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CONDITIONS OF AUSTENITE DIFFUSIONAL TRANSFORMATION IN STEEL OF Cr–3Ni–Mo–V-COMPOSITION WITH HIGH AUSTENITE STABILITY

Viktor V. TSUKANOV, Nadezhda V. LEBEDEVA, Yuliya M. MARKOVA
Central Research Institute of Structural Materials «Prometey» named by I.V. Gorynin of National Research Center «Kurchatov Institute», Saint-Petersburg, Russia

The paper investigated the bainitic steel of 10KHN3MFA grade, which is characterized by the increased tendency to display structural heredity in the forgings with large cross-sections. The samples have been studied for kinetics of diffusional transformation process both under continuous cooling and isothermal conditions, as well as its microstructure. It is determined that in the range of subcritical temperatures with cooling from 900 °C in the studied steel, the initial stage of separation of the ferrite phase takes place. It is shown for the first time that the diffusional ferrite-pearlite transformation fades. Previously it was believed that the beginning of transformation under isothermal conditions proceeds to the end. It was found out that the transformation begins immediately after the beginning of isothermal holding, without the generally accepted incubation period

Key words: diffusional transformation, dilatometry, isothermal holding, structural heredity, annealing, preliminary heat treatment

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Introduction. In various branches of mechanic engineering – mining, mineral processing, power plant engineering, shipbuilding – alloy steels are used for production of shafts, large-size gears and other elements of units with large cross sections [3, 10]. In this case, especially for heavy duty structural elements and units, they use alloy steels of high hardenability, including Cr-3Ni-Mo-V-compositions. These steels, providing a high level of strength and hardenability, are to varying degrees susceptible to the manifestation of structural heredity [12, 13], which consists in the preservation of large hereditary grains after the cycle of preliminary and final heat treatment, which accordingly leads to decrease in plasticity and cold resistance properties when operating at low temperatures.

In practices of heat treatment of heavy forgings in order to overcome the propensity to structural heredity (SH), the annealing operation must be performed in the early stages of heat treatment before forging, it helps to obtain a structure of a hypopearlitic type. For steels with low stability of austenite, this structure can be obtained by existing technological processes [15]. The formation of a hypopearlitic structure in steels with high alloy characteristic causes difficulties due to the increased stability of austenite to the transformation in the diffusion area.

The absence of this type of transformation or its long delay leads to complications in obtaining a qualitative structure and a high complex of mechanical properties [12, 16] in the following cases:

1) presence of heterogeneous and coarse-grained structure, even after the final heat treatment, which is associated with a great propensity to structural heredity of ordered structures;

2) possible increased content of hydrogen and, as a consequence, formation of flakes;

3) reduced plasticity and brittle fracture resistance characteristics.

Research techniques and analysis of results. We have studied the bainitic steel of 10KHN3MFA grade. When using this type of steel, as well as steels with similar type of alloying, in heavy forgings, the problem of structural heredity arises because of the prolonged high-temperature heating that occurs during forging, the higher end-forging temperature, and the difficulty in achieving the diffusional transformation of austenite at the annealing stage [16]. In heavy forgings with uneven deformation at different temperatures with several removals and heating up to 1200-1240 °C, there is natural non-homogeneity of grain sizes. After forging production cycle, preliminary heat treatment

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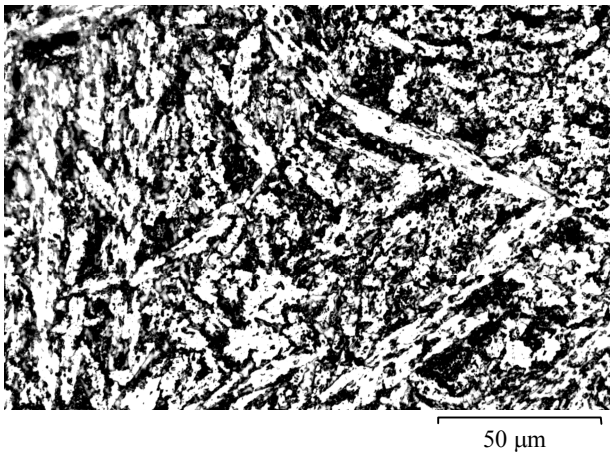


Fig.1. Microstructure of the studied steel after overheating. Zooming up 500^{x}

(PHT) is required, combining usually single or multiple heating above A_{C3}, less often they use isothermal holding and tempering.

This research has been carried out in accordance with the provisions developed in [4, 16] on the effectiveness of the application of isothermal annealing to obtain a more dispersed structure. This study was performed to examine the use of isothermal annealing for steel with increased austenite stability. The purpose of this paper is to determine the conditions for obtaining a hypopearlitic structure in Cr-3Ni-Mo-V steel with high austenite stability, in which only bainitic and martensitic transformations are observed according to generally accepted concepts.

To simulate the coarse-grained structure of the forging, the samples of the studied steel were subjected to overheating at a temperature of 1200 °C with an exposure time of 20 min and subsequent cooling in the furnace.

As a result of overheating, a widmanstatten bainite steel was obtained (Fig.1). It is known [4] that in steels having a crystallographic ordered structure, particularly widmanstatten one, large grains are formed as a result of heating to temperatures above A_{C3} at a heating rate of less than 10 °C/min. The heating of heavy forged pieces during the heat treatment operation is carried out at a speed of no more than 2-3 °C/min and, consequently, there is no correction of the coarse-grained structure. It is known [12, 16] that there is a relationship between the structural-phase composition in the initial state and the resulting structure after heat treatment. The degree of propensity to structural heredity is most often assessed as resistance to diffusional transformation when crystallographic ordered structures of martensite, bainite or their mixture of different state are formed. In case of heavy forgings, the most efficient method of overcoming the propensity to SH is annealing for obtaining hypopearlitic structures, this operation is part of the PHT (preliminary heat treatment) and HD (hydrogen diffusion). In both cases, the main task is to obtain structures with the most stable composition – high-tempered states or products of austenite diffusional transformation. Therefore, the determination of temperature conditions and kinetics of austenite transformation for target composition steel with sufficiently high austenite stability was an important task of the work (Fig.2).

Dilatometric studies on heating and cooling were performed on a high-speed quenching/deformation dilatometer Dil 805 A/D, dilatometric studies with isothermal exposures were carried out on a dilatometer DIL 402 C, the calculation of the temperature-time conversion characteristics was performed in the Proteus Analysis program, microstructure studies were performed on an inverted microscope Axio Observer.A1m, quantitative analysis of the microstructure was done by digital metallography methods [2, 9, 12, 14] in the Clemex Vision Pro software. The etching of the samples was carried out in a 4 % alcohol solution of nitric acid [8].

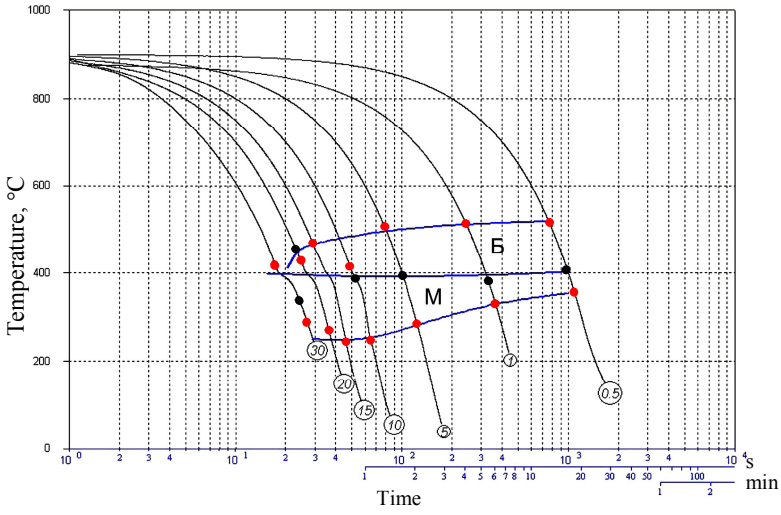


Fig.2. CCT diagram for steel 10KHN3MFA
Circled numbers are the cooling rates

The critical points were determined for a given steel with heating rate

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of 6 °C/min: A_{C1} = 730 °C and A_{C3} = 810 °C. The CCT diagram of the austenite transformation upon cooling from 900 °C was constructed (Fig.2). As it is seen from the chart, this type of steel in the entire range of cooling temperatures at a rate of more than 0.5 °C/s shows no diffusional transformation during continuous cooling.

Consequently, it is difficult to achieve the diffusional transformation during continuous cooling. To determine the conditions for possible diffusional transformation, additional studies were carried out. The steel was heated to an austenite state (900 °C), then cooled to subcritical temperatures and held at these temperatures in accordance with the schematic course of the experiment (Fig. 3).

The study of transformations in steel of 10KHN3MFA grade with isothermal holding at temperatures of 560 and 680 °C showed (Fig. 4, *a, b*) that diffusional transformation at these temperatures is observed in small quantities, with isothermal holding at 560 °C it is unstable. The initial stage of hyppopearlite transformation under isothermal exposure at a temperature of 680 °C occurs with the initial release of ferrite in a period of ≈300 minutes, then the diffusional transformation fades. The main transformation at both temperatures occurs in the bainite area when the sample is cooled.

The study of transformations in 10KHN3MFA steel with isothermal holding at temperatures of 600 and 640 °C showed that the main transformation at these temperatures takes place in the diffusion area, with prolonged isothermal exposure it is incomplete (Fig. 4, *c*), and upon cooling the material also undergoes transformation in the bainitic area.

The diffusional transformation is characterized (Fig. 4, *c*) by the almost complete absence of an incubation period prior to the separation of α-phase and further unstable transformation. The burst of the transformation intensity with respect to time of 1150-1200 min seems to be due to the concentration of carbon in the austenite, which facilitates the passage of the pearlite transformation after the initial phase of ferrite separation.

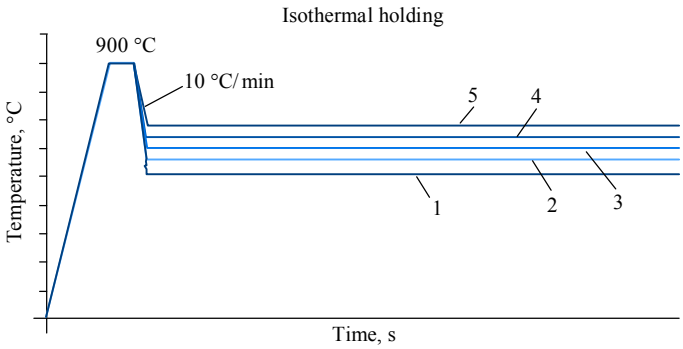


Fig.3. Scheme of the experiment
1 – 480 °C; 2 – 560 °C; 3 – 600 °C; 4 – 640 °C; 5 – 680 °C

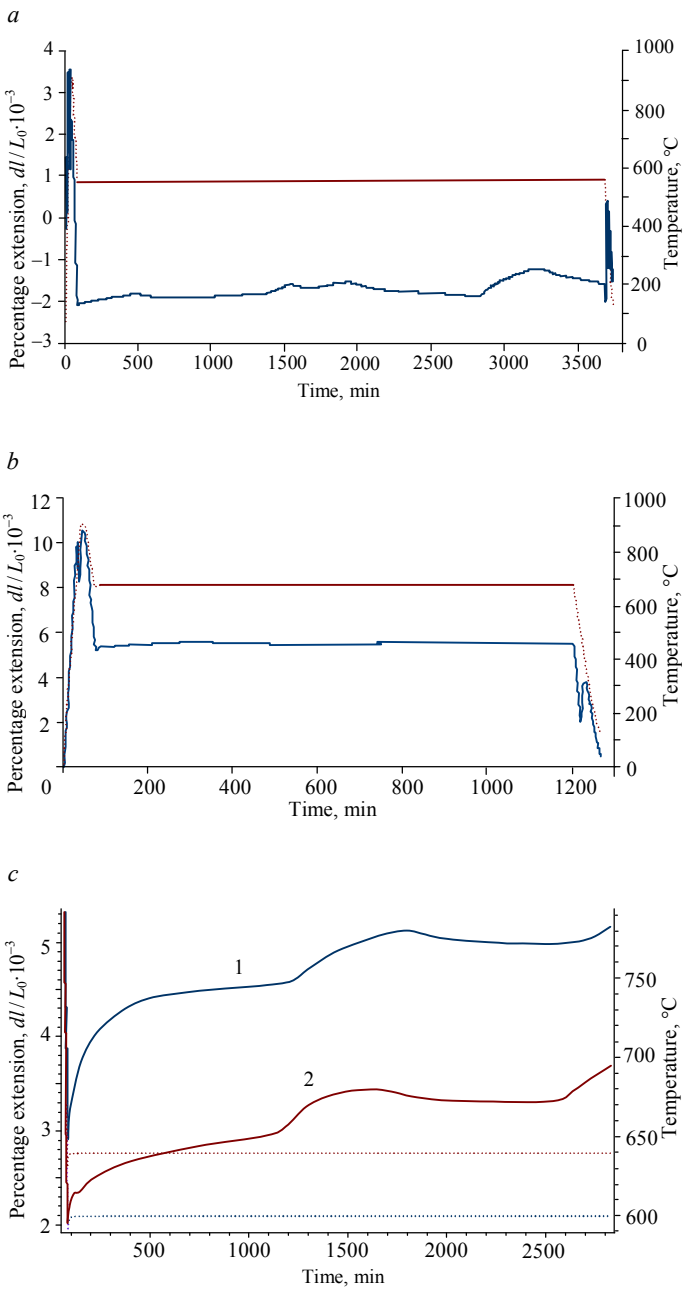


Fig.4. Transformation of steel 10KHN3MFA with isothermal holding at the temperature of:
a – 560 °C; *b* – 680 °C; *c* – 600 °C (1) and 640 °C (2)

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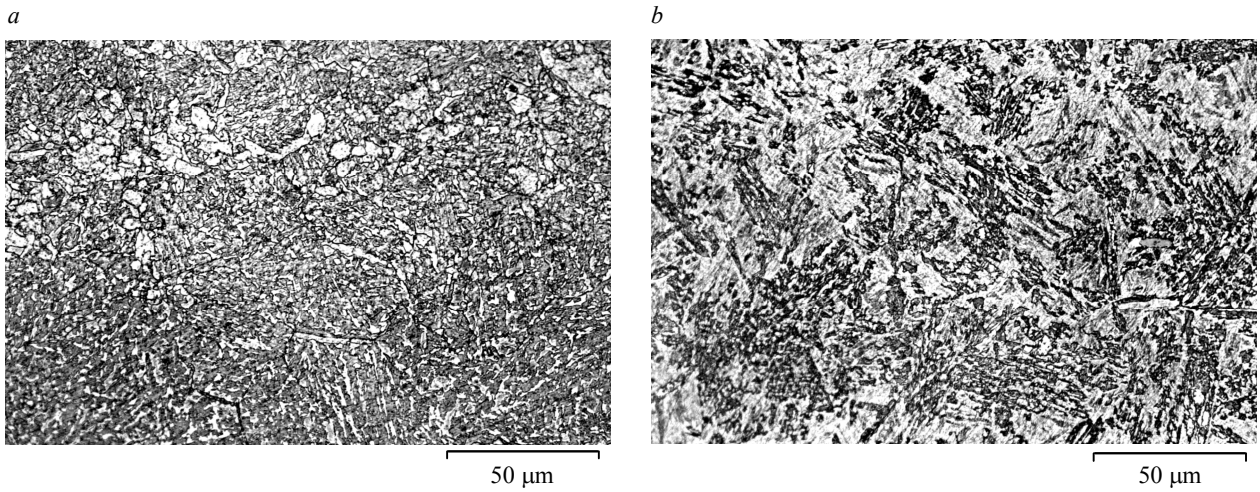
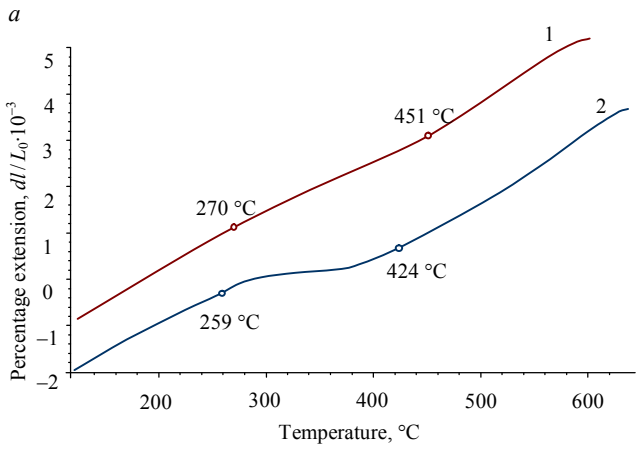


Fig.5. Microstructure of steel samples after isothermal holding at the temperature of 560 °C (*a*) and 680 °C (*b*)

It can be seen from Fig.4, *c* that under conditions of isothermal holding at a temperature of 600 °C, the diffusional transformation already occurs at a higher rate at the initial stage than at a temperature of 640 °C.

Microstructural studies showed that after isothermal holding at a temperature of 680 °C and cooling, an acicular bainitic martensite structure with a small content of the ferritic phase (<3 %) is formed, and the resulting structure after isothermal holding at 560 °C and cooling is bainite with a small content of ferrite (Fig.5). Most likely, this type of the transformation is confirmed by the fact that slight transformation at 680 °C occurs near the point Ar_3, and the transformation at 560 °C is near the lower boundary of the point Ar_1. This type of the dilatometric curve indicates that the process does not affect the potential diffusional transformation zone (passes along its upper or lower boundary). This explains the low intensity and presence of only initial phase of the diffusional transformation.

The temperatures of the beginning and the end of the transformation occurring upon cooling after tempering are shown on dilatometric curves (Fig. 6, *a*).



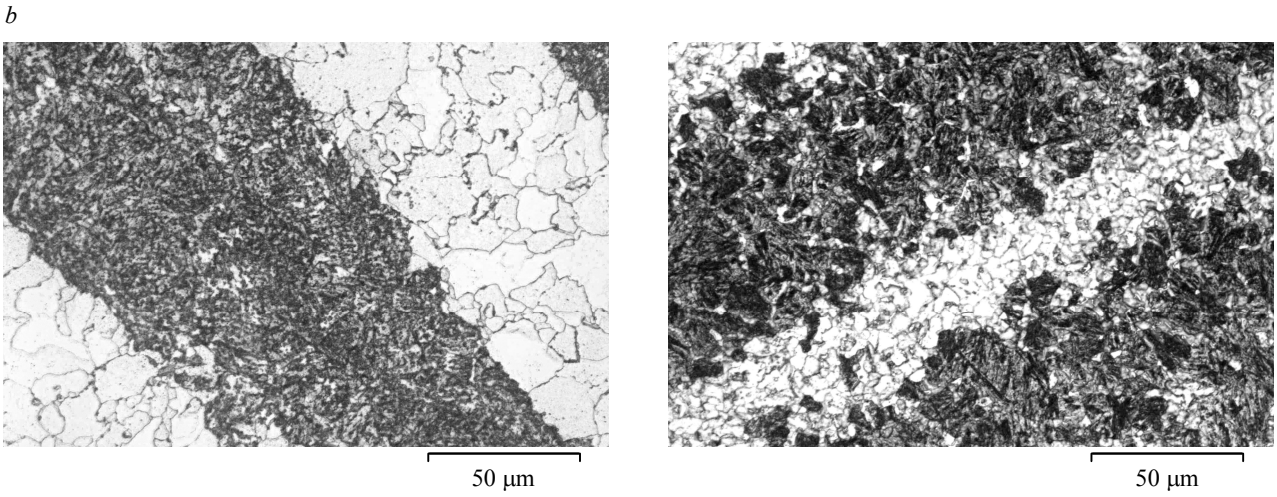


Fig.6. Transformation occurring during cooling after isothermal holding at the temperature of 600 °C (1) and 640 °C (2):
a – dilatometric curve; *b* – samples microstructure: 600 °C (on the left); 640 °C (on the right)

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Analysis of the cooling curves showed that the conversion upon cooling in both samples, after isothermal holding at a temperature of 600 °C, and at a temperature of 640 °C begins in the bainite area and ends already in the martensitic area. A comparative analysis of two dilatometric curves shows that after isothermal aging at a temperature of 640 °C, the transformation takes place in in a more intensive way upon cooling. Consequently, at a temperature of isothermal exposure of 600 °C, the diffusional transformation occurs in more intensively than at a temperature of 640 °C.

Microscopic analysis showed that after isothermal exposure at temperatures of 600 and 640 °C a mixed structure with ferrite and bainitic-martensitic areas is formed (Fig. 6, *b*).

Based on the results of microstructural quantitative analysis of the samples, it is also evident that, after isothermal holding at 600 °C, the content of the ferrite phase is ≈ 40 %, and at a temperature of 640 °C it is ≈ 20 %.

In in accordance with the schematic course of the experiment, we have also conducted a study of transformations in 10KHN3MFA steel with isothermal holding at $t = 480$ °C (Fig. 7).

The study of transformations in 10KHN3MFA steel with isothermal aging at $t = 480$ °C showed (Fig. 8) that when the temperature goes down from 900 °C before isothermal holding, a transformation occurs, its temperature-time characteristics show that this transformation is not a ferrite-pearlite, but bainitic one.

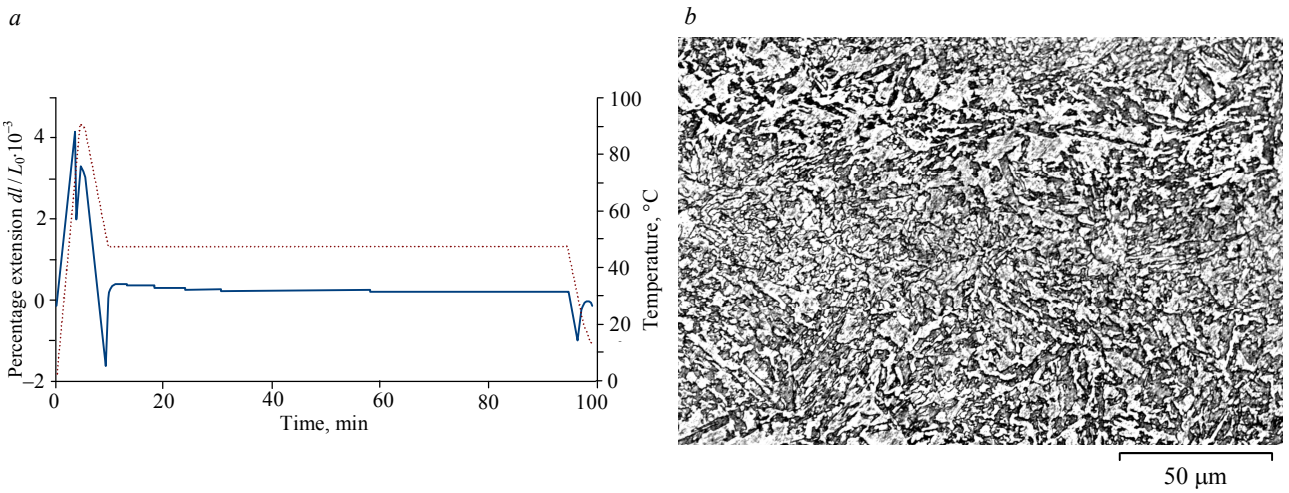


Fig.7. Transformation of steel 10KHN3MFA at isothermal holding at the temperature of 480 °C:
a – dilatometric curve; *b* – samples microstructure

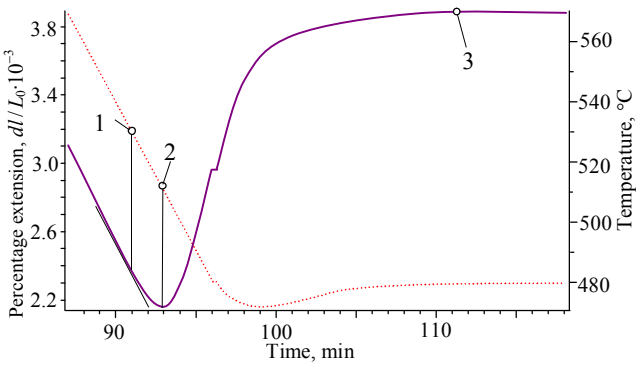


Fig.8. Bainitic transformation during cooling before isothermal holding at the temperature of 480 °C
1 – 91.1 min, 530 °C; 2 – 92.9 min, 512 °C; 3 – 111.3 min

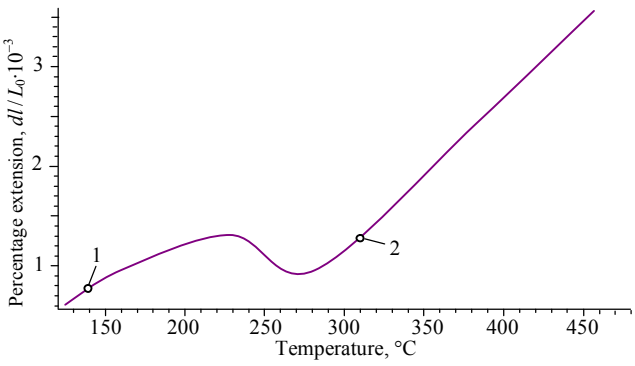


Fig.9. Transformation during cooling after isothermal holding at 480 °C
1 –138,5 °C; 2 – 310 °C

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From the graph (Fig. 8), it is seen that with continuous cooling the intermediate transformation begins at a temperature of 530 °C, and ends even during isothermal holding at a temperature of 480 °C. The transformation takes place ≈ 17 minutes. The curve at a temperature of 512 °C corresponds to the highest conversion intensity, then, under isothermal exposure, it fades and resumes with further cooling (see Fig. 7, Fig. 9)

In the studied sample the transformation occurs upon cooling in the region of lower temperatures (Fig. 9), the structures of lower bainite and martensite are formed, which is confirmed by the results of microstructural analysis (see Fig. 7, *b*).

Analysis of the research results showed that in the examined samples of steel, which is a steel with increased austenite stability, there is no diffusional transformation during continuous cooling and isothermal exposure at 480 °C.

Conclusions

1. In the subcritical temperature range with cooling from 900 °C in the steel of 10KHN3MFA grade, the initial stage of the ferrite phase separation takes place. This period is preliminary one at an isothermal holding temperature of 600 and 640 °C, after which the A → F + P conversion occurs.

2. It is shown for the first time that the diffusional transfromation A → F + p is damped. Earlier [11, 12] it was believed that the beginning of the transformation under isothermal conditions proceeds to the end. It has been found that the transformation begins immediately from the beginning of isothermal holding, without the generally accepted incubation period, as shown in the existing diagrams of the isothermal decomposition of austenite, including steels with a lower degree of alloying. When constructing the standard isothermal diagrams of the austenite transformation the cooling rate is sufficiently high (50-100 ° C/s), during the cooling time, the preliminary processes of austenite redistribution do not have time to occur, so an incubation period is observed which can last more than a day in steels of the martensitic class. Comparison with the obtained results shows the necessity of modeling the cooling rates in the construction of diagrams that correlate with the actual rates of heat treatment modes. With this information, one can assume that the studied character of the austenite transformation corresponds to the real conditions of heat treatment.

3. To a lesser extent, this process is observed at an isothermal holding temperature of 560 and 680 °C, since these temperatures are lower and higher than the temperature of the potential diffusional transformation.

4. The volume effects of the initial stages of transformation are very small, which is due to the low content of carbon in the studied steel.

5. The temperature of the isothermal exposure of 480 °C is much lower than the area of diffusional transformations and, naturally, upon cooling to this temperature, a bainitic transformation occurs, which does not happen under isothermal exposure and resumes upon cooling. This mode is not suitable for solving the assigned tasks.

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***Authors**:* **Viktor V. Tsukanov**, *Doctor of Engineering Sciences, Associate Professor, Head of laboratory, mail@crism.ru (Central Research Institute of Structural Materials «Prometey» named by I.V. Gorynin of National Research Center «Kurchatov Institute», Saint-Petersburg, Russia*), **Nadezhda V. Lebedeva**, *Candidate of Engineering Sciences, Associate Professor, Head of sector, mail@crism.ru* (*Central Research Institute of Structural Materials «Prometey» named by I.V. Gorynin of National Research Center «Kurchatov Institute», Saint-Petersburg, Russia*), **Yuliya M. Markova**, *engineer, mail@crism.ru* (*Central Research Institute of Structural Materials «Prometey» named by I.V. Gorynin of National Research Center «Kurchatov Institute», Saint-Petersburg, Russia*).

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