

# Development of the heat treatment mode for TRIP steel additionally alloyed with aluminium

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The article is devoted to the development of the heat treatment mode for TRIP steel with a new chemical composition. Aluminum was chosen as an additional alloying element, since by the nature of its effect on the stabilization of overcooled austenite in the temperature range of diffusion transformation, aluminium is similar to silicon, the main alloying element for steels containing metastable austenite in the structure. For the correct development of the heat treatment mode of modified TRIP steel, the critical points were determined by differential scanning calorimetry. After determining the temperature limits of the two-phase area, the cast samples were subjected to heat treatment. For all the studied samples, the exposure temperature in the bainite area was 400 °C, and the annealing temperature in the two-phase area was changed from minimum to maximum. The exposure time for both intervals was 20 min and was the same for all samples. As a result, the dependence of the amount of metastable austenite in the structure of modified TRIP steel on the annealing temperature was determined in the intercritical interval (area between the points  $A_{c1}$  and  $A_{c3}$ ). This dependence is of an extreme nature. The article also provides a comparison of the microstructure of modified steel in the cast state and after heat treatment. The analysis of the microstructure allowed us to conclude that continuous monotonous cooling of aluminium-modified TRIP steel made it impossible to obtain a structure containing metastable austenite. In addition, the modified chemical composition of TRIP steel contributes to sufficient stabilization of overcooled austenite in the temperature range of pearlite transformation. The data obtained in this work made it possible to develop a mode of heat treatment of TRIP steel modified with aluminium.

**Key words:** TRIP steel, chemical composition, heat treatment, critical points, annealing, metastable austenite, ferrite, bainite.

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## Introduction

TRIP steels belong to the class of modern high-strength steels (Advanced High Strength Steels - AHSS) with equally high parameters of tensile strength and elongation. Basically, the microstructure of TRIP steel consists of 50–55 % ferrite, 30–35 % bainite and 7–15 % metastable austenite. The structure of TRIP steel may also contain a certain amount of martensite (2–3 %) [1, 2].

The properties of these steels depend on the chemical composition and the mode of heat treatment, which directly affect the amount of metastable austenite preserved in the structure, its morphology, and the carbon content in it [3]. Heat treatment of these steels consists of two-stage isothermal annealing: at first in the intercritical interval, then in the interval of bainitic transformation.

Isothermal annealing in the two-phase area is necessary for the formation of the maximum possible amount of ferrite and the enrichment of austenite with carbon. If the an-

nealing temperature is close to the temperature  $A_3$ , then the structure will have the maximum amount of austenite with a minimum concentration of carbon in it. The positive thing is that the higher the exposure temperature in the two-phase area is, the faster and more complete the process of dissolution of the carbide phase is. However, a decrease in the annealing temperature will contribute to a greater saturation of austenite with carbon and increase its stability to decay, and, consequently, an increase of the incubation period of bainitic transformation [4, 5]. Besides temperature, the completeness of phase transformations is also affected by the exposure time and the cooling rate in the process of annealing. The longer the isothermal exposure in the two-phase area is the more complete the phase transformations are, i.e., equivalent to an increase in temperature.

The second isothermal exposure is carried out in bainitic transformation temperatures, i.e., about 400 °C and corresponds to the temperature of the lowest stability of overcooled austenite during bainite transformation. Exposure at

higher temperatures affects TRIP steels in a negative way, contributing to the formation of cementite and a decrease in the carbon concentration in overcooled austenite. Besides temperature, exposure time is also an important factor in isothermal exposure: the longer the exposure time is, the greater the degree of completion of the bainite transformation is. As the bainite transformation develops, the amount of high-carbon metastable austenite increases until it reaches a maximum. At the same time, the bainite transformation is accompanied by a decrease in the temperature of the Ms of the overcooled austenite remaining in the structure, and the possibility of the formation of quenching martensite with further cooling gradually decreases. With sufficient enrichment of overcooled austenite with carbon, the temperature of the beginning of the martensitic transformation drops to subzero temperatures. This contributes to the formation of a microstructure containing ferrite, bainite and overcooled austenite, stable at ambient temperature [6-13].

An increase in the stability of overcooled austenite to pearlite transformation, as well as a significant decrease in the intensity of the process of separation of excess phases is a consequence of the introduction of certain alloying elements.

Silicon is the main element in TRIP steels, which makes it possible to preserve overcooled austenite in the structure at a relatively low carbon content. Silicon additives contribute to the enrichment of overcooled austenite with carbon and, thus, affect the stability of overcooled austenite, which leads to the preservation of a certain amount of overcooled austenite at room temperature. Starting with a content of 1%, silicon significantly slows down the release of carbides, including the temperature range of the intermediate transformation with a simultaneous increase in its incubation period. All this contributes to carbon saturation of overcooled austenite and an increase in its resistance to decay when further cooled to room temperature [14-19].

Another element that has a similar effect on the stability of overcooled austenite in the temperature range of diffusion transformation is aluminium [20]. It also suppresses the process of carbides release during intermediate transformation and slightly reduces its incubation period [21-25]. Based on these data, a modified composition of TRIP steel was developed. The modified composition of TRIP steel was additionally alloyed with aluminium as a silicon substitute element.

Therefore, the research devoted to the development of the heat treatment mode of TRIP-steels becomes relevant. A change in the chemical composition can lead to a change in the temperature interval of the intercritical area (area between the points  $A_{c1}$  and  $A_{c3}$ ), and to a shift of the points  $A_{c1}$  and  $A_{c3}$  both in the direction of higher and lower temperatures. At the same time, the time of thermal process-

ing should be limited since prolonged exposure at such high temperatures contributes to the decarburization of steel. It is advisable to limit the exposure time to 20 minutes. The time limitation requires an accurate determination of the annealing temperature in the two-phase area to preserve the maximum amount of metastable austenite in the structure.

Prolonged (several hours) exposure at a temperature close to  $A_{c1}$  is inadvisable due to intensive decarburization, as well as a 20-min exposure at a temperature close to  $A_{c3}$  due to the possible formation of an irrational quantitative ratio between austenite and ferrite.

Since the composition of the experimental steel is new, and the heat treatment mode is limited by several factors, the aim of the work is to develop the technology of heat treatment of cast samples of TRIP steel, additionally alloyed with aluminium. To do this, it is necessary to instrumentally determine the critical points of steel with a modified chemical composition and to set a time limit of isothermal exposure.

### Materials and methods of research

The melting of experimental alloys (Table 1) was carried out in the foundry laboratory of the Nosov Magnitogorsk State Technical University in induction furnaces IST-06 with a main lining and a melting weight of 2 kg.

Materials were used as a charge for obtaining experimental alloys:

- carbon steel, 0.3 % C, GOST (Russian Standard) 380-94;
- ferrosilicon grade FS 65, GOST (Russian Standard) 1415-93;
- ferromanganese grade FMn 90, GOST (Russian Standard) 4755-91;
- aluminium of technical purity A85, GOST (Russian Standard) 11069-2001;
- graphite powder of high purity OSCH 8-4, GOST (Russian Standard) 23463-79.

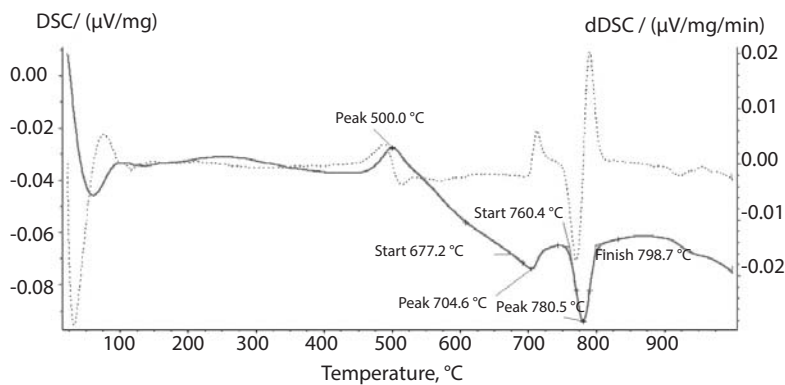
Deoxidation of experimental alloys was carried out with aluminium A85, as well as aluminium, which is part of the charge materials. The total melting time of 2 kg of material was 12-15 min. In the course of melting, steel scrap (St3ps steel) and graphite powder were melted, after which ferrosilicon, ferromanganese and aluminium of technical purity were added. All the components added, complete homogenization takes 2 min. After that, the melt poured into a preheated metal mold. The samples obtained were the size of 35x35x10 mm.

Heat treatment was carried out according to the following mode: heating and exposure in the intercritical interval, then sharp cooling to the temperature of isothermal exposure. Subsequent cooling was carried out in calm air.

Heating to a temperature and exposure in the two-phase area were carried out in an electric furnace SNOL-

Table 1. The modified chemical composition of the samples of TRIP steel

Mass portion, %											
C	Si	Mn	P	S	Cr	Ni	Cu	Al	Ti	V	Mo
0.37	3.36	2.92	0.015	0.012	0.048	0.040	0.073	0.085	0.005	0.004	0.004



**Fig. 1. DSC heating curve of an experimental cast sample of TRIP steel modified with aluminium**

1,4,2,5.1,2/12,5 in an oxidizing atmosphere. The samples were loaded into a preheated furnace, the exposure time was 20 minutes to complete the diffusion processes.

Isothermal exposure at a temperature of 400 °C for 20 minutes was carried out in a salt bath with a melting point of about 320 °C.

Thermal analysis to determine the critical points of the modified alloy was carried out on a NETZSCH STA 449 F3 Jupiter thermal analyzer in an inert argon atmosphere, the weight of the sample was 100 mg, the heating rate was 10 °C/min.

X-ray diffraction analysis was performed on a Shimadzu XRD-7000 X-ray diffractometer. The survey was carried out using an X-ray tube with a chrome anode at voltage of 40 kV and current of 30 mA. Angle  $2\theta = 66\text{--}71^\circ$ , shooting speed 0.1 degree/min. The quantitative assessment was carried out by the ratio of the integral intensities of diffraction maxima from the system of planes (111) austenitic phase ( $2\theta \approx 67.1^\circ$ ) and (110) martensitic ( $2\theta \approx 68.7^\circ$ ) phase. To measure the integral intensity, the standard package "Basic Process" in the Shimadzu XRD6100/7000 Ver 7.01 software was used. The data obtained were the intensity of the maxima ( $I$ ) and the intensity value of each peak, reduced to the intensity value of the maximum peak ( $I/I_{max}$ ), as well as the value of the Bragg angle  $2\theta$  for each maximum.

The amount of residual austenite  $K_\gamma$  was determined by the ratio of the integral intensities of the diffraction maxima from the system of planes of the martensitic  $I_\alpha$  and austenitic  $I_\gamma$  phases, considering the presence of the carbide phase  $K_k$  according to the formula.

$$K_\gamma = \frac{1 - K_K}{1 + \frac{I_M}{I_A} \cdot \frac{\Psi_A}{\Psi_M} \cdot \frac{P_A}{P_M} \cdot \frac{V_m}{V_l} \cdot \frac{S_A^2}{S_M^2} \cdot \frac{\sin^2\Theta_A}{\sin^2\Theta_M}}$$

where  $K_K$  – quantity of carbide phase, reduced to 1;  $I$  – integral intensity of diffraction maximum from the system of planes;  $\Psi$  – angular multiplier of diffraction maximum;  $P$  – multiplier of the multiplicity for the system of planes;  $V$  – volume of the unit cell, angstrom;  $S^2$  – structural fac-

tor: for BCC is equal to 4, for FCC is equal to 16;  $\sin^2\Theta$  – the second power of the half of Bragg angle.

Metallographic studies of TRIP-steel samples were carried out at the Research Institute of "Nanosteels" of the Nosov Magnitogorsk State Technical University. The preparation of the grinds was carried by pressing samples into the resin "Transoptic" on an automatic Simplimet 1000 press on the Buechler sample preparation system. The study of the surface of microgrinds in an unetched and etched form was carried out using an Axio Observer optical microscope at magnifications from 50 to 1000 times using the Thixomet PRO computer image analysis system. Determination of the average size of martensite needles was carried out manually using the "Linear Size" tool in the Thixomet PRO software product.

The study of the morphology features of microstructural components was carried out on a scanning electron microscope JSM 6490 LV with an accelerating voltage of 20 kV, equipped with an energy dispersive microanalysis system INCA Energy 450 x-MAX 50 Premium at magnifications of more than 1000 times.

### The obtained results

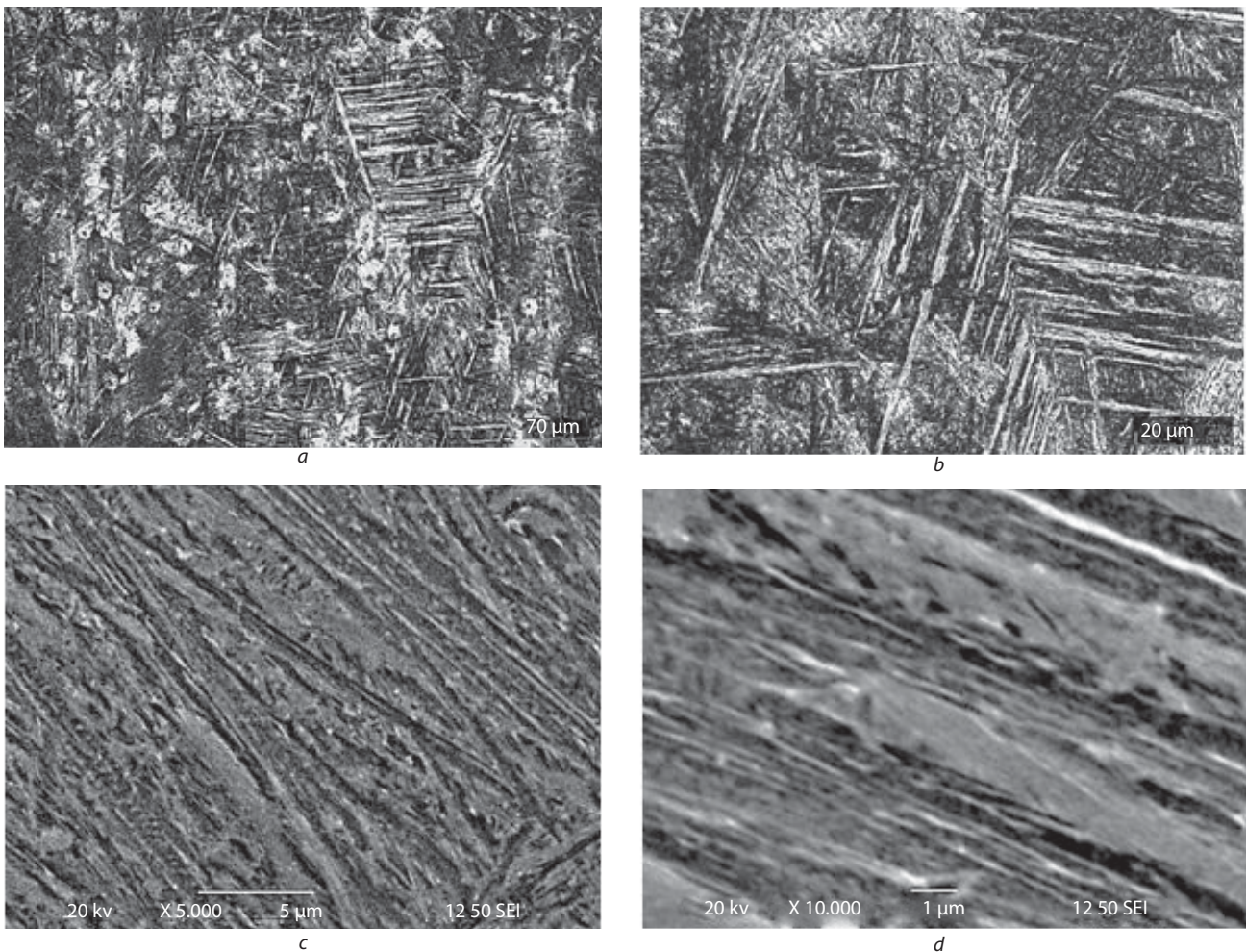
Differential scanning calorimetry (DSC) was performed to determine the critical points of TRIP steel with a modified chemical composition. The results of the DSC analysis of cast samples showed that the critical points  $A_{c1}$  and  $A_{c3}$  for steel of modified chemical composition are 705 °C and 800 °C, respectively (Fig. 1).

Thus, the intercritical interval for the steel modified with aluminium is 705–800 °C.

After determining the temperature limits of the two-phase area, the cast samples were subjected to heat treatment. For all the studied samples, the exposure temperature in the bainite area was 400 °C, and the annealing temperature in the two-phase area was 710, 730, 760, 780, and 800 °C. The exposure time for both intervals was the same for all samples and was 20 minutes. Quantitative X-ray diffraction analysis of the studied samples showed that the amount of residual austenite varies from 3.18 to 6.88%.

The microstructure of a sample of TRIP steel in its as-received (cast) state is shown in Fig. 2. It is a mixture of upper bainite (*a, b*) and lower bainite (*c, d*). The needles are located mainly at angle 120° relatively to each other.

After heat treatment, the structure of all studied samples does not differ qualitatively from each other, the difference lies in the quantitative ratio of structural components (Fig. 3). The microstructure of all heat-treated samples consists of ferrite (~ 5%) (Fig. 3, *a*), lower bainite (60–70%) (Fig. 3, *b*), martensite (Fig. 3, *c*), and residual austenite (Fig. 3, *d*). The average length of the rails is 26 microns, the distance between the rails is 0.8 microns. The data obtained correlate with the conclusions obtained in the study [26].



**Fig. 2.** Microstructure of a sample of TRIP steel modified with aluminium, in as-received (cast) state. Optical microscopy, x 200 (*a*, *b*), SEM x 5,000 (*c*) and x10,000 (*d*)

### Discussion

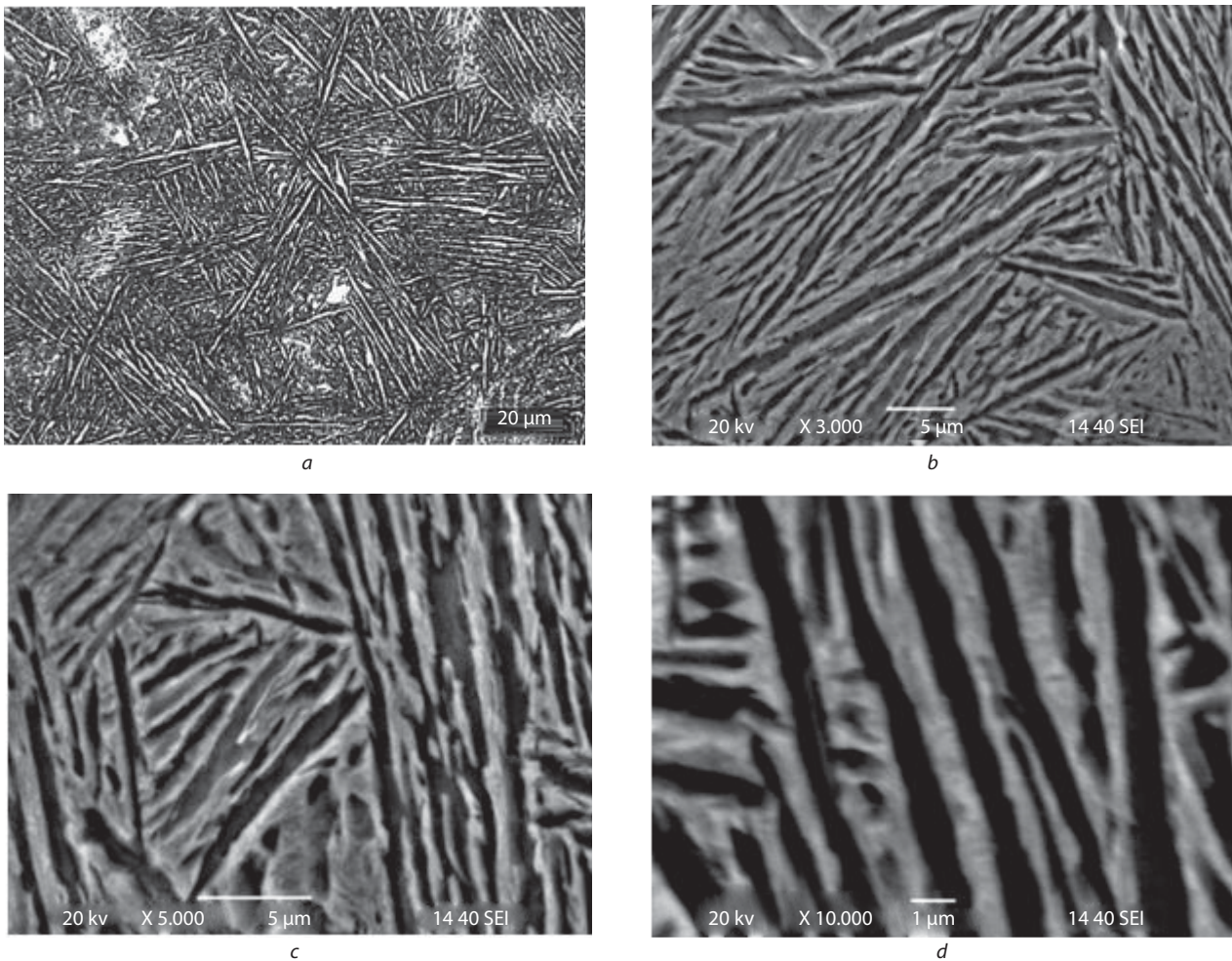
There is no ferrite in the structure of the cast sample, which is a consequence of the monotonous nature of the cooling of the alloy after its crystallization, since there is no isothermal exposure in the two-phase area. It is also typical that there are no diffusion transformation products in the microstructure. This suggests that the alloying complex is selected correctly to stabilize the overcooled austenite in the pearlite area. The presence of upper and lower bainite in the structure indicates that continuous cooling does not contribute to sufficient saturation of overcooled austenite with carbon. The formation of upper bainite with a high content of the carbide phase leads to a decrease in the carbon concentration in the solid solution, a decrease in its stability and complete transformation without preservation metastable austenite in the structure.

Ferrite is present in the microstructure of the heat-treated sample, which indicates the process of saturation of austenite with carbon during isothermal exposure in the two-phase area. There are no diffusion decay products in the structure, which indicates a sufficient cooling rate between isothermal exposures.

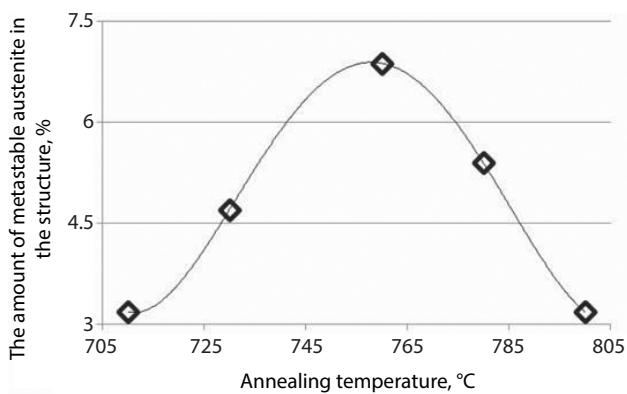
In addition to ferrite, the structure contains lower bainite, martensite and metastable austenite. The absence of the upper bainite and carbide phase in the microstructure is the result of choosing the correct isothermal exposure in the lower temperature area of the bainitic transformation. In this case, the overcooled austenite is saturated with carbon, stabilized, and preserved after cooling to room temperature.

The study of a series of samples with different temperatures of isothermal exposure in the two-phase area revealed the typical character of the influence of this temperature on the amount of metastable austenite preserved in the structure. **Fig. 4** shows the dependence of the amount of metastable austenite in the structure of modified TRIP steel depending on the annealing temperature in the intercritical temperature range. It has an extreme character with a maximum at a temperature of 760 °C.

After annealing at a temperature of 710 °C, 3.18 % of residual austenite remains in the structure. Such a small amount of residual austenite can be explained by the fact that at such a temperature (close to  $A_{c1}$ ), the exposure time was insufficient to complete the diffusion processes (dissolution of carbides



**Fig. 3. Typical microstructure of a sample of TRIP steel modified with aluminium after annealing at temperature 760 °C and subsequent isothermal exposure at temperature 400 °C for 20 min:**  
*a* – optical microscopy, *b-d* – SEM; x500 (*a*), x3,000 (*b*), x5,000 (*c*), x 10,000 (*d*)



**Fig. 4. Dependence of the amount of metastable austenite on the annealing temperature of TRIP steel modified with aluminium in the two-phase area**

and redistribution of carbon). An increase in the annealing temperature contributes to a more complete course of these processes over the same time, which affects the preservation

of almost 7% of the residual austenite in the structure of TRIP steel at room temperature (annealing at 760 °C). An increase in the annealing temperature above 760 °C leads to the fact that after isothermal exposure, the structure contains a large amount of austenite, but at the same time the concentration of carbon in it decreases. Consequently, with further cooling, its decay occurs, and the amount of residual austenite in the structure decreases to almost 3%.

The analysis of the data made it possible to set an annealing temperature of 760 °C in the two-phase area, whereas the end of the heat treatment cycle, about 7% of metastable austenite is preserved in the structure.

### Conclusions


The chemical composition of the modified TRIP steel, additionally alloyed with aluminium, contributes to sufficient stabilization of overcooled austenite in the temperature range of diffusion transformation.

With continuous monotonous cooling of TRIP steel modified with aluminium, it is impossible to obtain a structure containing metastable austenite due to the absence of

ferrite in the structure and the formation of the carbide phase of upper bainite.

For the modified composition of the TRIP steel under study, the critical points were determined by the thermal method. The critical points are 705 °C for  $A_{c1}$  and 800 °C for  $A_{c3}$ .

The maximum amount of 6.88 % metastable austenite is preserved in the structure of modified TRIP steel after annealing at a temperature of 760 °C. An increase and decrease in the annealing temperature of 760 °C leads to a decrease in the amount of metastable austenite to 3.18 %.

There has been developed a mode of heat treatment of modified TRIP steel additionally alloyed with aluminium. The mode includes annealing in the two-phase area at a temperature of 760 °C, annealing in the temperature range of bainite transformation at a temperature of 400 °C, isothermal holding time of 20 min. 

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