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Morphological features of retained austenite in thermo-mechanically processed C-Mn-Si-Al-Nb-Ti multiphase steel

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<u>ABSTRACT</u>

Purpose: The aim of the paper is to determine the influence of isothermal bainitic transformation temperature on morphological features and a fraction of retained austenite in a new-developed thermo-mechanically processed C-Mn-Si-Al-Nb-Ti multiphase steel.

Design/methodology/approach: The thermo-mechanical processing was realized in a multi-stage compression test by the use of the Gleeble thermomechanical simulator. The steel was isothermally held for 600 s in a bainitic transformation temperature range of 250 to 500°C. A fraction and stereological parameters of retained austenite were determined by a computer image analyser using an optical microscope. The details of the retained austenite morphology were revealed in a scanning electron microscope and using EBSD technique.

Findings: The maximum fraction of retained austenite (above 14%) was obtained for the temperatures of isothermal bainitic transformation from 400 to 450°C. Below 350°C, the largest grains of retained austenite located in a ferritic matrix transform to martensite and its fraction estimated by the use of computer image analysis is too high compared to X-ray investigations. Blocky, irregular grains located in a ferritic matrix are a main structural constituent of retained austenite in a temperature range up to 350°C. Increasing the isothermal holding temperature to a range of 400-450°C results in increasing a fraction of fine blocky and layer regions of the γ phase.

Research limitations/implications: To describe in detail morphological features of retained austenite in fine-grained multiphase structures, a combination of different methods characterized by various resolution is necessary.

Practical implications: The revealed morphological features of retained austenite are of great importance for mechanical stability of this phase during cold straining, affecting mechanical properties of advanced TRIP-assisted steels.

Originality/value: Combined colour etching, scanning electron microscopy and EBSD (Electron Backscattered Diffraction) methods were applied to characterize retained austenite in a modern group of thermomechanically processed TRIP steels with Nb and Ti microadditions.

Keywords: Metallic alloys; TRIP steel; Retained austenite; Multiphase structure; Thermo-mechanical processing

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1. Introduction

Development of multiphase steels is one of the biggest achievements of contemporary metallurgy in the field of advanced materials for motorization. They are C-Mn-Si, C-Mn-Si-Al, C-Mn-Al-type low-alloyed steels and recently also C-Mn-Al-(Mo)-(Nb)-(Ti), composed of soft ferritic matrix containing bainitic-austenitic islands. They are produced throughout intercritical annealing of cold rolled sheets in a temperature range between A_{c1} and A_{c3} and successive isothermal holding in the range of bainitic transformation [1-3] or using thermomechanical processing [4-10]. Retained austenite is the most important structural constituent of this group of steels and its fraction is equal from 10 to 20%. Strain-induced martensitic transformation of the phase influences an enhancement of mechanical properties of a finished part, but first of all, it leads to the increase of its plasticity [5, 11]. This is the reason why these steels are called TRIP-type steels (TRansformation Induced Plasticity). Obtaining a desired portion of retained austenite is achieved through selection of adequate chemical composition and conditions of heat treatment or thermomechanical processing, where temperature and time of isothermal bainitic transformation are most important [6-13]. These steels most often consist of 0.15 to 0.25%C. Manganese which concentration is usually equal around 1.5% has a significant meaning for stabilization of γ phase. The lack of precipitation of carbides in the conditions of bainitic transformation is a result of increased concentration of Si or Al up to 2%. Both elements have an impeding effect on cementite nucleation. They are used separately [4, 5, 8, 12] or together [1, 3, 6, 11], at times also with increased up to 0.1% P [2]. In order to enhance mechanical properties of TRIP steels, Mo in amount up to 0.2% [14, 15] and microadditions of Nb, Ti and V [7-11, 14] have been recently introduced.

Determination of y phase participation is most often achieved using the methods of microscopic image analysis, X-ray or neutron diffraction and magnetic method, whilst the concentration of carbon in austenite is analyzed with X-ray methods [13-18]. Recently, the EBSD technique performed in scanning electron microscope has gathered essential significance for determination of morphology characteristics of retained austenite [19, 20]. So far the details of microstructural structure of multiphase steels produced with conventional production technology, i.e. heat treated after cold rolling, have been characterized in a sufficient way. For instance, the kinetics of austenite decomposition during cooling of 0.2C-1.5Mn-1.5Si steel from the temperature above A_{c1} to the temperature of isothermal holding in a range from 250 to 550°C was investigated by Pietrzyk et al. [21], who found that depending on the temperature of transformation, the decomposition of austenite occurs in one, two or three stages. Austenite decomposes into ferrite and carbides in the single-stage course of the curve noted above 500°C. Two-stage character of the course was obtained at 250 and 300°C, where the transformation occurred very slowly and also at 450°C, where austenite decomposes into a mixture of ferrite and carbides, directly after formation of bainitic ferrite. Optimal decomposition of supercooled austenite occurs in a temperature range from 350 to 420°C, for which three distinct stages of transformation can be noted. Carbide-free and characterized with high density of dislocations bainitic ferrite is formed during the first stage. The transformation is stopped in the second stage for the time dependent on the temperature of isothermal holding; carbides precipitate in the third stage, what decreases plasticity of steel to a significant degree [21].

Current state of the problem regarding hot-rolled sheets is much poorer and requires explanation of many phenomena that have a different character as distinct from sheets produced with conventional method - mainly in respect of diversified initial microstructure. In case of sheets austenitized between A1 and A3 temperatures it's a microstructure of recrystallized ferrite and austenite formed from pearlite and in case of hot-rolled sheets microstructure of austenite with various grade of defects dependent on the conditions of hot plastic deformation. Different structural state has a principal impact on fraction, morphology and stability of retained austenite during cooling to room temperature and also during successive forming. Moreover, among limited number of investigations focused on production of hot-rolled sheets, great majority regards to steels with conventional concentration of silicon equal approximately 1.5% [5, 10, 22]. Such concentration of Si causes some technological difficulties connected with obtaining adequate surface of sheets during hotrolling [23] and their protection against corrosion through galvanizing [2, 24]. Improvement of galvanizing susceptibility and obtaining desired portion of retained austenite is achieved thanks to partial or entire substitution of Si with Al [1, 11, 24, 25].

It was found [3-7, 10-17] that retained austenite is present in the form of blocky grains or thin layers located between lamellas of bainitic ferrite. Morphological details of its microstructure are dependent on the location in a matrix. The combinations of retained austenite distribution are as follows [10, 14]:

- block type in polygonal ferrite matrix,
- block type or a film surrounded with acicular ferrite,
- block type or a film surrounded with packets of bainitic ferrite laths,
- films surrounded with laths of martensite,
- martensitic-austenitic islands.

Morphological details of retained austenite in 0.23C-1.54 Mn-1.36 Si type steel, produced with thermomechanical processing method have been investigated by Li et al. [26], among the others. They found that after plastic deformation below the temperature of austenite recrystallization, the grains of γ phase undergo fragmentation caused by formed deformation bands, which portion increases along with the increase of a degree of deformation, leading to increased ferrite grain refinement. Obtained microstructure of fine-grained ferrite has also a positive influence on the size and distribution of bainite and increased fraction of fine grains of retained austenite. The effect of hotworking conditions and successive cooling of steel on structural parameters of products of supercooled austenite transformation was also the subject of research performed by Koh et al. [27]. On the basis of 0.2C-1.5 Mn-2Si steel examination they found that insufficient control of finishing rolling conditions can lead to the formation of coarse-grained bainite, what is strictly dependent on microstructure and participation of formed ferrite. In case of excessively fast cooling of steel in a range of $\gamma \rightarrow \alpha$ transformation, ferrite is formed only along grain boundaries of austenite, what leads to transformation of the remaining part of poorly carbon enriched y phase into coarse-grained bainite. In case of slow cooling, ferrite is formed also in deformation bands, favouring the formation of larger fraction of α phase and therefore causing refinement and uniform distribution of bainite. Novy et al. [28] state that deformation of austenite in the final stage of hot-working which is too low can also be the cause of presence of coarse-grained bainite. In opposite to researches mentioned above, Ryu et al. [4], basing on the investigation of 0.21 C-1.49 Mn-1.95 Si steel, found that participation of ferrite is not a critical factor deciding about the amount of retained austenite. They found that an increase of the cooling rate after deformation at the temperature of 920°C - from 10 to 25°Cs⁻¹ causes a considerable decrease of ferrite fraction, nevertheless at the increase of participation of retained austenite. The results indicate that the factor which is decisive for the amount of γ phase is not only the ferrite fraction but also bainite morphology.

The influence of bainite on morphology and stability of retained austenite in TRIP type steels, cooled directly from the temperature of hot plastic deformation finish were investigated in detail by Timokhina et al. [5, 14, 15] and by Li and Wu [26]. They revealed that multiphase steels obtain their optimal mechanical properties for bainite with granular morphology, i.e. consisting of irregular grains of ferrite and particles of the second phase, which most often are austenitic or austeniticmartensitic islands [2, 29]. Fine islands of austenite located between laths or grains of bainitic ferrite have optimal conditions for carbon enrichment during holding of steel in the bainitic range. It arises from short diffusion length of carbon and small dimensions of γ phase regions, what favours stabilization of austenite. Equally significant is the vicinity of harder laths of bainitic ferrite which are a limitation against transfer of deformation to austenite, that initially locates itself in ferrite. In this regard, grains located in α phase matrix undergo martensitic transformation during cold forming in the first place and the deformation of austenite located in granular bainite proceeds in the further stage of deformation, ensuring gradual course of martensitic transformation [5, 4, 15, 17, 20, 26].

2. Experimental procedure

The new-developed low-alloyed C-Mn-Si-Al-type steel with Nb and Ti microadditions was investigated. The steel contains: 0.24% C, 1.55% Mn, 0.87% Si, 0.4% Al, 0.034% Nb and 0.023% Ti. Chemical composition of elaborated steel was optimized in respect of obtaining an optimal fraction of retained austenite in the conditions of hot-working and controlled cooling. In comparison to TRIP-type steels used most often, the developed steel is characterized by decreased to 0.87% concentration of silicon, which decrement was compensated by addition of Al. Moreover, the steel is characterized by high metallurgical purity connected to low concentration of phosphorus (P=0.01%) and sulphur (S=0.004). The melt together with modification of nonmetallic inclusions by rare-earth elements was done in the Balzers VSG-50 vacuum induction furnace in The Institute for Ferrous Metallurgy in Gliwice. 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 prepared.

The cubicoid specimens were deformed under conditions of plane state of strain using DSI Gleeble 3800 thermomechanical simulator in The Institute for Ferrous Metallurgy in Gliwice. After four-cycle compression, the specimens were cooled with a route shown in Fig. 1. Especially important was isothermal holding of the specimens in a bainitic temperature range from 250 to 500°C for 600s. The samples were inserted in a vacuum chamber, where they were resistance-heated. Tantalum foils were used to prevent sticking and graphite foils as a lubricant.

After the thermo-mechanical treatment was performed, samples for metallographic observations were prepared. 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 in Leica MEF 4A light microscope, equipped with Leica Qwin image analyzer.



Fig. 1. Parameters of the thermo-mechanical processing carried out by the use of the Gleeble simulator

Stereological parameters of retained austenite were determined with the use of digital image analysis in the Image-Pro Plus 4.5 application. Analyses were conducted basing on detailed measurements of 5 microstructure regions (for a specific variant), at the magnification of 1000x. The following stereological parameters were determined:

- grain area determined on the basis of evaluation of square pixels present in geometric figure which is an outline of a specific grain;
- grain height (d) a parameter evaluated on the basis of side length of rectangle described on a grain of retained austenite in respect of vertical axis;
- grain width (1) a parameter evaluated on the basis of side length of rectangle described on a grain of retained austenite in respect of horizontal axis;
- grain length (a) a parameter evaluated by connecting two most distant points with a line passing in the centre of gravity of a specific grain;
- aspect ratio represents grain shape. It is a proportion of grain width to its height ($\lambda = l/d$).

Morphological details of microstructural constituents of steel were observed implementing SUPRA 25, Zeiss scanning electron microscope using an observation technique with the use of backscattered electrons. Observations were performed on nital etched samples at the accelerating voltage of 20kV and magnification of 4000x. In order to evaluate the fraction of retained austenite, EBSD (Electron Backscatter Diffraction) method was implemented. Research done with EBSD was performed in the Inspect F scanning electron microscope equipped with Shottky's field emission. Scanning was conducted applying 0.3 μ m steps. After classical grinding and polishing, specimens were polished with Al₂O₃ with granularity of 0.1 μ m. The final stage of sample preparation was their ion polishing in GATAN 682 PECS system for about 2 hours, at the voltage of U=2keV.

3. Results and discussion

In the result of conducted thermomechanical processing finegrained ferritic-bainitic microstructures were obtained with fraction of ferrite equal from 60 to 70% and amount of retained austenite dependent on isothermal holding in the bainitic range (Fig. 2). Average size of ferrite matrix grains is equal approximately 6µm. Uniformly distributed bainitic-austenitic islands with diversified size are located in the ferritic matrix. Considerable part of austenite grains located in ferrite and the part of bainitic regions are characterized with elongation in the direction of plastic flow, what indicates that they underwent a transformation of austenite deformed at the temperature lower than recrystallization finish temperature of the phase.

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), which are just delicately etched to distinguish better retained austenite. Austenite in bainitic islands has practically undergone a total transformation and remains only in the boundary regions of the islands. The grains of γ phase have characteristic polygonal shape and size from 1 to 7µm. Lamellar topography typical for martensite can be noted in many large grains with shape characteristic for retained austenite. It's also proven by different colour of these grains when comparing to laths of bainite located in larger islands (Fig. 2a).

Presence of martensite is a result of martensitic transformation occurring during the last stage of cooling of samples to room temperature. The transformation indicates too low carbon enrichment of austenite due to low isothermal holding temperature. In the previously performed X-ray analyses [13] it was found that the concentration of carbon in austenite for the temperature of 250°C is equal only 0.42% wt. The M_s temperature of γ phase determined on this basis is equal 320°C, what explains the transformation of the majority of austenite grains.

Increase of isothermal holding temperature to 300°C doesn't change the bainite morphology but martensite fraction definitely decreases (Fig. 2b). During the last cooling stage, the martensitic transformation proceeds only in grains of the largest size, whereas fine grains are mechanically stable. Still, austenite is not a meaningful constituent of bainitic islands.

Further temperature increase to 350° C results in increase of retained austenite fraction and a portion of grains transformed into martensite is small (Fig. 2c). It derives from decreasing M_s temperature of austenite much below room temperature, which is an effect of diffusion enrichment of γ phase with carbon up to the concentration of about 1.5% [13]. Apart from grains located in ferritic matrix, the increase of the grains located in boundary regions of bainitic islands is characteristic.

Uniform distribution of retained austenite is confirmed on binary maps presented as an example in Fig. 2d. Very characteristic irregular block type grains of austenite with sharp-ended edges are mainly located in ferritic matrix. A certain part consists of smaller grains with sharp edges, which are limiting regions of bainitic islands. The shape of retained austenite grains results from the conditions of ferrite growth during $\gamma \rightarrow \alpha$ transformation and successively of bainite during isothermal holding of steel at the temperature of 350°C. Relatively small fraction consists of thin layers of y phase located in bainitic islands. The participation of retained austenite as well as its stereological parameters, depending on the temperature of isothermal holding, are set together in Table 1. Apart from determination of retained austenite fraction assigned on the basis of binary maps obtained with the help of image analyzer, calculations of austenite fractions which are a part of bainitic islands and are present in the form of irregular grains in ferritic matrix were also performed. Determination of boundary area of austenite which delimitate blocktype grains present in ferritic matrix from fine grains or regions of austenite, which are a component of bainitic islands had a substantial meaning for the correctness of the analysis. On the basis of detailed microscopic observations it was accepted that the limiting area of grains was equal from 2 to $3\mu m^2$, depending on the temperature of isothermal bainitic transformation (Table 1).

Apart from that, Table 1 includes mean area of retained austenite with the division for dual character of grains, the factor characterizing grains elongation and their mean length, width and height. Exemplary distribution of specific stereological parameters of retained austenite for isothermal holding temperature of 350°C is presented in Fig. 3. The largest group consists of fine γ phase grains (Fig. 3a). Together with the increase of grain size, their number gradually decreases. It concerns both, grains located in ferritic matrix (Fig. 3c) as well as grains which are the component of bainitic islands (Fig. 3b). However, it should be taken into consideration, that although fine grains are in the majority, their total fraction is lower than it is for grains with larger dimensions. It derives in a simple way from the size of austenite grains; it's confirmed by data provided in Table 1 - surface fraction of grains located in ferritic matrix is equal 8.5% and the portion of fine grains with size smaller than $3 \mu m^2$, present mainly in bainitic islands, is equal 3.4%.

A considerable increase of y phase fraction occurs after increasing the temperature of isothermal holding in the bainitic range to 400°C (Table 1). Part of grains of retained austenite forms a discontinuous halo around α phase grains and the remaining part is present on the phase boundaries of ferrite and bainite (Fig. 2e). Characteristic increase of retained austenite participation in the form of fine granules located in bainitic islands can be noted (Fig. 2f). This phenomenon intensifies after increasing the temperature to 450°C, for which the fraction of retained austenite is equal over 14% (Table 1). It's also confirmed in histograms presented in Fig. 4. The distribution of parameters that are characteristic for retained austenite is analogical as for the temperature of 350°C (Fig. 3), with the exception of substantial increase of number of y phase grains located in bainitic islands. On the basis of X-ray analyses [13] it was found that in spite of large portion of thermally stable retained austenite, carbon concentration in γ phase decreases from 1.5% (for the temperature of 350°C) to about 1.28%. It's a result of increased carbon diffusion rate at 450°C when comparing to the temperature of 350°C.

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Fig. 2. Multiphase structures with various fractions of retained austenite for the thermo-mechanically processed specimens, isothermally held in a bainitic transformation temperature of 250° C (a), 300° C (b), 350° C (c), 400° C (e), 450° C (g), 500° C (h) and selected binary maps showing regions corresponding to retained austenite for the specimens held at 350° C (d) and 400° C (f)

Table 1.

The fraction, mean area, aspect ratio, mean length, mean width and mean height of retained austenite of the steel thermo-mechanically processed according to variants of I-VI

	Retained austenite fraction [%]	Mean area of γ _r grain [μm ²]	Aspect ratio $\lambda = \frac{1}{d}$	Mean length of γ _r grain a [μm]	Mean width of γ _r grain l [μm]	Mean heigth of γ _r grain d [μm]	Variant number
Retained austenite grains	9.88	2.70	1.64	2.78	2.67	1.77	Variant I 250°C
	$^{+}_{-}$ 0.21	$^{+}_{-}$ 0.40	+ 0.03	$^{+}_{-}$ 0.29	$^{+}_{-}$ 0.29	$^{+}_{-}$ 0.18	
Grains of γ_r in bainitic islands	2.16	0.85	1.60	1.68	1.58	1.13	
	$^{+}_{-}$ 0.27	$^{+}_{-}$ 0.12	$^{+}_{-}$ 0.05	$^{+}_{-}$ 0.17	$^{+}_{-}$ 0.16	$^{+}_{-}$ 0.12	Size of the
Grains of γ_r in ferritic matrix	7.72	6.55	1.73	5.06	4.95	3.12	frontier grain area: 2µm ²
	$^{+}_{-}$ 0.38	$^{+}_{-}0.88$	$^{+}_{-}$ 0.06	$^{+}_{-}$ 0.39	$^{+}_{-}$ 0.46	$^{+}_{-}$ 0.18	
Retained austenite grains	10.04	2.44	1.36	2.34	2.14	1.67	Variant II 300°C Size of the frontier grain area: 2µm ²
	$^{+}_{-}$ 0.53	$^{+}_{-}$ 0.50	$^{+}_{-}0.07$	$^{+}_{-}$ 0.22	$^{+}_{-}$ 0.22	$^{+}_{-}$ 0.17	
Grains of γ_r in bainitic islands	3.01	0.91	1.30	1.49	1.34	1.13	
	$^{+}_{-}$ 0.46	$^{+}_{-}0.06$	$^{+}_{-}0.06$	$^{+}_{-}$ 0.04	$^{+}_{-}$ 0.05	$^{+}_{-}0.06$	
Grains of γ_r in ferritic matrix	7.03	6.24	1.52	4.46	4.16	3.03	
	$^{+}_{-}$ 0.94	$^{+}_{-}$ 0.72	$^{+}_{-}$ 0.10	$^{+}_{-}$ 0.33	$^{+}_{-}$ 0.37	$^{+}_{-}$ 0.16	
Retained austenite grains	11.90	2.80	1.53	2.61	2.45	1.80	Variant III 350°C
	$^{+}_{-}$ 1.41	$^{+}_{-}$ 0.51	$^{+}_{-}$ 0.12	$^{+}_{-}$ 0.16	$^{+}_{-}$ 0.21	⁺ _0.13	
Grains of γ_r in bainitic islands	3.44	0.96	1.52	1.74	1.60	1.22	
	$^{+}_{-}$ 0.84	$^{+}_{-}$ 0.12	$^{+}_{-}$ 0.15	$^{+}_{-}$ 0.14	$^{+}_{-}$ 0.15	$^{+}_{-}0.08$	Size of the frontier grain area: 3µm ²
Grains of γ_r in ferritic matrix	8.46	8.68	1.56	5.38	5.15	3.64	
	⁺ _1.03	$^{+}_{-}$ 0.52	$^{+}_{-}0.05$	$^{+}_{-}$ 0.21	⁺ 026	$^{+}_{-}$ 0.17	
Retained austenite grains	14.08	1.32	1.54	2.01	1.86	1.43	
	$^{+}_{-}$ 0.57	$^{+}_{-}$ 0.10	$^{+}_{-}$ 0.24	$^{+}_{-}$ 0.18	$^{+}_{-}$ 0.22	$^{+}_{-}0.07$	Variant IV 400°C Size of the frontier grain area: 3µm ²
Grains of γ_r in bainitic islands	3.85	0.71	1.55	1.68	1.56	1.18	
	$^{+}_{-}$ 0.24	$^{+}_{-}$ 0.02	$^{+}_{-}$ 0.25	$^{+}_{-}$ 0.20	$^{+}_{-}$ 0.24	$^{+}_{-}$ 0.07	
Grains of γ_r in ferritic matrix	10.23	7.22	1.38	5.18	4.75	3.86	
	$^{+}_{-}$ 0.58	$^{+}_{-}$ 0.27	⁺ _0.13	$^{+}_{-}$ 0.42	$^{+}_{-}$ 0.44	$^{+}_{-}$ 0.20	
Retained austenite grains	14.07	1.32	1.69	2.33	2.27	1.49	
	$^{+}_{-}$ 0.74	⁺ _0.13	$^{+}_{-}$ 0.12	$^{+}_{-}$ 0.11	$^{+}_{-}$ 0.12	$^{+}_{-}0.08$	Variant V 450°C
Grains of γ_r in bainitic islands	5.85	0.71	1.62	1.63	1.55	1.09	
	$^{+}_{-}0.25$	$^{+}_{-}$ 0.02	$^{+}_{-}$ 0.14	$^{+}_{-}$ 0.06	$^{+}_{-}0.08$	$^{+}_{-}$ 0.05	Size of the frontier grain area: 3µm ²
Grains of γ_r in ferritic matrix	8.22	7.11	1.78	5.78	5.74	3.49	
	$^{+}_{-}0.16$	$^{+}_{-}$ 0.54	$^{+}_{-}$ 0.06	$^{+}_{-}$ 0.25	$^{+}_{-}$ 0.24	$^{+}_{-}$ 0.21	
Retained austenite grains	6.06	1.54	1.31	2.04	1.79	1.56	Variant VI 500°C
	$^{+}_{-}0.93$	$^{+}_{-}0.46$	$^{+}_{-}$ 0.19	$^{+}_{-}$ 0.18	$^{+}_{-}$ 0.15	$^{+}_{-}$ 0.11	
Grains of γ_r in bainitic islands	1.92	0.63	1.29	1.56	1.35	1.22	
	$^{+}_{-}0.15$	$^{+}_{-}$ 0.14	$^{+}_{-}$ 0.23	$^{+}_{-}$ 0.31	$^{+}_{-}$ 0.26	$^{+}_{-}$ 0.18	Size of the frontier grain area: 2µm ²
Grains of γ_r in ferritic matrix	4.12	5.89	1.30	4.22	3.81	3.29	
	$^{+}_{-}$ 0.20	$^{+}_{-}$ 0.64	$^{+}_{-}$ 0.18	$^{+}_{-}0.27$	$^{+}_{-}$ 0.24	$^{+}_{-}$ 0.41	

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Fig. 3. Statistical parameters characterizing retained austenite in a structure of the steel thermo-mechanically processed according to the variant III; a - distribution of the surface area of retained austenite grains, b - distribution of the surface area of retained austenite grains located in bainitic islands, c - distribution of the surface area of retained austenite grains localized in a ferritic matrix, d - aspect ratio of retained austenite grains located in a ferritic matrix.



Fig. 4. Statistical parameters characterizing retained austenite in a structure of the steel thermo-mechanically processed according to the variant V; a - distribution of the surface area of retained austenite grains, b - distribution of the surface area of retained austenite grains located in bainitic islands, c - distribution of the surface area of retained austenite grains localized in a ferritic matrix, d - aspect ratio of retained austenite grains located in a ferritic matrix



Fig. 5. Influence of the isothermal holding temperature on a fraction of retained austenite obtained by computer image analysis; fractions of retained austenite determined in [13] by X-ray quantitative analysis also included

The regions of γ phase in bainitic islands are present in the form of elongated thin layers or fine polygonal grains with sizes from 1 to 3 µm (Fig. 2g). An increase of holding temperature to 500°C causes a considerable decrease of γ phase amount connected with initiation of carbides precipitation. In such conditions, only fine grains of austenite located in bainitic islands are stable (Fig. 2h); they are probably characterized with higher concentration of carbon when comparing to the regions on the boundaries of ferrite that have undergone a transformation into upper bainite or fine-lamellar pearlite.

Summary of changes regarding the fraction of retained austenite in the function of isothermal holding temperature in the range of bainitic transformation is shown in Fig. 5. It can be noticed, that the participation of retained austenite increases from approximately 10% for the temperature of isothermal bainitic transformation equal 250°C, to 14% - assuming the maximum in a temperature range from 400 and 450°C. The fraction of retained austenite strongly decreases after this temperature is exceeded. The change of γ phase participation is strictly connected with the changes of carbon concentration in austenite, what was analyzed in detail in [13]. The fraction of γ phase grains located in bainitic islands increases along with the increase of total portion of retained austenite. Although it can be assumed from the data set together in Table 1 and histograms presented in Fig. 3 and 4 that in terms of quantity fine grains with size up to 2µm present in bainite are in a definite majority - the total portion of retained austenite is higher for the grains located in ferritic matrix. Relative surface fraction of grains present in bainitic islands for the holding temperature of 250°C is equal approximately 20% of all retained austenite grains and reaches the maximum close to 45% at the temperature of 450°C (Fig. 5). It arises from much bigger area of γ phase grains located on the boundaries of ferrite grains, which is equal from 5 to $10\mu m^2$ (Table 1).



Fig. 6. Influence of isothermal holding temperature on a mean retained austenite grain area in a ferritic matrix as well as in bainitic islands

What results additionally from data presented in Table 1 is that the grains of retained austenite located in ferrite demonstrate a tendency to elongate in a direction of plastic flow what is confirmed by the value of λ aspect ratio equal from 1.4 to 1.8. The elongation of grains is the result of deformation of steel below the temperature of austenite recrystallization finish. Static recovery occurs during cooling of steel from the temperature of deformation finish to the temperature of accelerated cooling which does not influence the shape of γ phase grains. Fine grains or regions of austenite present between laths of bainitic ferrite have lower elongation factor. Average aspect ratio for this fraction of austenite is equal about 1.4. In general, the temperature of isothermal holding has no influence on the size of austenite grains present in ferritic matrix with average surface area equal from 6 to $8\mu m^2$ (Fig. 6). However, along with its increase, average surface area of regions which are a part of bainitic islands decreases from $1\mu m^2$ to about $0.7\mu m^2$ for the temperature of 500°C.

The line in Fig. 5, which responds to the fraction of retained austenite determined in X-ray analyses, was drawn for comparative purposes [13]. It can be noticed that up to the temperature of 350°C the amount of austenite determined applying the method of image analysis is overestimated. It arises from the fact that binary maps of retained austenite include morphological regions responding to γ phase that have probably undergone martensitic transformation in the last stage of steel cooling. In case of specimens isothermally held at 250 and 300°C the fraction of these regions is considerable (Fig. 2a, b). The quality of metallographic specimens etching strongly influences final results of distinguishing austenite and martensite. The next problem was to identify lamellar regions of retained austenite which are a component of bainitic islands. These problematic regions classified as retained austenite are marked in Fig. 2c, for instance. In turn, the reason of slightly understated fraction

of γ phase for the temperatures of isothermal holding of 400 and 450°C is the lack of possibility to detect finest grains of retained austenite, which fraction increases considerably in analyzed temperature range (Fig. 5). This limitation is not valid for X-ray analysis, which results should be acknowledged as more authoritative [13].

a)

b)



Fig. 7. Morphological features of retained austenite of the steel isothermally held at a temperature of 350° C (a) and 450° C (b)

Doubts concerning the identification of specific structural constituents of multiphase microstructures with high refinement can be minimized when applying microscopic techniques with resolution higher than it's offered by light microscopy. As an example, Fig. 7 presents microstructures of steels obtained in scanning electron microscope. It's particularly important to reveal the morphology of retained austenite which is the component of bainitic islands. Identification of lath-type regions of γ phase, located between lamellas of bainitic ferrite was possible for applied magnification; their univocal detection was impossible when performing observations by means of light microscope.

It's characteristic that at the temperature of 350°C layers of austenite packed in parallel are continuous (Fig. 7a), while at the temperature of 450°C their character is discontinuous (Fig. 7b). Moreover, lath-type regions prevail in the central part of the islands and irregular block-type grains with diversified size and morphology are located in their peripheries (Fig. 7b).

Additional information concerning precise determination of size, shape and distribution of retained austenite is brought by grain distribution maps, obtained in scanning electron microscope with the use of EBSD technique (Fig. 8). Exemplary analysis performed for the steel isothermally held at the temperature

of 450°C confirms that retained austenite is present in a form of fine block-type grains in ferritic matrix as well as lath regions usually located in bainitic islands. Distribution of retained austenite is quite homogeneous. Generated image quality map (Fig. 8c) reflects the presence of specific structural constituents really well.

There is no problem to distinguish retained austenite since its identification is possible on the basis of its crystal lattice, different from the matrix - it roughly responds to black regions in Fig. 8c. Distinguishing bainitic regions and ferrite can be done on the basis of greyness shades of specific grains. The grains with greater shading respond to the regions with inferior quality

of diffraction, which can be identified as bainite [20]. Moreover, bainite can be identified on the basis of its characteristic elongated shape and its usual presence in the vicinity of retained austenite. Additionally, it comes from Fig. 9 that the fraction of fine regions of retained austenite with area smaller than 3 μ m² is equal around 80%. Great part of these grains is the component of bainitic islands.

Larger fraction of fine regions of γ phase in comparison with the one determined with image analysis in light microscope (Fig. 5) arises from the possibility of detecting even finest regions of retained austenite with EBSD method. The size of the next 8% of grains is equal from 3 to 4.5 μ m² and should be assigned mainly to boundary regions of bainitic islands. The biggest grains with size of over 6 μ m² (Fig. 9) correspond to block-type grains of γ phase, located in ferritic matrix (Fig. 8). Additional information regarding morphological characteristics of retained austenite can be obtained on the basis of misorientation angles of the phase, what will be the subject of further investigations. a)



b)



c)



Fig. 8. Structure image of the steel isothermally held at 450°C (variant V) obtained by the EBSD technique: a) distribution map of the grains with a various crystallographic orientation, b) marked regions of retained austenite, c) image quality map



Fig. 9. Distribution of retained austenite grain area in the steel isothermally held at 450° C obtained by EBSD technique

4. Conclusions

Identification of retained austenite in multiphase microstructures with high grade of refinement is a complex problem, requiring application of complementary research techniques for complete description of morphological features of γ phase. In particular, it was found out that:

- the temperature of isothermal bainitic transformation has a decisive influence on the fraction and morphology of retained austenite. Optimal conditions for carbon enrichment of γ phase, at its possibly highest participation are noted in the temperature range between 400 and 450°C. Transformation of large fraction of austenite into martensite occurs below this temperature range and after exceeding it - initiation of pearlitic transformation and precipitation of carbides in upper bainite.
- up to isothermal holding temperature equal 300°C austenite is present mainly in the form of irregular block-type grains with sharp edges distributed on the boundaries of ferrite grains. At the temperature of 350°C retained austenite is present also in the boundary regions of bainitic islands as well as in the form of thin layers, located between laths of bainitic ferrite.
- increased fraction of fine block-type grains and discontinuous layer regions located in bainitic islands occurs in the temperature range between 400 and 450°C. After an increase of isothermal holding to 500°C, only fine regions within bainitic islands keep their stability.
- precise disclosure of retained austenite morphology which is a component of bainitic islands requires implementation of microscopic techniques with higher resolution than it's offered by light microscopy.
- application of EBSD technique in scanning electron microscope allows to detect even finest regions of retained austenite, which amount is bigger than the amount determined with computer image analysis with the use of light microscope. The area of γ phase regions located in bainitic islands does not exceed $3\mu m^2$.

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