Effect of isothermal holding temperature on retained austenite fraction in medium-carbon Nb/Ti-microalloyed TRIP steel

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Manufacturing and processing

ABSTRACT

Purpose: The aim of the paper is to determine the effect of the isothermal holding temperature in a bainitic transformation range on a fraction of retained austenite for a new-developed medium-carbon TRIP steel containing Nb and Ti microadditions.

Design/methodology/approach: The thermo-mechanical processing was carried out by a multi-stage compression test using the Gleeble thermomechanical simulator. The steel was subjected to 5 variants of processing with an isothermal bainitic transformation temperature in a range from 250 to 450°C. Identification of structural constituents was done using microscopic observations and X-ray diffraction. To determine the fraction of retained austenite the Rietveld method was applied.

Findings: It is possible to obtain a high fraction of retained austenite characterized by the high thermodynamic stability in a C-Mn-Si-Al steel containing 0.43% C. The maximal fraction of austenitic phase equal above 20% was obtained for the wide temperature range of isothermal holding from 350 to 450°C. The maximal carbon content in the retained austenite equal 1.84 wt.% is present for the temperature range from 350 to 400°C. Below 350°C due to relatively low carbon diffusivity and high Msγ temperature, a part of austenite transforms to martensite. Above 400°C there is still a high fraction of retained austenite but it contains a lower C content.

Practical implications: The obtained austenite volume fraction and carbon content in a γ phase determined as a function of isothermal holding temperature can be useful in optimization of thermo-mechanical processing conditions for medium-C TRIP steels.

Originality/value: The research was performed on a new-developed medium-carbon Si-Al steel microalloyed with Nb and Ti. There is a lack of data on microstructure and stability of retained austenite in such advanced group of high-strength TRIP steels.

Keywords: Metallic alloys; TRIP steel; Thermomechanical processing; Hot-temperature deformation; Retained austenite; Microalloying; Bainitic steel; Rietveld method

1. Introduction

Automotive industry belongs to one of the branches of the global market which creates continuous demands of constructional materials with higher strength and required formability, especially drawing and stretch forming. A great meaning has also a proportion of strength and density of the material, deciding about the vehicle's mass and therefore fuel consumption and ecological damages caused by emitted exhaust gas. In the period of last twenty years, numerous new groups of steels have been elaborated [1-4], finding their application in automotive industry because of proper selection of chemical composition and technologies assuring the microstructure which guarantees favourable balance of mechanical properties and formability. This combination of properties has a meaning not only in the production process of steel and car parts, especially bodies, but also during possible accident while using the car because of relatively high reserve of plasticity in the zones of controlled energy absorption of car being damaged [5-7].

The most complex influence of diversified processes for increasing the level of mechanical properties occurs for multiphase TRIP-type steels. Their microstructure consists of ferrite, bainite and retained austenite. The role of retained austenite which transforms into martensite during cold-forming is very important, contributing to hardening and improvement of steel ductility by TRIP (TRransformation Induced Plasticity) effect [1-4]. Different chemical composition strategies mainly related to hot dip galvanizing path and cold- or hot-rolled sheets are used, i.e. C-Mn-Si, C-Mn-Si-Al, C-Mn-Al, C-Mn-Al-P-type [1-4,7-14]. Recently, to further increase the mechanical properties of TRIP steels, Mo in the amount up to 0.2% [15,16] and microadditions of Nb, Ti and V [2,11,12,16-20] have been introduced. Simultaneously, for elements which require the high stretch-flangeability the bainitic ferrite or annealed martensite matrix is preferred instead of polygonal ferrite [21-27]. It was found that the lower is the strength difference between the matrix and strain-induced martensite the higher is the stretch-flangeability [23-26]. Strengthening the bainitic ferrite matrix by disperse precipitations of carbonitrides is a further step in increasing both the strength and stretch-flangeability of TRIP steel sheets [26-28].

The mechanical properties of TRIP-aided steels are dependent on the relative fractions of individual phases, and especially a fraction of retained austenite. However, it is interesting to note that the optimal ductility of sheets is not usually obtained for the steels containing the highest fraction of γ phase. The critical factor is the optimal kinetics of strain-induced martensitic transformation strictly related to the mechanical stability of retained austenite. It is in turn mainly dependent on the carbon content in austenite [1,7,10,11,14,15]. Thermodynamic stability of γ phase is defined by the martensite start temperature (M_s). When the carbon content in retained austenite is too low, martensitic transformation occurs already during cooling or in the early stage of technological forming. When it is too high the retained austenite is stable and does not contribute to ductility by TRIP effect. Additional factors influencing the mechanical stability of γ phase are the size and arrangement of particles as well as its strength and the stress state [13-15,17,29].

Results of the determination of the stability of retained austenite for TRIP steels containing conventional C content (i.e. between 0.15 and 0.25 wt.%) are reported by many authors [8,10,11,14,15,17]. However, there are very few reports on mechanical stability of retained austenite for medium-carbon steels [25, 28, 30]. It was observed in [30] that the mechanical stability of retained austenite is not a simple function of C content in steel but this problem is more complex. It is because of the complex morphology of bainitic ferrite and γ phase and additionally it depends on the interactions between all the phases during straining [15,28]. The problem is especially interesting in TRIP steels containing disperse particles of carbonitrides, affecting the stability of retained austenite in different, inverse ways [11,17,26]. In our previous work [28] on mechanical stability of retained austenite in the 0.55C-1.35Si-0.6Mn bainitic steel, two-stage course of the strain-induced martensitic transformation was observed. It is related to the various morphology of γ phase. At the initial stage of the cold deformation the blocky grains of the larger size and lower carbon content are transformed. At the further deformation stage the transformation of films of retained austenite - occurring at the large fraction for TRIP-aided bainitic steels - has taken place. The similar results were observed by other authors [31,32]. Additionally, the more difficult transformation of films of retained austenite can be explained by the protecting vicinity of hard bainite plates [22-27]. Our earlier results on 0.55C-1.35Si-0.6Mn steel [28,33] confirmed that it is possible to obtain TRIP-aided bainitic steel containing over 20 vol. % of retained austenite and characterized by the UTS TEl product over 35000 MPa·%.

It is well known that the fraction of retained austenite and its carbon content are mainly dependent on the temperature and time of isothermal holding of steel in a bainitic transformation range. For example, on the basis of our earlier work [20] on 0.24C-1.5Mn-0.9Si-0.4Al-Nb-Ti steel it was found that the maximum fraction of retained austenite (about 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 $M_{s\gamma}$ temperature has a negative value, stabilizing the retained austenite. The problem is much less investigated in TRIP-aided bainitic steels containing microadditions and it is a main goal of the present work.

Multiphase steels containing microadditions are characterized by high grain refinement. In this regard, the qualitative and quantitative identification of individual structural constituents is especially important. Determination of γ phase volume fraction is most often achieved using computer image analysis after colour etching, X-ray or neutron diffraction and magnetic methods [10,15,20,34,35]. Unfortunately, the optical microscopy has limited resolution, making it unsuitable for observing retained austenite islands which often do not exceed more than 1 μm. Recently, the EBSD technique in scanning electron microscope (SEM) has gathered essential significance for determination of the fractions and morphological features of retained austenite as well as polygonal ferrite, bainitic ferrite and martensite [36,37]. 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 [38]. Another possibility of X-ray investigations to determine the weight fraction of retained austenite is to apply the Rietveld method, described in detail in [20,39-41].

2. Experimental procedure

Investigations were conducted on Mn-Si-Al medium-carbon steel containing Nb and Ti microadditions (Table 1). Compared to the earlier research on TRIP-aided bainitic alloys [28,33] the steel has lower C content and higher Mn, Si and Al contents. Additionally, the steel contains Nb and Ti microadditions and has the high metallurgical purity related to low concentrations of S and P. The melt was done in the Balzers VSG-50 vacuum induction furnace. Liquid metal was cast into ingot moulds in argon shield. Obtained 25 kg ingots were subjected to open die forging in order to get 220 mm wide and 20 mm thick flat bars, from which cuboidal 15x20x35 mm samples were made for the thermomechanical processing tests.

Table 1.
Chemical composition of the investigated steel

Mass contents, (%)							
C	Mn	Si	Al	P	S	Nb	Ti
0.43	1.45	0.98	1.00	0.010	0.004	0.033	0.010

At the initial state (after casting and hot forming) the steel is characterized by the pearlitic-ferritic microstructure with a ferrite halo on the primary austenite grains (Fig. 1). Thermo-mechanical processing was carried out using the Gleeble 3800 simulator, compressing the samples under 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 both surfaces were covered with Ni-based lubricant.
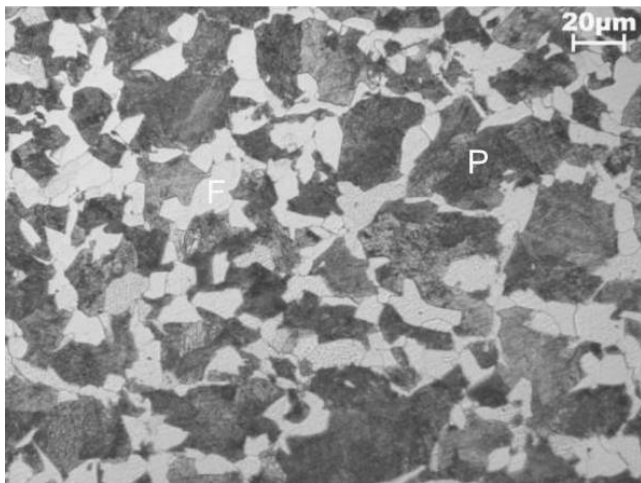

Fig. 1. Pearlitic-ferritic microstructure of the steel in the initial state
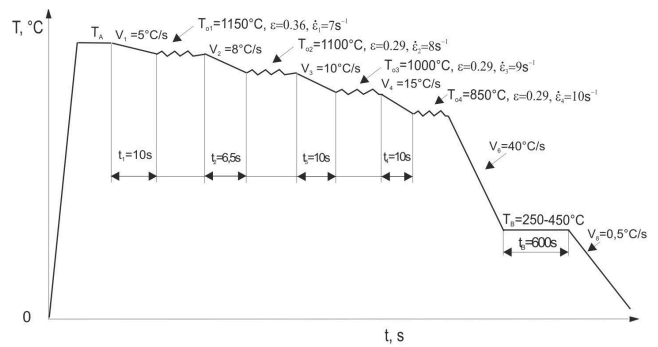

Fig. 2. Parameters of the thermomechanical processing realized in the Gleeble simulator

Detailed parameters of the thermo-mechanical treatment paths are shown in Fig. 2. The samples were subjected to 5 variants of processing, for which the finishing temperature of hot deformation was equal 850°C and the cooling conditions to the isothermal holding temperature were designed basing on the DCCT curves included elsewhere [19]. The fundamental goal of the test was to diversify the temperature of isothermal bainitic transformation in a range from 250 to 450°C with a gradation of 50°C. The holding time of samples was equal 600 s.

After the thermo-mechanical processing was performed, samples for metallographic observations were prepared. The specimens were prepared in the plane consistent with the direction of plastic flow. For the purpose of the best identification of retained austenite, etching in 10% water solution of sodium metabisulfite was applied. Metallographic observations were carried out using Leica MEF 4A optical microscope at the magnification of 1000x. The average fraction of retained austenite (on the basis of 5 micrographs) was determined by the use of the Leica Qwin image analyzer.

The X-ray investigations 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^{TM} [42] 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 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 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. $(110)\alpha$ and $(111)\gamma$. 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 [39,40].

The positions of maxima of the diffractions lines of austenitic phase were used to determine the lattice constant of austenite a_γ. This parameter is necessary to calculate the carbon content in retained austenite. The simple dependence mostly used for TRIP-type steels was used [43]:

$$a_\gamma = 3.578 + 0.033C_\gamma \qquad (1)$$

where:

a_γ - lattice parameter of the austenite ($\mathring{A}$),
C_γ - carbon concentration in the austenite (wt. %).

Martensite start temperature was calculated with taking into account the concentration of Si and Al on the basis of the dependence (2) [44]:

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

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

3. Results and discussion

The result of the thermomechanical processing carried out (according to Fig. 2) are the microstructures in Fig. 3. The plastically-deformed undercooled austenite transforms into bainite-martensite constituents depending on the isothermal holding temperature. A characteristic feature of all the microstructures is the presence of retained austenite of various fraction and morphology. After isothermal holding of steel at the temperature of 250°C a typical martensite/lower bainite microstructure of a distinct lath morphology and a small fraction of non-transformed austenite (about 5 vol.%) was obtained (Fig. 3a). It is in accordance with the DCCT diagram of steel, where the martensite start temperature of steel was evaluated as 300°C [19]. The presence of martensite is a result of martensitic transformation probably occurring during the last cooling stage of samples to room temperature. Increase of the isothermal bainitic transformation temperature to 300°C does not change in a meaningful way the character of a lath martensite/bainite matrix. The fraction of retained austenite is slightly higher (Fig. 3b). Retained austenite occurs as thin laths in bainitic regions or as blocky grains located along primary γ phase grains. Increasing the volume fraction of austenite can be observed with increasing the isothermal holding temperature to 350°C. The austenitic phase is uniformly distributed in the microstructure having usually the characteristic shape of needles dividing individual packets of bainitic laths (Fig. 3c). A relatively high is also a fraction of blocky grains with a diameter up to 4 μm. The thickness of interlath austenite is considerably higher when compared to the specimens isothermally held at lower temperatures.

The highest fraction of retained austenite occurs with the further increase of the isothermal bainitic transformation temperature to 400 and 450°C. The retained austenite is uniformly distributed as a mix of polygonal sharply-edged blocky grains with a size up to 7 μm and interlath regions with a thickness up to 1 μm (Fig. 3d-f). The relative fractions of two morphological forms of γ phase are roughly the same. The morphology of bainite has significantly changed for the specimen held at 450°C. It is similar to the degenerated upper bainite where interlath retained austenite occurs instead cementite between bainitic ferrite laths [16]. A more detailed microstructural analysis of bainite morphology using SEM and TEM will be reported elsewhere.

The effect of the isothermal holding temperature in a range of bainitic transformation is very similar to the results obtained for the steel with a lower carbon content (0.24 wt.%) investigated earlier [20]. The retained austenite fraction increases from 6% to about 17% for the temperature range between 400 and 450°C (Fig. 4). At temperatures higher than 450°C there is a drop in a retained austenite fraction due to carbide precipitation what was confirmed in many works for different steels [13,15,20,21]. The drop in the austenite fraction and carbon content in steels containing over 0.5 wt.% C occurs usually at a slightly lower temperature because of the higher tendency to carbide precipitation. For instance, it was observed in [28,33] for the 0.55C-0.6Mn-1.35Si-0.6Cr steel, where cementite started to precipitate just below 400°C. At 450°C carbides occurred as spherical particles and above 500°C the classical upper bainite with interlath cementite was dominating. Carbide precipitation slows down the carbon diffusion to austenite what has a detrimental effect on the stability of austenitic phase.

Determination of retained austenite fraction and its carbon content were performed using X-ray phase analysis basing on the Rietveld method. Selected X-ray diffraction patterns containing differential curves for the most essential variants of the thermo-mechanical processing are shown in Figs. 5 and 6. The differential curves confirm quite good matching of experimental and analytical diffraction patterns. The highest misfit occurs for the overlapping peaks of Feγ(111) and Feα(110) - Fig. 5. Table 2 presents the determined fractions of retained austenite together with the austenite lattice constant and carbon content in the austenitic phase evaluated on the basis of the dependence (1) as well as the martensite start temperature of austenite calculated using the relation (2). The fraction of retained austenite increases along with increasing the isothermal holding temperature, reaching over 20 vol. % for the quite wide temperature range from 350 to 450°C (Fig. 7). It is important to note that the obtained values are much higher compared to those in Fig. 5, evaluated by means of image analysis. The understated fraction of γ phase is due to a lack of precise identification of fine granules and films of retained austenite being components of bainitic phase. This limitation is not valid for X-ray examination, which results should be acknowledged as the more representative than the image analysis. The consistency between X-ray and image analysis is better for ferrite-based TRIP steels, where a fraction of retained austenite in bainitic islands in much lower [20].

The fraction of γ phase is strictly related to the austenite lattice constant, which in turn is dependent on the C content in retained austenite (Fig. 8). The value of lattice constant varies in a quite wide range, from 3.6051 to 3.6387Å. It is clear in Fig. 8 that a relatively small fraction of retained austenite at a temperature of 250°C is a result of too poor enrichment in carbon of austenite, up to the concentration of about 0.82%. The calculated martensite start temperature of austenitic phase is considerably higher than room temperature (Table 2), what is consistent with the bainitic-martensitic microstructure observed in Fig. 3a.

With increasing the isothermal holding temperature the diffusion rate of C increases and the enrichment of austenite in C is easier. It results in the increase of the lattice constant and carbon content of austenite and the corresponding raise of austenite volume fraction and lowering the $M_{s\gamma}$ temperature (Table 2). The highest carbon content in austenite equal 1.84% occurs for the temperature range from 350 to 400°C, for which the fraction of retained austenite is equal above 20% (Fig. 7). Under these conditions the martensite start temperature of austenite is very low having the stabilizing effect for the high residual fraction of γ phase.

a) T$_B$=250°C

b) T$_B$=300°C

c) T$_B$=350°C

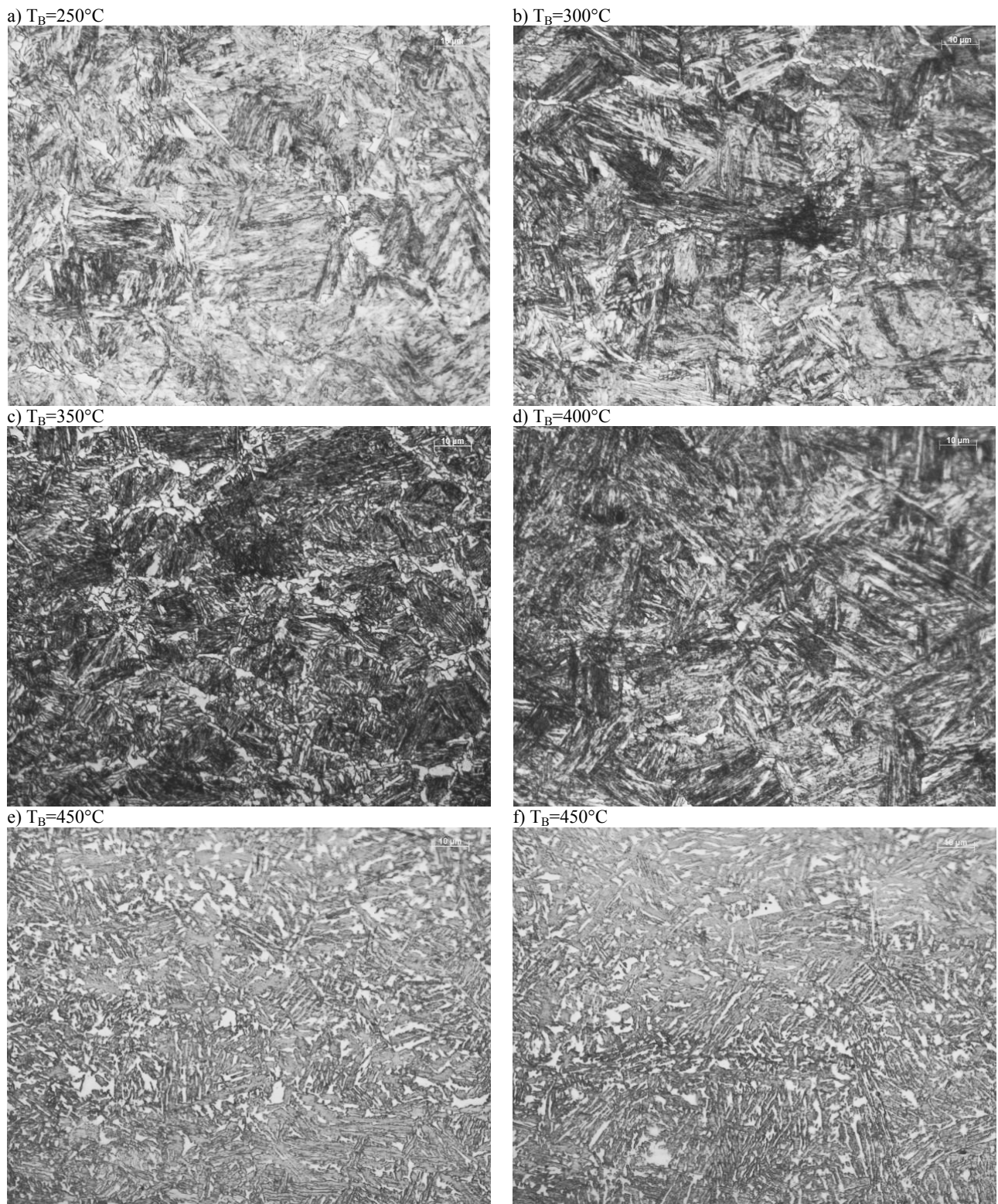d) T$_B$=400°C

e) T$_B$=450°C

f) T$_B$=450°C

Fig. 3. Bainitic microstructures containing a various fraction of retained austenite and martensite (only a, b) for the specimens isothermally held for 600 s at the temperature of 250°C (a), 300°C (b), 350°C (c), 400°C (d), 450°C (e, f)
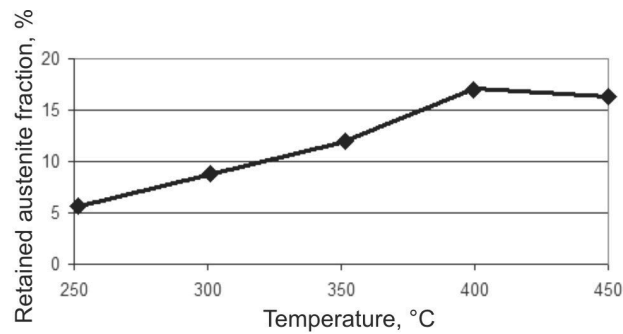
Fig. 4. Influence of the isothermal holding temperature on a fraction of retained austenite determined by image analysis

Table 2.
Fraction of retained austenite, lattice parameter, carbon content in the γ phase and $M_{s\gamma}$ temperature of the austenitic phase for the steel isothermally held in the bainitic transformation temperature range from 250 to 450°C

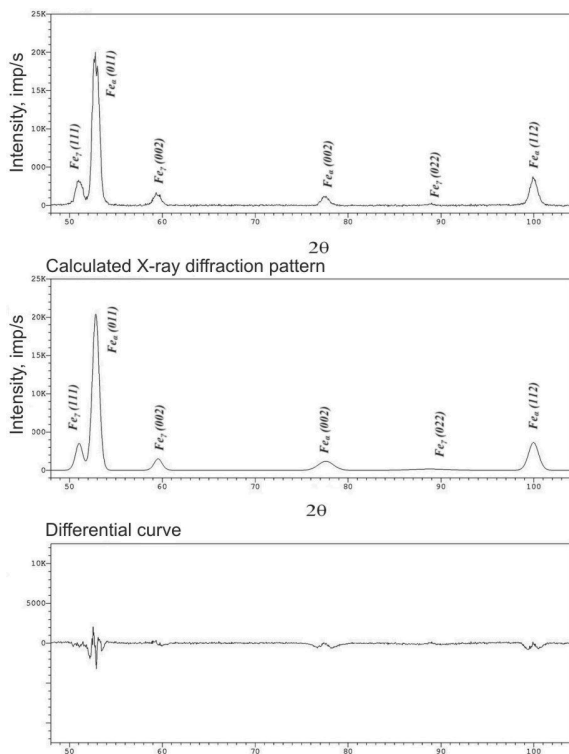| Isothermal holding temperature, °C	Austenite fraction, f_γ	Lattice parameter of austenite, a_γ, Å	Carbon content in the austenite, C_γ, wt.%	$f_\gamma \times C_\gamma$, wt.%	$M_{s\gamma}$, °C
250	0.111 ± 0.015	3.6051 ± 0.0047	0.82	0.0910	215
300	0.151 ± 0.015	3.6197 ± 0.0034	1.26	0.1903	29
350	0.209 ± 0.016	3.6386 ± 0.0026	1.84	0.3845	- 217
400	0.206 ± 0.015	3.6387 ± 0.0025	1.84	0.3790	- 217
450	0.219 ± 0.009	3.6293 ± 0.0007	1.55	0.3395	- 94

Fig. 5. The experimental (the upper pattern), calculated (in the middle) and difference (the lower curve) patterns of the steel after isothermal holding at the temperature of 350°C
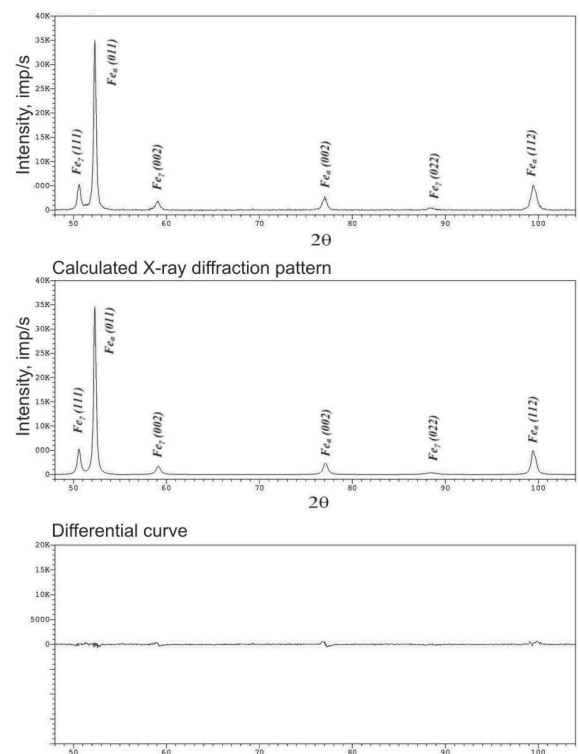
Fig. 6. The experimental (the upper pattern), calculated (in the middle) and difference (the lower curve) patterns of the steel after isothermal holding at the temperature of 450°C
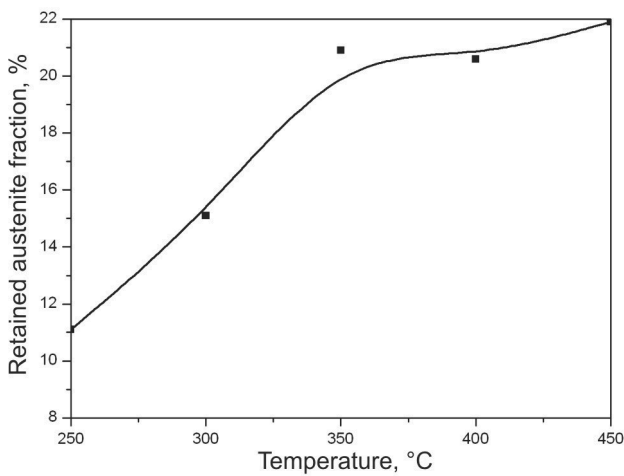
Fig. 7. Influence of the isothermal holding temperature on a fraction of retained austenite determined by the X-ray diffraction

Fig. 8. Influence of the isothermal holding temperature on the lattice parameter and carbon content in retained austenite

It is interesting that with further increasing the isothermal bainitic transformation temperature to 450°C the volume fraction of retained austenite slightly rises (Fig. 7) whereas the lattice constant and carbon content of austenite decrease (Fig. 8). In this regard, the most reliable parameter describing the stability of austenitic phase is a product of retained austenite fraction f_γ and carbon concentration C_γ in this phase (Table 2). Its change as a function of isothermal holding temperature is shown in Fig. 9. As the retained austenite content increases (Fig. 7), the C content in austenite above 400°C decreases significantly (Fig. 8), the corresponding product $f_\gamma \times C_\gamma$ decreases only a little (Fig. 9). The importance of $f_\gamma \times C_\gamma$ parameter is confirmed by the $M_{s\gamma}$ temperature, which is still much lower than room temperature despite lowering the carbon concentration in austenite to 1.55% (Fig. 8). The precipitation process of carbides destabilizing austenite enhances after the increase of isothermal holding temperature to 500°C but these results will be reported elsewhere.
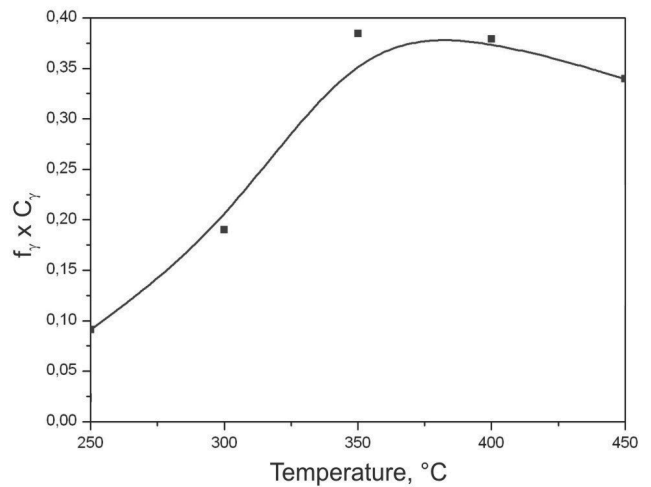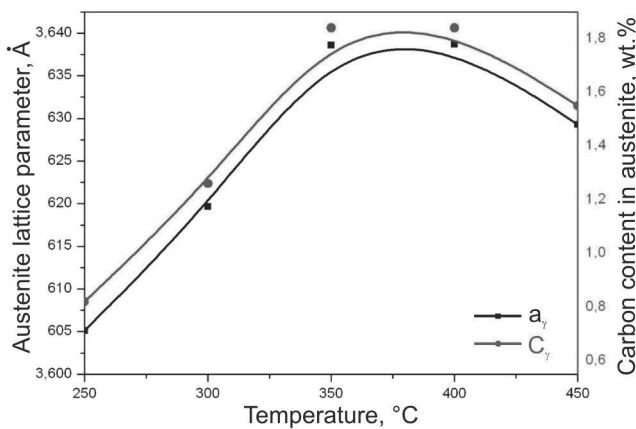

Fig. 9. Influence of the isothermal holding temperature on the mechanical stability of retained austenite defined by the product of retained austenite fraction and carbon content in austenite

The fraction of retained austenite and its carbon content are higher compared to the C-Mn-Si-Al steel containing 0.24% C investigated earlier [18,20]. The change of retained austenite content and C content in austenite as a function of temperature are similar except the peak values of γ phase fraction. The presence of a desired volume fraction of retained austenite in steels containing a typical C content of about 0.2 wt.% requires isothermal holding of steel at a temperature at least 400°C [20], whereas the maximum values of austenite fraction for the investigated medium-carbon steel occur for a quite wide temperature range from 350 to 450°C.

4. Conclusions

The isothermal holding temperature has a significant effect on a fraction of retained austenite and parameters characterizing its thermodynamic stability for the new-developed medium-carbon TRIP steel containing Nb and Ti microadditions. The image analysis is not a useful method for evaluating the fraction of austenitic phase because it does not allow to detect fine granules and interlath regions of this phase distributed in a bainitic matrix. X-ray phase analysis allows better determination of volume fraction of γ phase together with its C content, what has a fundamental meaning for the stability of austenite. The maximum fraction of austenitic phase (between 20 and 22%) mainly of interlath morphology occurs in a wide range of isothermal holding temperature from 350 to 450°C. It is related to the high carbon concentration in retained austenite achieving 1.84 wt.% for a temperature range from 350 to 400°C. Above 400°C the carbon content in austenite reduces probably due to initiation of carbide precipitation but at 450°C is still high enough to lower the martensite start temperature much below room temperature, stabilizing about 22% of interlath and blocky-type retained austenite in a bainitic matrix.

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