

Effect of isothermal bainitic transformation temperature on retained austenite fraction in C-Mn-Si-Al-Nb-Ti TRIP-type steel

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ABSTRACT

Purpose: The aim of the paper is to determine the influence of isothermal bainitic transformation temperature on a fraction of retained austenite for a new-developed C-Mn-Si-Al-Nb-Ti TRIP-type steel.

Design/methodology/approach: The thermo-mechanical processing was realized in a multi-stage compression test by the use of the Gleeble 3800 thermomechanical simulator. The steel was subjected to six variants of processing with an isothermal bainitic transformation temperature in a range from 250 to 500°C. Identification of phase composition was achieved using microstructure observations and X-ray diffraction. To determine the fraction of retained austenite the Rietveld method was applied.

Findings: The maximum fraction of retained austenite equal up to 16% can be obtained for the temperatures of isothermal bainitic transformation from 400 to 450°C, while the maximum carbon content in the γ phase equal 1.5 wt.% is present at the temperature of 350°C. Below 350°C due to high M_s temperature, the largest grains of retained austenite located in the ferritic matrix transform to martensite. In a temperature range from 350 to 450°C, the M_{sy} temperature has a negative value, stabilizing the retained austenite.

Research limitations/implications: To determine in detail the influence of isothermal bainitic transformation conditions on a fraction of retained austenite, the knowledge of the effect of isothermal holding time is also important.

Practical implications: The obtained microstructures and especially retained austenite fraction dependent on an isothermal bainitic transformation temperature can be useful in optimization of thermo-mechanical processing conditions of C-Mn-Si-Al TRIP-type steels.

Originality/value: Combined colour etching and X-ray diffraction methods were applied for microstructure identification of modern group of TRIP steels predicted to use in the automotive industry.

Keywords: Metallic alloys; TRIP steel; Thermo-mechanical processing; Compression test; Retained austenite

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1. Introduction

Since the nineties of 20th century, automotive industry has reported high demands for steels connecting high strength and plastic properties as well as cold formability. A great contribution in development of new generation of steels was brought by international projects with participation of numerous steel industry companies which goal was to elaborate and produce components from high strength steels, using modern methods of forming and combining body parts and other assemblies of a vehicle [1-3]. Fulfilling the expectations of automotive industry was possible thanks to elaboration of the group of multiphase steels, consisting of soft ferritic matrix containing islands of martensite, bainite or bainitic-austenitic islands. Steels with such microstructure feature with characteristics of a composite material, perfectly combining high strength with required plasticity. They are steels with ferritic-martensitic microstructure DP-type (Dual Phase) [4, 5] and ferritic-bainitic with retained austenite TRIP-type (Transformation Induced Plasticity), undergoing hardening during technological forming of products as a result of martensitic transformation of γ phase [6-10].

These steels are characterized by ferritic-bainitic microstructure with retained austenite, which gradually transforming into martensite during pressing causes strong strain hardening, taking effect with a delay of sheet thinning, and in the conditions of tensile test – delay of neck initiation. It leads to obtaining high strength finished product and high plasticity, allowing application of high technological strains in a production process. These steels most often contain from 0.15 to 0.5% C, 1-2% Mn and up to 2% Si, which nowadays is completely or partially substituted by Al, introduced in the similar concentration [8, 11]. Such combination of chemical composition and proper heat treatment carried out after cold rolling of sheets, results in obtaining from 5 to 15% of retained austenite [6-8]. Heat treatment of TRIP steels consists in annealing of sheets in a range of temperatures between A_{c1} and A_{c3} with successive rapid cooling up to the isothermal bainitic transformation temperature in a range from 300 to 500°C [8, 11, 12]. Obtaining suitable fraction of retained austenite is possible due to the presence of Si or Al, effectively delaying precipitation of cementite during isothermal holding in a range of bainitic transformation.

The final mechanical properties depend upon kinetics of austenite into martensite transformation, which is strictly connected with γ phase stability, mostly dependent on carbon concentration in austenite [12-14], the size and arrangement of particles of this phase as well as its strength and the present state of stress [15, 16]. Thermodynamic stability of austenite is described by the temperature of martensitic transformation start M_s , expressed most often by a well known Andrews formula [17]:

$$M_s = 539 - 423C - 30.4Mn - 17.7Ni - 12.1Cr - 7.5Mo \quad (1)$$

where: C, Mn, Ni, Cr, Mo – mass contents of the elements in the austenite.

Moreover, in the case of multiphase steels, taking into consideration silicon and aluminium is also very essential. The modification of Andrews equation regarding silicon was achieved by Li and others [18], while the effect of aluminium on retained

austenite was a subject of research performed by Mintz [19], who modified the dependence (1) into the following form:

$$M_s = 539 - 423C - 30.4Mn - 17.7Ni - 12.1Cr - 7.5Mo - 7.5Si + 30Al \quad (2)$$

where: C, Mn, Ni, Cr, Mo, Si, Al – mass contents of the elements in the austenite.

On the basis of dependence (2), it can be observed that Si has a small influence on the M_s temperature, as distinct from Al, which has a destabilizing effect on retained austenite. Moreover, preservation of retained austenite in microstructure is favoured by increased concentration of Mn. Unfortunately, the effect of γ phase stabilization obtained by Mn is substantially weaker when comparing to carbon. For this reason, determination of changes of C concentration in γ phase according to the conditions of heat treatment has a fundamental significance for TRIP steels.

In the great number of cases, optimum plastic properties of multiphase steels are obtained in the conditions of heat treatment, being effective with the maximum fraction of retained austenite [20, 21]. However, Matsuda and others [22], basing on the research of 0.1C-1.6Mn-1.1Si steel, revealed that samples heat treated in the conditions assuring maximum participation of retained austenite are characterized with lower total and uniform elongation in comparison with samples consisting of 2% lower fraction of γ phase. It has a close connection with carbon concentration in retained austenite, which was equal 0.96% for the steel with lower participation of γ phase and 0.77% for the steel with maximum fraction of this phase. The result of understated concentration of carbon was a transformation of a part of austenite into martensite already during cooling from the temperature of heat treatment finish, and successively a rapid martensitic transformation in the initial stage of cold plastic deformation. In this regard, the most reliable parameter of mechanical stability of γ phase is considered in recent times to be a product of retained austenite fraction f_γ and carbon concentration c_γ in this phase [12, 23, 24].

There are several methods to determine the fraction of retained austenite, each of them has its own advantages and disadvantages. The most popular are: image analysis [9, 16], X-ray diffraction [12-14], neutron diffraction [21, 25], magnetic method [23, 26] and also the newest technique – EBSD [27]. The application of X-ray diffraction to calculate the fraction of retained austenite gives the advantage over the other methods because the lattice constant of austenite can be calculated, giving at the same time the value of carbon content in austenite.

One of the fundamental solutions to retained austenite's problem was formulated by Averbach – Cohen approach [28]. According to this approach, the volume fraction of the austenite V_A is calculated as following:

$$V_A = \frac{1/n_A \sum \frac{I_A^{hkl}}{R_A^{hkl}}}{1/n_\alpha \sum \frac{I_\alpha^{HKL}}{R_\alpha^{HKL}} + 1/n_A \sum \frac{I_A^{hkl}}{R_A^{hkl}} + V_C} \quad (3)$$

where:

I - integrated intensity of a diffraction line,

hkl, HKL - Miller indices of austenite's and α -phase's diffraction lines, respectively,

n - a number of the diffraction lines, taken to calculations,
 R - a factor, which depends on a chemical composition of a phase being examined, its crystal structure and on a chosen diffraction line, i.e. its Miller indices and an angular position of the maximum of the line,
 V_C - a volume fraction of carbides (if included in calculations),
 A - describes the parameters of austenite,
 α - describes the parameters of α -phase.

The equation (3) is often used in its simplified form [29, 30]:

$$V_A = \frac{1.4I_A}{I_\alpha + 1.4I_A} \quad (4)$$

where:

I_A - integrated intensity of austenite's diffraction lines,

I_α - integrated intensity of α -phase's diffraction lines.

There are several ways of choosing the diffraction lines to be integrated and incorporated in calculations. The most often used are: (200) α , (211) α and (200) γ , (220) γ , (311) γ [30]; (211) α and (220) γ , (311) γ [29]; (211) α and (220) γ [14]; (110) α and (111) γ [13]. Apart from the above mentioned techniques, in which some single diffraction lines are used, there is also a possibility to apply the Rietveld method [31, 32] to determine the weight fraction of retained austenite. The Rietveld method was defined as a method of pattern-fitting structure-refinement, used for a powder diffraction pattern of a crystalline chemical compound. The application of this kind of pattern-fitting method to quantitative calculations of multiphase data, has two important advantages; the first one is that the overlapped diffraction lines can be included in the calculations which – in this particular case - always gives the possibility to analyse the strongest lines of both α and γ phases, i.e. (110) α and (111) γ . The second advantage is concerned with the analysis of the full diffraction pattern; it means that all measured diffraction lines are taken into account and the preferred orientation is minimised [33, 34]. The application of the Rietveld method to quantitative calculations needs an experimental data and a correct structural model of an elementary cell, of every crystalline compound of the polycrystalline sample, to obtain a calculated diffraction pattern. The intensity in every i point of the calculated pattern y_i^{cal} is given, according to [35]:

$$y_i^{cal} = s \sum_k p_k L_k A |F_k|^2 G(\Delta 2\theta_{ik}) P_k + y_i^{back} \quad (5)$$

where:

k - represents the Miller indices for a Bragg reflection,

s - the scale factor,

p_k - the multiplicity factor,

L_k - the Lorentz and polarization factor,

A - an absorption factor,

F_k - the structure factor for K th Bragg reflection,

$G(\Delta 2\theta_{ik})$ - a diffraction line profile function,

P_k - a preferred orientation function,

y_i^{back} - the calculated background intensity at the i th point.

The well-known procedure of the least-squares refinement is applied to obtain the best fit of theoretical data to the observed pattern. In a multiphase mixture, the refinement values of the

scales factors are used to calculate the weight fractions of every crystalline component in the sample, in this kind of steel – the contents of both crystalline components: α and γ phases.

The influence of the temperature and the time of isothermal bainitic transformation on the fraction of retained austenite and carbon concentration in this phase has been researched to a quite high degree in steels produced using conventional heat treatment method [12, 20, 27]. However, regarding techno-economic considerations, production of hot-rolled sheets, connecting hot-working with the direct several-stages heat treatment taking advantage of rolling heat, has an increasing meaning [5, 9, 25]. Beneficial influence of hot plastic deformation on the increase of retained austenite fraction and its mechanical stability is confirmed by numerous investigations [6, 16, 30].

Nevertheless, they are mainly carried out on conventional C-Mn-Si steels and are limited to the influence of isothermal holding time in the selected temperature of bainitic transformation [13, 27, 32]. Yet, there is no detailed information on the impact of the whole temperature range of isothermal holding on the participation of retained austenite and carbon concentration in this phase, particularly for the C-Mn-Si-Al type steel with silicon partially substituted with aluminium and containing microadditions, allowing to take full advantage of thermo-mechanical treatment.

2. Experimental procedure

The examination was performed on C-Mn-Si-Al steel with Nb and Ti microadditions and chemical composition presented in Table 1. In comparison to TRIP-type steels used most often, the elaborated steel is characterized by decreased to 0.87% concentration of silicon, which decrement was compensated by addition of Al. The steel is characterized by high metallurgical purity connected to low concentration of phosphorus and sulphur. The melts were done in Balzers VSG-50 vacuum induction furnace. Liquid metal was cast in the argon shield into ingot moulds with capacity of 25kg. Successively, the ingots were forged into flat bars with 220 mm width and 20 mm thickness, from which cuboidal 15x20x35 mm samples were made.

Table 1.
Chemical composition of the investigated steel

Mass contents, (%)							
C	Mn	Si	Al	P	S	Nb	Ti
0.24	1.55	0.87	0.40	0.010	0.004	0.034	0.023

Thermo-mechanical processing was carried out in Gleeble 3800 simulator, stamping the sample in the form of 15x20x35mm cuboid, in the conditions close to the plane state of strain. In order to decrease the friction between the surface of a sample and the surface of a die, very thin graphite foils were used and additionally both surfaces were covered with nickel based lubricant. Detailed parameters of the thermo-mechanical processing are shown in Fig. 1. The samples were subjected to six variants of processing, for which the temperature of hot-working finish was equal 850°C and the course of cooling in a temperature range from 850 to 600°C was designed basing on the curves of

plastically deformed supercooled austenite transformations, included in [36]. The fundamental goal of the experiment was to diversify the temperature of isothermal bainitic transformation in a range from 250 to 500°C with a gradation of 50°C. The holding time of samples in such temperature was equal 600s.

After the thermo-mechanical treatment was performed, samples for metallographic observations were prepared. Metallographic specimens were prepared in the plane consistent with the direction of compression and responding to the plastic flow of steel. For the purpose of retained austenite identification, etching in 10% water solution of sodium pyrosulphate was applied. Metallographic observations were carried out with the use of Leica MEF 4A light microscope applying magnification ranging from 500 to 1000x.

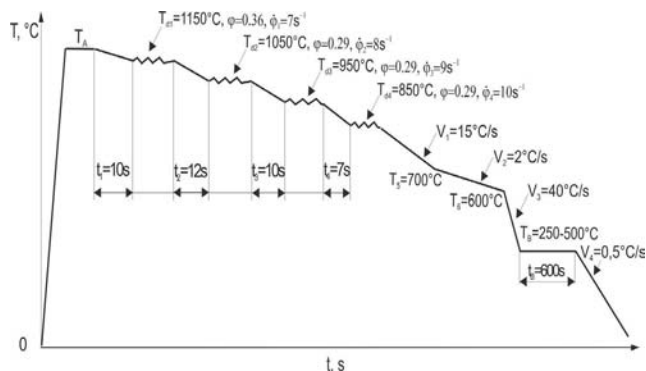


Fig. 1. Parameters of the thermo-mechanical processing realized in the Gleeble simulator

The X-ray diffraction experiments were done using Philips PW 1140 diffractometer with cobalt radiation and a graphite monochromator on a diffracted beam. The phase identification was done according to the data from PDF-4 from International Centre for Diffraction Data. SIROQUANT™ [37] software, in which the Rietveld method was adapted to quantitative calculations in multiphase polycrystalline mixtures, was applied to calculate the fractions of retained austenite. The positions of maxima of the diffractions lines of austenite were used to determine the lattice constant of austenite a_γ . This parameter is necessary to calculate the concentration of carbon in retained austenite. The dependence mostly used for TRIP-type steels was used [29]:

$$a_\gamma = 3.578 + 0.033C_\gamma \quad (6)$$

where:

a_γ - lattice parameter of the austenite (Å),

C_γ - carbon concentration in the austenite (wt. %).

3. Results and discussion

Cooling of samples in controlled conditions in accordance with the scheme shown in Fig. 1 led to obtaining microstructures typical for multiphase steels (Fig. 2). The matrix of steel for each of variants of thermo-mechanical processing consists of ferrite with participation from 45 to 60%. Advantageous influence of

multi-stage compression is primarily visible in fine-grained microstructure of ferrite with average grain size of approximately 10µm.

Bainitic-austenitic islands with various fraction of γ phase and bainite morphology are present in the matrix depending on the isothermal bainitic transformation temperature. Isothermal holding of steel in the temperature of 250°C leads to achievement of retained austenite mainly located on grain boundaries of ferrite (Fig. 2a). They have characteristic polygonal shape and size from 1 to 7µm. Topography typical for martensite can be noted in many grains with shape characteristic for retained austenite. It's also proven by different colour of these grains when comparing to clear laths of bainite located in larger islands. Presence of martensite is a result of martensitic transformation probably occurring during the last stage of cooling of samples to room temperature. The transformation indicates about too low carbon enrichment of austenite due to low isothermal holding temperature. Increase of isothermal bainitic transformation temperature to 300°C doesn't change the character of lower bainite; however participation of martensite definitely decreases (Fig. 2b). Further temperature increase to 350°C doesn't change fraction of retained austenite in a meaningful way. However, apart from grains located in ferritic matrix, certain fraction of retained austenite locates in boundary regions of bainitic islands (Fig. 2c). Considerably higher fraction of γ phase is present after increasing the holding temperature to 400°C. Increase of fraction of retained austenite in a form of fine granules located in bainite is characteristic (Fig. 2d). This phenomenon intensifies after further increase of isothermal bainitic transformation temperature to 450°C, for which the fraction of retained austenite is the highest (Fig. 2e). Increase of temperature to 500°C results in substantial decrease of fraction of retained austenite, what is probably connected to initiation of precipitation of carbides in bainite. Darker regions in Fig. 2f allow also assuming that a part of austenite could undergo pearlitic transformation. In such conditions, stable austenite is present only in bainitic islands.

Determination of retained austenite fraction together with assigning concentration of carbon in γ phase were determined using X-ray quantitative phase analysis basing on the Rietveld method. Figures 3 and 4 present selected X-ray diffraction patterns together with a differential curve for the most essential variants of thermo-mechanical processing. The values close to zero speak for quite good matching of experimental and analytical diffraction patterns. Table 2 presents determined fractions of retained austenite together with the austenite lattice constant, carbon concentration in austenite determined on the basis of the dependence (6) as well as the martensite start temperature of retained austenite calculated using the equation (2).

It arises unequivocally from Fig. 5, that the fraction of retained austenite increases along with the increase of the temperature of isothermal bainitic transformation, reaching maximum 16% for the temperatures of 400 and 450°C. The fraction of retained austenite is strictly connected to the austenite lattice constant, which in turn is dependent on the carbon concentration in γ phase (Fig. 6). The value of lattice constant varies in a quite wide range, from 3.592 to 3.627 Å. From the analysis of Fig. 6 it arises that the small fraction of retained austenite in a range of the isothermal bainitic transformation temperature from 250 to 300°C is connected with too poor carbon

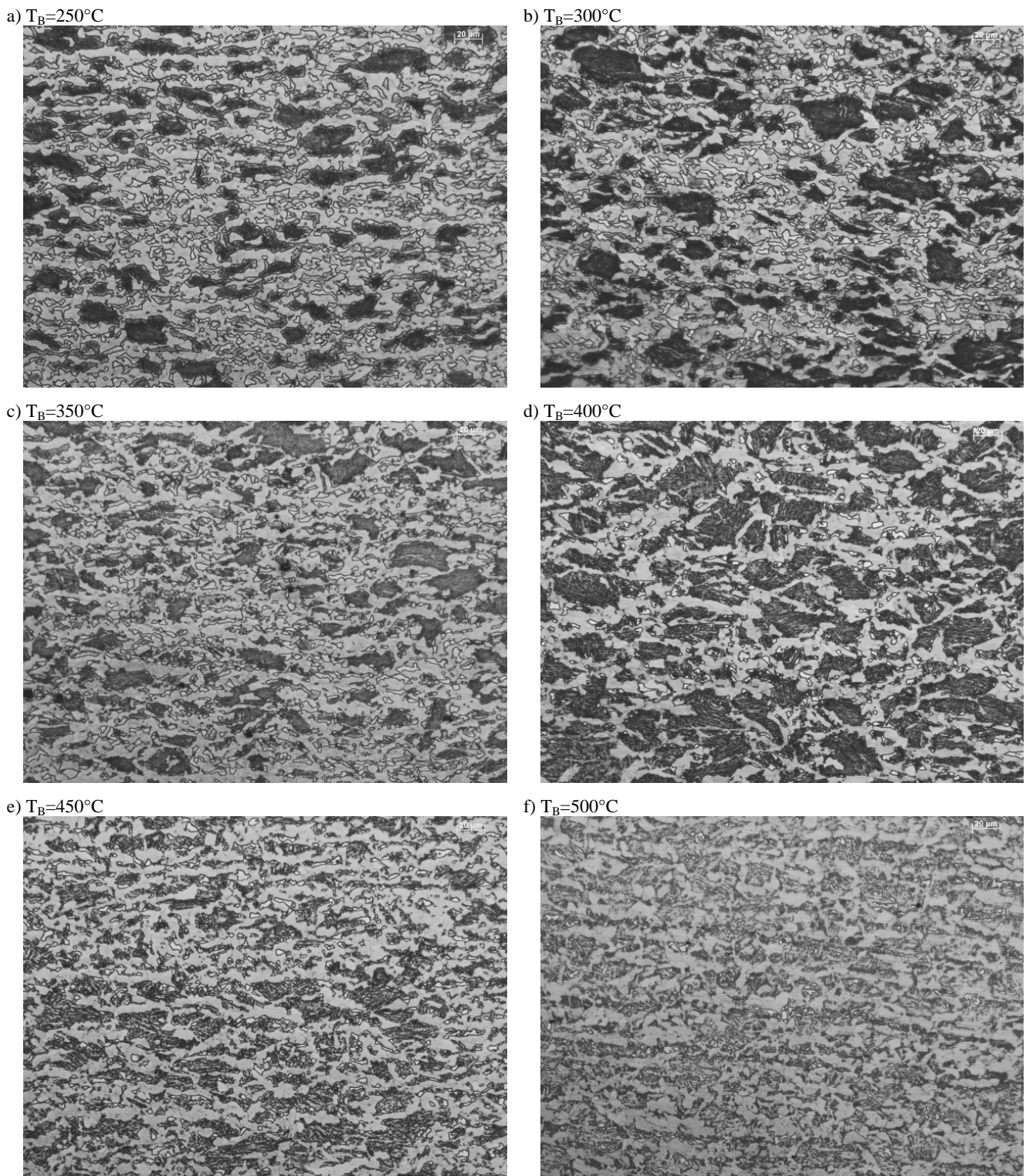


Fig. 2. Ferritic-bainitic structures with various fraction of retained austenite for the specimens isothermally held for 600s at temperatures: (a) 250°C, (b) 300°C, (c) 350°C, (d) 400°C, (e) 450°C, (f) 500°C

Table 2.

Fraction of retained austenite, lattice parameter, carbon concentration in the γ phase and $M_{s\gamma}$ temperature of the retained austenite for the steel isothermally held in the bainitic transformation temperature range from 250 to 450°C

Treatment variant	Austenite fraction, f_γ	Lattice parameter of austenite, a_γ , Å	Carbon content in the austenite, C_γ , wt.%	$f_\gamma \times C_\gamma$, wt.%	$M_{s\gamma}$, °C
850°C–250°C–600s	0.056 ± 0.011	3.5917 ± 0.0063	0.42	0.0235	320
850°C–300°C–600s	0.066 ± 0.014	3.5980 ± 0.0055	0.61	0.0403	239
850°C–350°C–600s	0.096 ± 0.016	3.6274 ± 0.0036	1.50	0.1440	-137
850°C–400°C–600s	0.160 ± 0.013	3.6248 ± 0.0013	1.42	0.2272	-103
850°C–450°C–600s	0.168 ± 0.008	3.6201 ± 0.0009	1.28	0.2150	-44

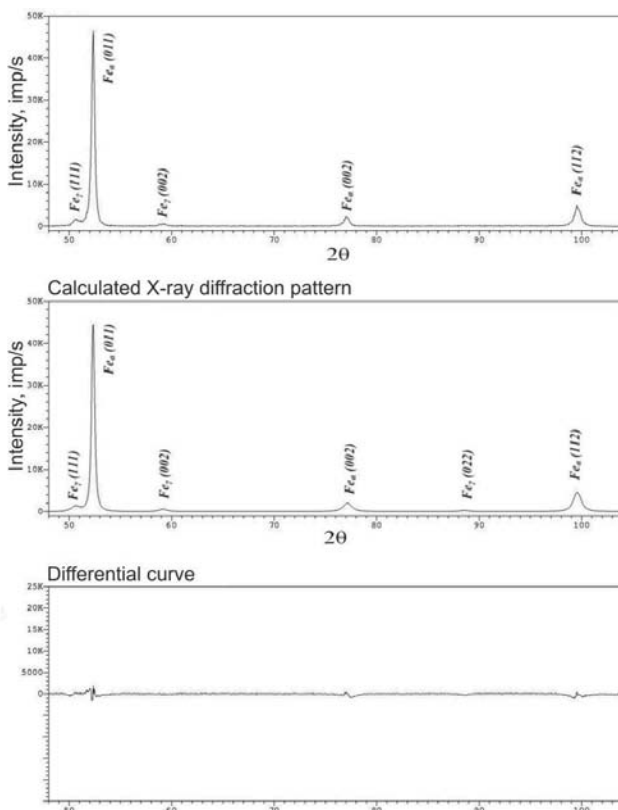


Fig. 3. The experimental (the upper pattern), calculated (in the middle) and difference (the lower curve) patterns of the steel after isothermal holding in temperature of 350°C

enrichment of austenite, up to concentration of approximately 0.6%. The $M_{s\gamma}$ temperature, calculated basing on the above, is considerably higher than room temperature (Table 2), what is consistent with observed grains, which underwent martensitic transformation in the last stage of cooling.

It's interesting, that the highest concentration of carbon in austenite equal 1.5% is present for the temperature of isothermal holding equal 350°C, for which the fraction of retained austenite

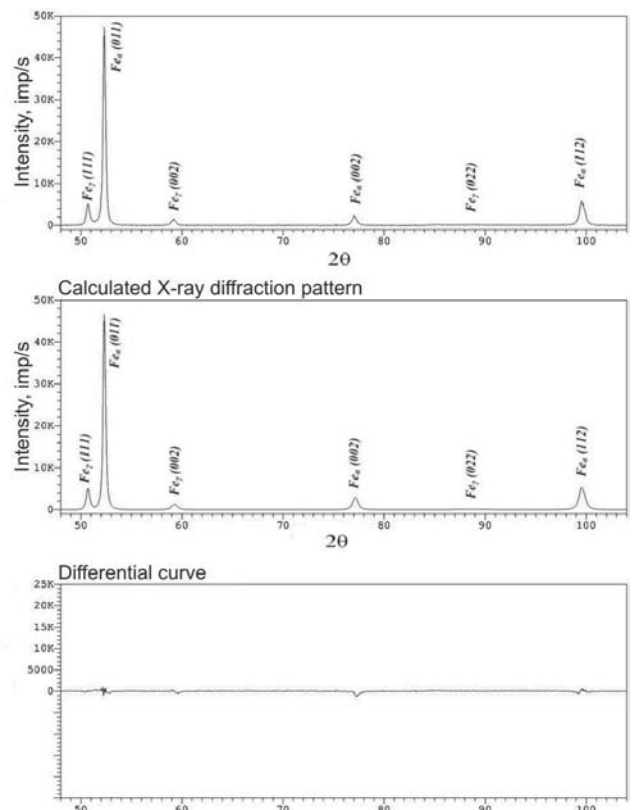


Fig. 4. The experimental (the upper pattern), calculated (in the middle) and difference (the lower curve) patterns of the steel after isothermal holding in temperature of 450°C

receives medium values and is equal around 10% (Fig. 5). It can be explained by heterogeneity of carbon distribution in the austenite grains. It applies in particular to the large grains present in the ferritic matrix; their participation is much higher for samples held at the temperature of 350°C.

Regarding limited diffusion rate of carbon at the temperature of 350°C, the central areas of grains are enriched in carbon to a lower degree in a result of which they undergo bainitic or

martensitic transformation in the final stage of cooling. Retained austenite with high concentration of carbon is then present mainly in the corners of bainitic islands or polygonal grains (Fig. 2c). Along with the increase of the temperature to 400°C, carbon diffusion rate is high enough to enrich austenite grains in a more uniform way, all the more that a large fraction of austenite consists of fine grains located in bainitic islands (Fig. 2d), what is connected to the carbon diffusion for smaller distances. More uniform distribution of carbon favours stabilization of higher participation of austenite to the room temperature. Increase of isothermal holding temperature to 450°C intensifies this process, although obtained fraction of retained austenite equal 16.8% confirms that carbon concentration in austenite equal 1.28% (Table 2) is sufficient for thermal stabilization of a large fraction of γ phase. A lack of martensite in the structure of the specimens isothermally held in a temperature range of 350 to 450°C is due to low values of martensite start temperature, which are considerably below room temperature.

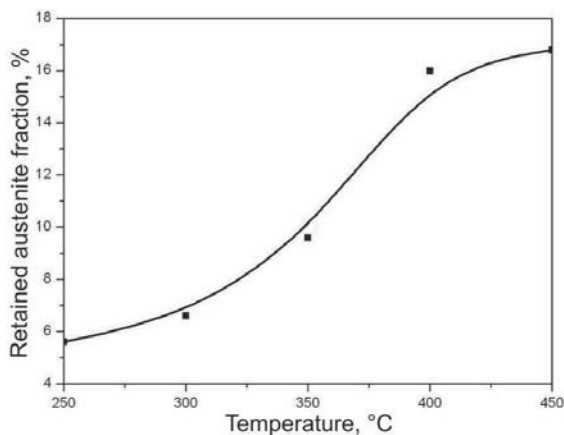


Fig. 5. Influence of isothermal bainitic transformation temperature on a fraction of retained austenite

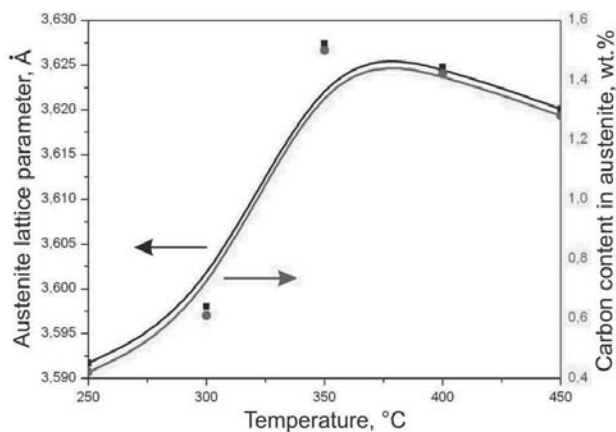


Fig. 6. Influence of isothermal bainitic transformation temperature on the lattice parameter and carbon concentration in the retained austenite

4. Conclusions

Complementary methods of light microscopy and X-ray phase analysis allowed detailed determination of morphological characteristics as well as the fraction of retained austenite in the function of isothermal bainitic transformation temperature for the newly elaborated TRIP-type steel with silicon partially substituted by aluminium, containing Nb and Ti microadditions. It was found that the maximum participation of γ phase equal up to 16% can be obtained for the temperatures of isothermal holding ranging from 400 to 450°C, while the maximum carbon concentration in retained austenite equal approximately 1.5 wt.% is present at the temperature of 350°C. Below the temperature of 350°C relatively slow course of carbon diffusion precludes sufficient carbon enrichment of retained austenite in the time of 10 min, what results in high M_s temperature of γ phase and partial martensitic transformation of large block grains of this phase located in the ferritic matrix. In a temperature range from 350 to 450°C the conditions for retained austenite stabilization are more advantageous, what is reflected by the negative M_s temperature of γ phase and uniform distribution of retained austenite with the fraction of the phase located in bainitic islands, increasing together with the increase of the temperature. Rapid decrease of γ phase fraction occurs at the temperature of 500°C, what is linked to carbides precipitation and to the transformation of supercooled austenite into bainitic-pearlitic regions.

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