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FORMATION OF MEDIUM CARBON TRIP STEEL MICROSTRUCTURE DURING ANNEALING IN THE INTERCRITICAL TEMPERATURE RANGE

KSZTAŁTOWANIE MIKROSTRUKTURY ŚREDNIOWĘGŁOWEJ STALI TYPU TRIP PODCZAS WYŻARZANIA W ZAKRESIE TEMPERATUR KRYTYCZNYCH

The paper presents the results of research conducted on austenite formation in the microstructure of 41MnSi6-5 TRIP steel during annealing in the intercritical temperature range. The influence of the annealing temperature on the volume fraction of retained austenite in the microstructure of the investigated steel after water quenching was also determined.

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 Ac_1 , but rather within some, possible to determine, temperature range which is bounded by the values Ac_{1s} and Ac_{1f} .

Moreover, through X-ray analysis, it was stated that the largest amount of retained austenite remained in the samples which were annealed at the lowest temperatures in the Ac_{1s} - Ac_{1f} range prior to quenching. Increasing the annealing temperature to a two-phase $\alpha + \gamma$ (ferrite + austenite) range, resulted in a decrease of the volume fraction of retained austenite.

It was also found that during annealing in Ac_{1s} - Ac_{1f} temperature range, austenite is also formed from ferrite simultaneously. This could be the reason for the decrease the carbon content in the formed austenite and consequently the decrease in the volume fraction of retained austenite in the microstructure of the investigated steel, which was quenched after having reached temperatures higher than $Ac_{1s} + 30^\circ\text{C}$.

Keywords: phase transformations, critical temperatures, retained austenite, TRIP steels

W pracy przedstawiono wyniki badań nad tworzeniem się austenitu w mikrostrukturze stali 41MnSi6-5 typu TRIP podczas wyżarzania w zakresie temperatur krytycznych. Określono również wpływ temperatury takiego wyżarzania na udział objętościowy austenitu szczątkowego, jaki pozostaje w mikrostrukturze badanej stali po zahartowaniu od takich temperatur.

Na podstawie wyników analizy dylatometrycznej oraz badań metalograficznych stwierdzono, że w badanej stali przemiana perlit – austenit nie przebiega w stałej temperaturze określanej jako Ac_1 lecz w pewnym, możliwym do określenia jej zakresie, którego granice wyznaczają wartości Ac_{1s} i Ac_{1f} .

Ponadto, metodą analizy rentgenowskiej wykazano, że najwięcej austenitu szczątkowego pozostawało w tych próbkach z badanej stali, które przed zahartowaniem były wyżarzane przy najniższej temperaturze z zakresu Ac_{1s} - Ac_{1f} . Podwyższenie temperatury wyżarzania do zakresu dwufazowego $\alpha + \gamma$ (ferryt+ austenit) było przyczyną zmniejszenia udziału austenitu szczątkowego.

Stwierdzono, że podczas wyżarzania w zakresie temperatur Ac_{1s} - Ac_{1f} tworzy się również z ferrytu. Mogło to być przyczyną zmniejszenia zawartości węgla w tworzącym się austenicie i spadku udziału austenitu szczątkowego w mikrostrukturze badanej stali po jej zahartowaniu od temperatur wyższych od $Ac_{1s} + 30^\circ\text{C}$.

1. Introduction

Steels of a multiphase microstructure with mechanically unstable retained austenite are known as the TRIP (*TR*ansformation *I*nduced *P*lasticity) steels [1-3]. In the contemporary automotive industry, due to a very favourable relation between their strength, plasticity and production costs, these materials can still compete with Al and Mg alloys and even with composites. It can be assumed that these universal properties of steels are caused by the presence of metastable austenite in their microstructure. The presence of this phase in the microstructure facilitates forming elements made of the

TRIP steel. In addition, a possibility of a mechanical destabilisation of retained austenite and its transformation to martensite causes that during forming process they are becoming strengthened [4,5]. Thus, in consequence, after finishing the technological process, elements produced of the TRIP steel can preserve high strength at a reduction of their cross-section [6-10].

Usually the fraction of unstable retained austenite in the microstructure of the TRIP type steel, in dependence of its chemical composition, is within a range from 5 to 15 vol.% [11]. Since these are rather low-carbon and less often middle-carbon steels leaving in their microstructure such

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large volume fraction of retained austenite can be not easy. On account of this reason, investigations aimed at increasing this phase volume fraction in the TRIP steels, are still conducted [12-16].

The most often these investigations concern optimisation of the heat treatment technology of TRIP steel. Usually such treatment is of a multistage character, and one of the most important stages is annealing in the intercritical temperature range. The purpose of such annealing is the formation of carbon-saturated austenite. The second, also important stage of such treatment, which occurs during cooling after annealing, is based on an isothermal temperature holding of steel in the bainitic transformation range. That time, a further enriching with carbon of not transformed austenite occurs, due to which this phase remains as retained austenite in the microstructure of the heat treated steel. A majority of the mentioned previously research on increasing the retained austenite volume fraction is focused on modifications of an isothermal holding in the range of the bainitic transformation. Whereas, a potential of the TRIP steel annealing in the intercritical temperatures range remains not exploited fully. According to the authors of the hereby paper, it is possible – during such annealing to form the more favourable microstructure of these steels, which can give a chance of remaining a larger than ever volume fraction of retained austenite in the microstructure of the TRIP steel.

2. Material and experimental procedure

The chemical composition of the steel under investigation, expressed in wt.% was: 0.41%C, 1.52%Mn, 1.22%Si, 0.015%S, 0.015%P, 0.02%Cr, 0.02%Ni, 0.05%Cu, 0.015%Al, bal Fe. Based on its chemical composition this steel was marked as 41MnSi6-5. The microstructure of the investigated steel in as-delivered condition is presented in Fig. 1. It is easy to notice that is hypoeutectoid steel with a significant, nearly 85 vol.% pearlite fraction.

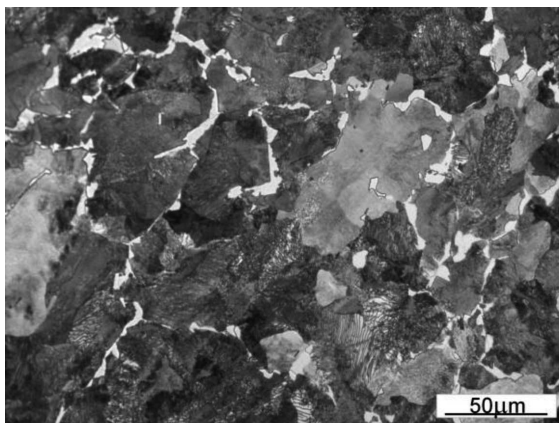


Fig. 1. The microstructure of 41MnSi6-5 steel in as-delivered condition

Dilatometric tests were performed as the preliminary stage of experiments. The tested steel sample of dimensions $\phi 2 \times 12$ mm was placed in the dilatometer DT1000 of the Adamel Company and continuously heated to 1200°C, with a rate of 3°C/min. During this heating, the sample relative elongation $\Delta L/L_0$ was recorded as the temperature T func-

tion. The critical temperatures of the austenite formation were determined from this dependence (see Table 1).

TABLE 1

Symbols and description of critical temperatures which are applied in the hereby paper, acc to [17]

Symbol	Process description	Changes in microstructures during heating
Ac_{1s}	Start of the pearlite \rightarrow austenite transformation	<i>First precipitates of austenite (γ) occur</i>
Ac_{1f}	Finish of the pearlite \rightarrow austenite transformation	<i>Last precipitates of pearlite (pearlitic cementite) disappear</i>
Ac_3	Finish of the ferrite \rightarrow austenite transformation	<i>Last precipitates of ferrite (α) disappear</i>

During the next stage of investigation the samples (12×10×10 mm) were heated within the determined intercritical temperatures range (730÷870°C). After holding at the selected temperature (30 min.), in order to fix areas, in which austenite could be formed during such annealing, samples were water cooled causing the transformation of possible austenite to martensite.

Metallographic microsections were made on the heat treated samples and by means of the X-ray quantitative phase analysis the assessment of the retained austenite volume fraction was performed. These studies were carried out using the X-ray diffractometer TuR-M61 applying filtered radiation of the cobalt lamp ($\lambda_{K\alpha} = 1.7902 \text{ \AA}$) and recording reflections 111_γ and 110_α .

Directly after X-ray tests, to reveal structural components, metallic microsections of the samples were etched using 2% nital. The revealed microstructures were observed by the light microscope Axiovert 200MAT and the scanning microscope FEI Nova NanoSEM 450. The volume fractions of visible microstructural components were determined using manual procedures of quantitative metallography (point-count method – lattice variant).

The Vickers hardness (at a load 30 kg) was measured on all heat treated samples. To verify the identified microstructural components on the sample annealed at 750°C, additional measurements of microhardness were performed. The Knoop indenter was applied at a load of 10 g.

3. Results and discussion

The dilatometric curve $\Delta L/L_0$ recorded during the sample heating as well as the corresponding differential curve $d(\Delta L/L_0)/dT$ are presented in Fig. 2.

The currently applied procedure of the annealing temperature of the TRIP steel in the range of critical temperatures assumes performing this operation in the two-phase range: $\alpha + \gamma$, it means directly after finishing the austenite formation from carbon-rich pearlite and directly after starting the ferrite to austenite transformation [2, 18]. Temperatures limiting this range are referred to, in the literature, as Ac_1 (the constant temperature of the pearlite transformation to austenite) and Ac_3 (the transformation finish temperature of ferrite to

austenite) and the most often are calculated on the bases of equations [19].

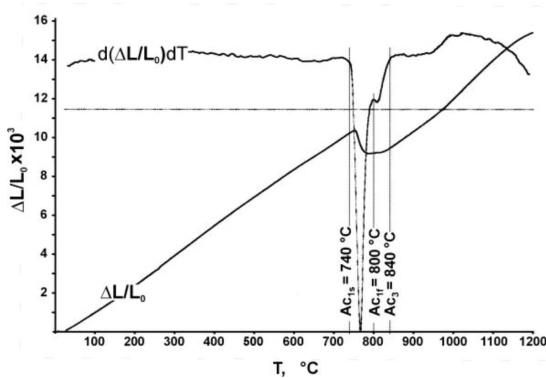


Fig. 2. Dilatometric curve of heating $\Delta L/L_0 = f(T)$ with corresponding differential curve $d(\Delta L/L_0)/dT = f(T)$ made for 41MnSi6-5 steel

However, the results of dilatometric investigation indicate that in contrast to pure Fe-C alloys, the pearlite transformation to austenite in steels does not occur at the constant temperature A_{c1} but within a certain temperature range bounded by values of A_{c1s} and A_{c1f} (compare Table 1). It is difficult to indicate implicitly the ferrite transformation to austenite start temperature, while this transformation finish temperature is simple to be determined (A_{c3}). Based on the analysis of curves from Fig. 2 these temperatures – for the tested steel – were determined as: $A_{c1s} = 740^\circ\text{C}$, $A_{c1f} = 800^\circ\text{C}$ and $A_{c3} = 840^\circ\text{C}$. The difference between A_{c1s} and A_{c1f} seems to be a quite high however, such results correspond to the results of studies on steels of similar chemical composition with increased manganese content [20].

The small difference between temperatures A_{c1f} and A_{c3} (only 40°C) draws attention. On the one hand it can indicate

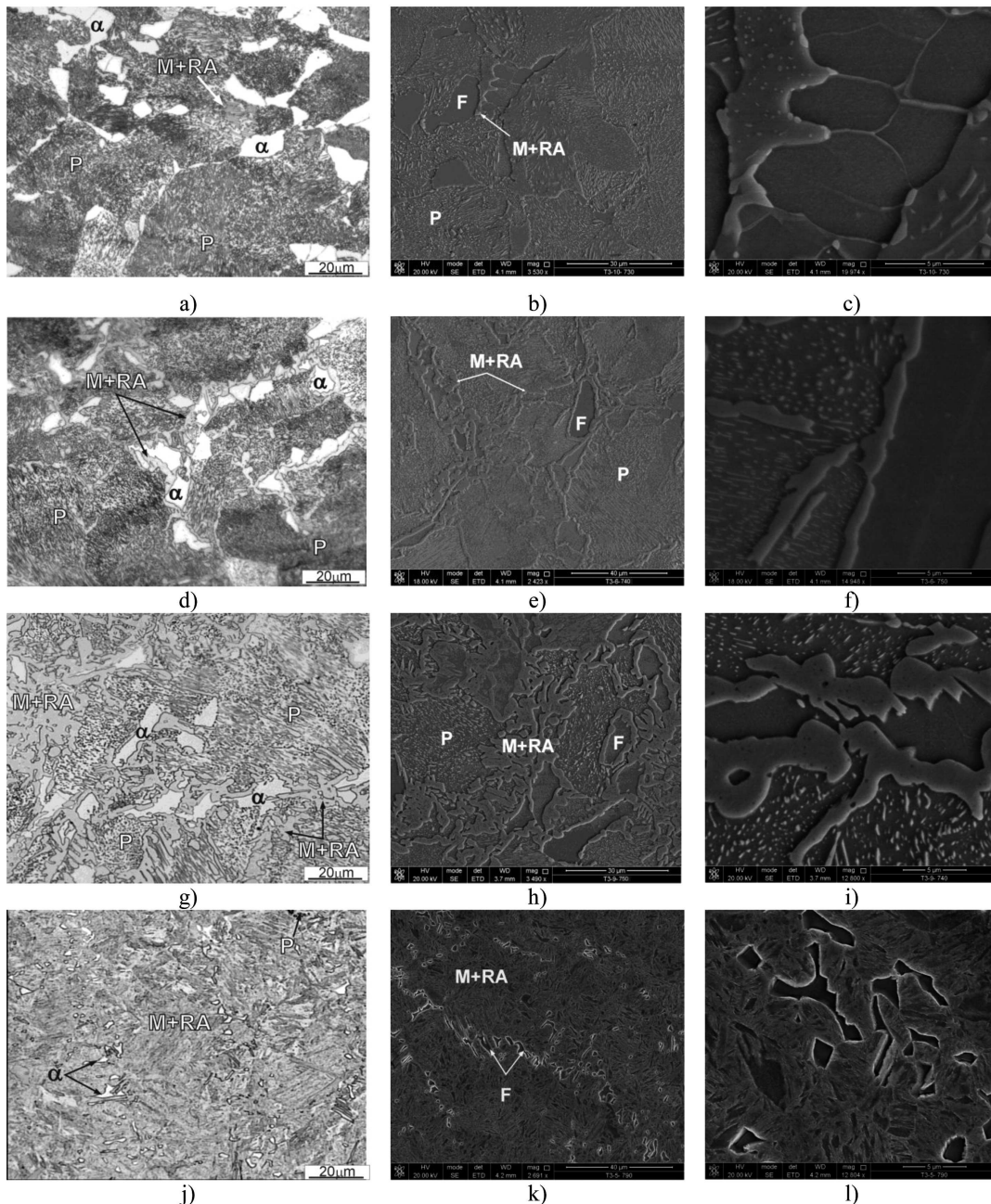


Fig. 3. The changes in the microstructure of the 41MnSi6-5 steel quenched from 730°C (a, b, c), 740°C (d, e, f), 750°C (g, h, i) and 790°C (j, k, l): α – ferrite, P – pearlite, M+RA – martensite (with retained austenite)

a small volume fraction of ferrite, which in this temperature range can be transformed to austenite. On the other hand, it can not be excluded that during annealing of the tested steel – being still in the course of the pearlite transformation to austenite (in the temperature range: $Ac_{1s} \div Ac_{1f}$) – a part of ferrite can be transformed to austenite [21].

The most interesting changes in the microstructure of the tested steel with the annealing temperature are seen in Fig. 3.

The microstructure of the sample annealed at 730°C, presented in Fig. 3, is composed of pearlite and ferrite and is nearly the same as the microstructure in the initial state (compare Fig. 1). However, more detailed analysis of this microstructure (Fig. 3 b and c) allows to find an occasional occurrence of the third structural component. This component was revealed as grey-etched areas – mainly within the pearlite areas. Although very small sizes of these areas renders impossible their explicit identification, it can be assumed that isothermal holding at 730°C for 30 min. caused a local austenite formation, which was transformed to martensite during cooling.

A temperature increase to 740°C (Ac_{1s}) caused a significant increase of areas taken by the grey-etching microstructural component (compare Fig.3 d,e,f). It is interesting that these areas are localised – first of all between pearlite and ferrite grains.

The annealing temperature increase to 750°C caused a further increase of areas taken by the third structural component (Fig. 3 g,h,i). It is not difficult to notice that these areas are mainly in places, which at lower temperatures were taken by pearlite. The size of these areas was sufficiently large to allow their identification by measuring $HK_{0,01}$ microhardness (compare Table 2).

TABLE 2
Detailed results of the Knoop microhardness ($HK_{0,01}$), performed on the sample annealed at 750°C

Microstructural component	$HK_{0,01}$					
	1	2	3	4	5	average
α	373	348	294	354	373	348
P	434	373	378	434	467	417
M+RA	885	867	867	962	787	874

It is seen from the data in Table 2, that microhardness determined for the third structural component is twice as large as microhardness of the remaining – easy for identification – microstructures of ferrite and pearlite. Thus, so high values of $HK_{0,01}$ could be the result of the hard martensite formation in the sample.

At a higher annealing temperature being 790°C (Fig.3 j,k,l), products of martensitic transformation dominate already in the microstructure of the tested steel. Pearlite occurs only incidentally – in places, which in the light microscope were difficult to be noticed or to be explicitly identified.

The comparison of microstructures of samples annealed at 750 and 790°C allows to state, that simultaneously with the progressing pearlite transformation to austenite and vanishing of pearlite areas, bright-etching ferrite areas are significantly decreased (compare Fig.3 g and 3 j). Thus, it confirms the thesis, mentioned in the discussion of dilatometric tests, con-

cerning the possibility of starting the ferrite transformation to austenite below the Ac_{1f} temperature, it means still prior to finishing the pearlite-to-austenite transformation.

Such austenite in samples annealed at 740 and 750°C was rich in carbon since it was mainly formed due to the pearlite transformation. On the other hand after annealing at higher than 750°C temperatures vanishing of ferrite areas in the microstructure was observed.

After annealing the samples at the temperature range: 810÷870°C, it means at temperatures higher than Ac_{1f} , none significant changes in their microstructure were found. It can be only mentioned that further vanishing of ferrite areas, for which the ferrite-to-austenite transformation was responsible, was observed up to 830°C.

The influence of the annealing temperature on the retained austenite volume fraction remaining in the microstructure of the investigated steel after quenching is shown in Fig. 4.

It can be easily noticed that the largest, close to 9 vol.% volume fraction of this phase was found in the sample annealed at the lowest temperature in this range, i.e. at 740°C (Ac_{1s}). Slightly smaller volume fraction of this phase was found in the samples annealed at 750°C (approx. 7.5 vol.%) and 770°C (approx. 7 vol.%). Meanwhile, in the microstructure of the samples which were annealed before the quenching in the two-phase $\alpha + \gamma$ range (810 and 830°C) significantly less austenite (about 2 vol.%) remained. Continuation of raising the temperature from which the samples were quenched did not contribute to changes in the volume fraction of this phase, which remained close to 2%. These findings were confirmed by hardness (HV30) measurements on the same samples.

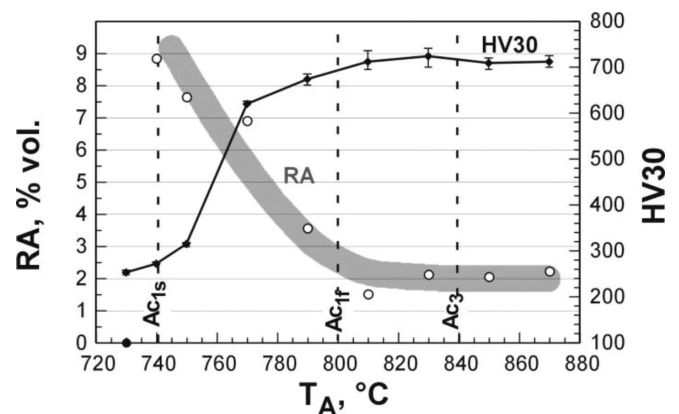


Fig. 4. Influence of annealing temperature on the volume fraction of retained austenite (RA) and hardness (HV30) of the 41MnSi6-5 steel

The results of the metallographic and X-ray analysis were used to construct the diagram presented in Figure 5. It shows how the volume fraction of the main structural components of the microstructure of investigated steel was changing with the annealing temperature.

As it can be seen in Figure 5, the greatest changes in the volume fraction of structural components occur during their annealing between Ac_{s1} and Ac_{1f} temperatures, i.e. in the pearlite-to-austenite transformation range. Annealing of the investigated 41MnSi6-5 steel at temperatures corresponding to the initial stage of such transformation, i.e. 740÷770°C ($Ac_{1s} + 30^\circ C$) contributes to retain the largest amounts of austenite in the microstructure after quenching. However, as

seen from Figures 4 and 5, the volume fraction of that phase in the microstructure of the investigated steel samples, which before the quenching were annealed in the typical for the TRIP steels way in the two-phase $\alpha + \gamma$ range (between Ac_{1f} and Ac_3), was minimal.

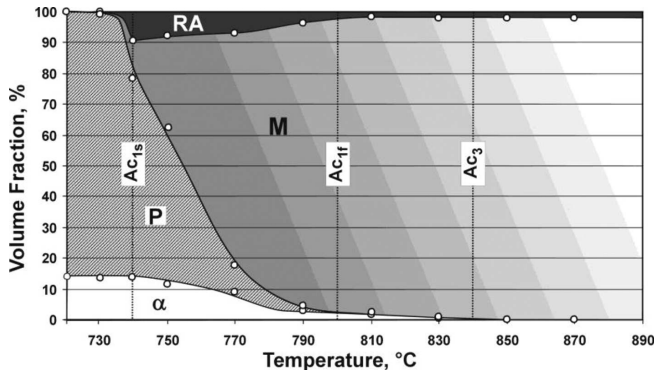


Fig. 5. Influence of the annealing temperature on the volume fraction of: ferrite (α), pearlite (P), martensite (M) and retained austenite (RA) in the microstructure of the investigated 41MnSi6-5 steel

One can assume that the noted difference in the volume fraction of retained austenite remaining after quenching is caused by different carbon contents in austenite formed at different annealing temperatures. During annealing at lower temperatures (740-770°C) austenite is formed only from pearlite. Whereas, when the annealing temperature was 770°C or higher, the vanishing of the ferrite area in the microstructure of investigated steels yet before the end of pearlite-to-austenite transformation (see Fig. 3 and 5) was observed. This phenomenon was probably caused by the transformation of this phase to low-carbon austenite, which could "take over" carbon from the austenite formed from pearlite. As a result of such carbon migration, with increasing of the annealing temperature from 770 to 840°C, the progress of the ferrite-to-austenite transformation increased. This could contribute to decreasing the average carbon content of the formed austenite. Subsequent to such changes in the austenite carbon content, the M_s temperature could rise during quenching, which resulted in a greater advancement of the martensitic transformation and in consequence in decreasing the volume fraction of retained austenite in the microstructure of quenched steel (see Fig. 4).

4. Summary and conclusions

Outlined in this paper the results of research on the formation of austenite in the microstructure of 41MnSi6-5 TRIP steel during annealing in the intercritical temperatures range, give the reason to consider the possibility of the verification of used so far, heat treatment technologies of this kind of steel.

In the investigated steel the largest volume fraction of retained austenite was found in the microstructure of the samples, which were annealed at the lowest temperatures (in the Ac_{1s} - Ac_3 range). It was also shown that the ferrite to low-carbon austenite transformation takes place not after the end of the transformation of pearlite to austenite – as it is commonly believed – but runs simultaneously with it. Therefore, according to the authors of this paper, when the new technologies of the TRIP steels heat treatment are designed,

for increasing of carbon content in austenite as well as the final retained austenite volume fraction in the TRIP steel microstructure, the consideration should be given to such modification which would allow to lower the annealing temperature range to: $Ac_{1s} + \max. 30^\circ\text{C}$.

The results set out in this paper indicate only one of the possible modification directions of the heat treatment of the TRIP steels, which could provide an increase of the volume fraction of retained austenite in the microstructure of these steels. The authors are aware, that the correct heat treatment technology of such steels requires that after annealing in the critical temperatures range the cooling with an isothermal holding in the bainite transformation range is performed. During such holding a redistribution of carbon from bainitic ferrite to austenite should occur, which should result in an additional increasing in the volume fraction of retained austenite in the microstructure after of the so-treated steel. And indeed, the use of the complete heat treatment route (i.e. with isothermal holding at 430°C for 10 min after annealing at $Ac_{1s} + 10^\circ\text{C} = 750^\circ\text{C}$) resulted in increasing of volume fraction of retained austenite in the microstructure of 41MnSi6-5 steel up to 25% [22].

REFERENCES

- [1] V.F. Zaky, M.D. Bhandarkar, E.R. Parker, The role of deformation-induced phase transformation in the plasticity of some iron base alloys, *Metallurgical Transactions* **41**, 351-403 (1978).
- [2] Y. Sakuma, O. Matsumura, H. Takechi, Mechanical properties and retained austenite in intercritically heat-treated bainite-transformed steel and their variation with Si and Mn additions, *Metallurgical Transactions A* **22A**, 489-498 (1991).
- [3] M. Mukherjee, S.B. Singh, O.N. Mohanty, Microstructural characterization of TRIP-aided steels, *Materials Science and Engineering A* **486**, 32-37 (2008).
- [4] M. Suliga, Z. Muskalski, The influence of single draft on TRIP effect and mechanical properties of 0.09C-1.57Mn-0.9Si steel wires, *Archives of Metallurgy and Materials* **54/3**, 677-684 (2009).
- [5] S. Wiewiórska, Analysis of the influence of drawing process parameters on the mechanical properties of TRIP-structure steel wires, *Archives of Metallurgy and Materials* **58/2**, 573-578 (2013).
- [6] K. Sugimoto, R. Kikuchi, S. Hashimoto, Development of high strength low alloy TRIP-aided steels with annealed martensite matrix, *Steel Research* **73**, 253-258 (2002).
- [7] E. Doege, S. Kulp, C. Sunderkötter, Properties and application of TRIP-steel in sheet metal forming, *Steel Research* **73**, 303-308 (2002).
- [8] B. Ehrhardt, T. Gerber, Property related design of advanced cold rolled steels with induced plasticity, *Steel Grips* **4**, 247-255 (2004).
- [9] A. Pichler, S. Taint, T. Hebesberger, P. Stiaszny, E.A. Werner, Processing of thin sheet multiphase steel grades, *Steel Research* **78**, 216-223 (2007).
- [10] J. Galán, L. Samek, P. Verleysen, K. Verbeken, Y. Houbert, Advanced high strength steels for automotive industry, *Revista de Metalurgia* **48**, 118-131 (2012).
- [11] B.C. De Cooman (ed.), *Proc. of International Conference on TRIP-Aided High Strength Ferrous Alloys*. Ghent, 2002.

- [12] S.J. Jiao, J. Penning, F. Leysen, Y. Houbaert, E. Aernoudt, Theory of modeling the isothermal austenite grain growth in a Si-Mn TRIP steel, *Steel Research* **71**, 340-344 (2000).
- [13] B.C. De Cooman, Structure – properties relationship in TRIP steels containing carbide-free bainite, *Current Opinion in Solid State & Materials Science* **8**, 285-303 (2004).
- [14] A.K. Srivastava, G. Jha, N. Gope, S.B. Singh, Effect of heat treatment on microstructure and mechanical properties of cold rolled C-Mn-Si TRIP-aided steel, *Materials Characterization* **57**, 127-135 (2006).
- [15] A. Grajcar, H. Krztoń, Effect of isothermal holding temperature on retained austenite fraction in medium-carbon Nb/Ti-microalloyed TRIP steel, *Journal of Achievements in Materials and Manufacturing Engineering* **49**, 391-399 (2011).
- [16] A. Grajcar, R. Kuziak, W. Zalecki, Third generation of AHSS with increased fraction of retained austenite for the automotive industry, *Archives of Civil and Mechanical Engineering* **12/3**, 334-341 (2012).
- [17] B. Pawłowski, Determination of critical points of hypoeutectoid steels, *Archives of Metallurgy and Materials* **57/4**, 957-962 (2012).
- [18] Y. Sakuma, O. Matsumura, O. Akisue, Influence C content and annealing temperature on microstructure and mechanical properties of 400°C transformed steel containing retained austenite, *ISIJ International* **31**, 1348-1353 (1991).
- [19] K.W. Andrews, Empirical formulae for the calculation of some transformation temperatures, *Journal of the Iron and Steel Institute* **203**, 721-727 (1965).
- [20] A. Jedrzejewska-Strach, Influence of magnesium on the kinetics of phase transformations, structure and properties of model structural steel alloys, Ph.D. Thesis, AGH University of Science and Technology, Krakow (1995).
- [21] D. San Martin, T. de Cock, A. Garcia-Junceda, F.G. Caballero, C. Capdevila, C. Garcia de Antres, Effect of heating rate on re-austenitisation of low carbon niobium microalloyed steel, *Materials Science and Technology* **24**, 266-272 (2008).
- [22] A. Kokoszka, Evaluation of the retained austenite mechanical stability in the medium-carbon TRIP steel, *Journal of Achievements in Materials and Manufacturing Engineering* **55/2**, 323-330 (2012).

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