible to unequivocally determine the  $Ac_3$  i.e. the temperature, at which the ferrite to austenite transformation should be finished. Based on analyses of microstructure (**Fig. 2f**) as well as a dilatometric and differential curves in **Fig. 3**, it can be only guessed that this temperature was 900 °C or slightly higher.

### 3.3. Dilatometric analysis of holding curves

All dilatometric curves which were recorded during isothermal annealing in the temperature range 700 ÷ 900 °C are summarized in **Fig. 4**. Whereas an example of dilatogram, recorded during the isothermal holding of the investigated steel samples at 740 °C is shown in **Fig. 5**.



**Fig. 4** Elongation changes of 41MnSi6-5 steel samples ( $\Delta L$ ), recorded during isothermal holding in 700 ÷ 900 °C temperature range



**Fig. 5** Dilatometric curve  $\Delta L = f(t)$  and differential curve  $d(\Delta L)/dt = f(t)$  recorded during isothermal holding at 740 °C for 7200 s

As seen in **Fig. 4**, a noticeable contraction, occurred in the initial stage of isothermal holding of all samples. Within the temperature range 700 ÷ 740 °C the observed shrinkage is bigger and occurs the faster the higher is the temperature. This can indicate that a diffusion process occurs in the samples microstructure in such temperature range. More detailed analysis of dilatograms reveals small but significant changes after approx 2700 s (at 730 °C) and approx 1700 s (at 740 °C - see **Fig. 5**). These could be the result of the austenite formation, although it is not confirmed by the results of metallographic examinations (**Fig. 2b and 2c**). Therefore, it was decided to make the final settlement of these uncertainties only after analysis of dilatograms, which were recorded during quenching from these temperatures (see Subchapter 4.4).

Analysis of elongation changes of the sample which was annealed at 750 °C, led to a similar conclusion. However, a new structural component was easily found in the microstructure of this sample (**Fig. 2d**). Therefore, it can be assumed that the noticed dilatation effect recorded during isothermal holding at 750 °C were caused not only by the pearlitic cementite coagulation but also by the start of the pearlite to austenite transformation.

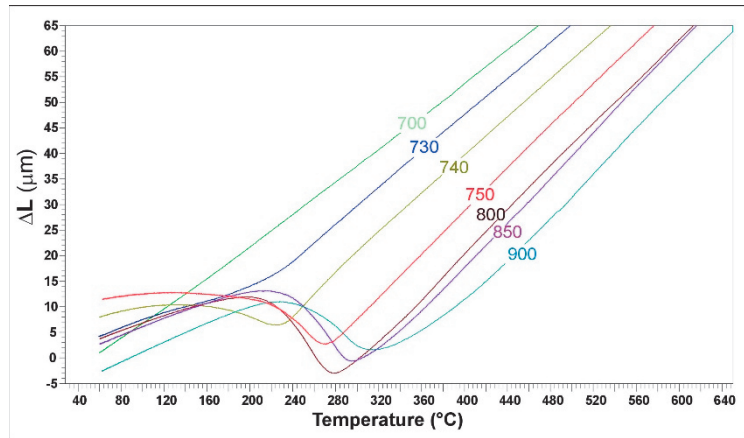
Dilatometric curves recorded during the isothermal holding of samples in the range 800 ÷ 900 °C, do not differ in their nature from the previously discussed curves (**Fig. 4**). However, for these curves initial shrinkages can not be responsible the coagulation process because the pearlite to austenite transformation finished already during the heating (**Fig. 2 and Fig. 3**). Therefore, it can be assumed that this dilatation effect corresponds probably to the homogenization process and increase of austenite grains sizes. After a longer annealing time further changes in elongation curves inclinations were observed. For sample annealed at 800 °C these changes occurred after approx. 700 s. For the sample annealed at 850 °C it was approx 350 s. Such determined

time, in combination with metallographic analyses may therefore indicate the moments when the perlite (at 800 °C) and ferrite (at 850 °C) to austenite transformations was finished.

### 3.4. Dilatometric analysis of cooling curves

The purpose of cooling the samples at the constant rate of 100 °C/s was to achieve the martensitic transformation of austenite, which was formed in the microstructure of samples during the earlier stages of the heat treatment. The formation of martensite confirms clearly, a positive dilatation effects on the dilatometric cooling curves, which are well visible in **Fig. 6**.

As can be seen, during the cooling the sample from the lowest temperature (700 °C) a linear relationship between temperature and elongation was obtained which proves that during the cooling process none transformations occurred. Comparing these results with the microstructure (**Fig. 2a**) as well as heating and isothermal holding dilatograms (**Fig. 3 and Fig. 5**) it can be reasonably stated that austenite is not formed in the 41MnSi6-5 steel microstructure during annealing at 700 °C for 7200 second.



**Fig. 6** The effect of the isothermal holding temperature on the magnitude of dilatation caused by the martensitic transformation

A positive dilatation effect, which proves that the martensitic transformation occurred, was observed in the other dilatometric curves during cooling from the higher temperatures. It is worth to notice, that the curve of cooling from 730 °C shows the very weak but clear effect of dilatation. Temperature at which it occurred was determined as 235 °C. Such low  $M_s$  temperature and a small changing of the angle of the curve inclination below this temperature may indicate that a small amount of austenite was created in the sample microstructure during the isothermal holding. However, most likely, such austenite was highly enriched in carbon.

The similar situation occurred in the sample cooled from 740 °C. However, in this case slight changes in an elongation could be detected on the isothermal holding curve (see **Figs. 4 and 5**). In **Fig. 2c**, it is also difficult to see areas that could be considered as martensite (former austenite). However a clear effect of dilatation caused by martensitic transformation found on the cooling curve confirms that in this sample a noticeable amount of austenite was formed.

Progressive increasing of the isothermal holding temperature up to 900 °C resulted in appearing of the martensitic transformation effect during cooling at much higher temperatures (**Fig. 6**).

Results of dilatometric studies summarized in **Fig. 6**, made possible to determine the effect of annealing temperatures on the  $M_s$  temperature. This relationship is shown in **Fig. 7a**. It is a confirmation the conclusions of the investigations described in previous subchapters, concerning more advanced process of the austenite formation with increasing of the annealing temperature. The observed increasing of the  $M_s$  temperature can also indicate that the carbon concentration in the formed austenite decreased.

Additional tests were performed for the annealing at a temperature of 750 °C. During these investigations the holding time effect on the  $M_s$  temperature was evaluated. The results of these studies are summarized in **Figure 7b**. Surprisingly, the effect of the martensitic transformation was observed in the cooling dilatogram of

the sample, which was annealed at 750 °C for only 10 seconds. These results indicates that 10 seconds is already enough to forming a highly enriched carbon austenite at 750 °C.

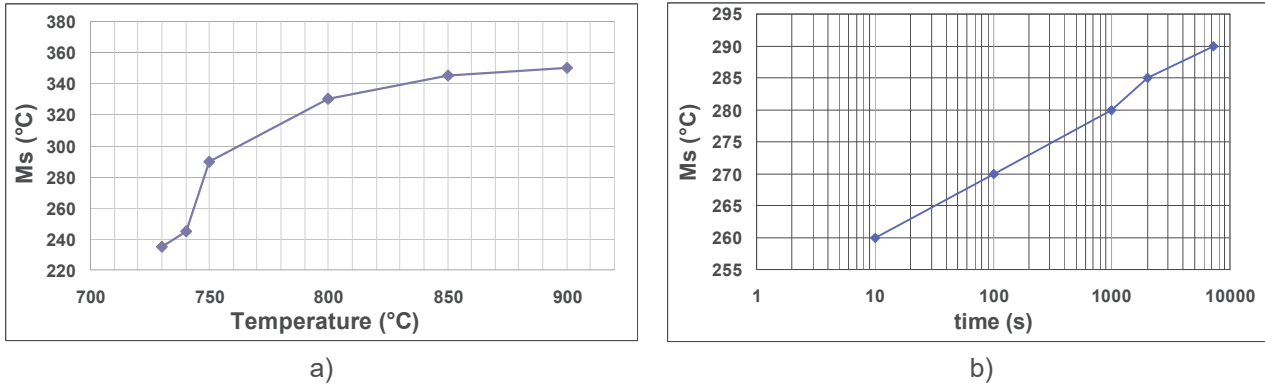


Fig. 7 Relationship between the Ms temperature and annealing parameters: a) Temperature, b) Holding time

### SUMMARY AND CONCLUSIONS

The schematic, isothermal diagram Time-Temperature-Transformations (during austenitising) for investigated 41MnSi6-5 steel is shown in Fig. 8. The drawn up diagram is based on only four pairs of time-temperature coordinates, which were designated on the basis of the analysis of dilatometric holding diagrams. Therefore, these studies were also supported with the annealed microstructure analysis, and the observations and conclusions from the dilatometric analysis of dilatation changes during heating and cooling. Only such comprehensive interpretation of all data made it possible to develop the diagram in Fig. 8.

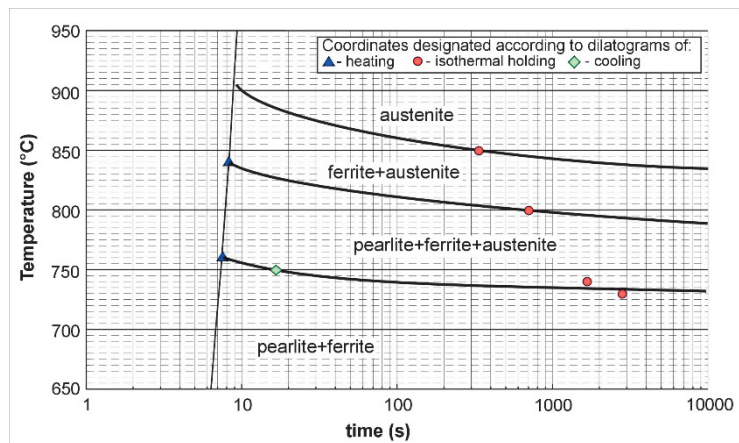


Fig. 8 Isothermal time-temperature-transformation (IHT) diagram for 41MnSi6-5 steel

Although the shown IHT diagram is incomplete (from the side of the shortest time) and will require additional research it allows to specify how is the influence of annealing parameters on the kinetics of the austenite formation. After supplement, the diagram can become a useful tool for design and optimization of heat treatment parameters of 41MnSi6-5 TRIP steels.

Regardless of the developed diagram, based on the carried out investigations the following conclusion can be drawn:

1. At designing the heat treatment of TRIP steels the fact that the pearlite to austenite transformation does not occur at a constant temperature  $Ac_1$ , but within a certain range of temperatures, which is restricted by  $Ac_{1s}$  and  $Ac_{1f}$ , should be considered.
2. Lowering the intercritical annealing temperature from  $Ac_1-Ac_3$  to  $Ac_{s1}-Ac_{1f}$  range can contribute to the increasing carbon concentration in formed austenite. This way lowering of the  $M_s$  temperature of such austenite can be obtained, which gives a chance to an additional increase of the volume fraction of the retained austenite after the finish of the heat treatment of TRIP steels.

3. Shortening the time of such annealing can cause a similar effect. However, at changing this annealing parameter, one should exercise caution in order to ensure the formation of such austenite quantity that the expected microstructure will be obtained, after the later stages of the TRIP steels heat treatment.

## ACKNOWLEDGEMENTS

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