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## STUDY OF STRUCTURAL TRANSFORMATION OF HOT-ROLLED CARBON BILLETS FOR HIGH-STRENGTH ROPES FOR RESPONSIBLE APPLICATIONS VIA THE METHOD OF THERMAL ANALYSIS

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## ABSTRACT

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Key words:

shot-rolled steel, structural and phase transformations, critical points, differential scanning calorimetry, DSC curves The method of differential scanning calorimetry (DSC) has been used for examination of structural transformations (forming and decomposition of austenite) in hot-rolled high-carbon steel 80. All three stages of forming of homogeneous austenite can be observed distinctly on the DSC heating curve for this steel. It was established that austenite transformation during continuous cooling (with rates 10 and 50 °/min) occurs in correspondence with the type of carbide-ferrite mixture, with extraction of  $\alpha$ -phase of one morphological structure. The critical points at steel 80 heating are determined. Influence of cooling rate on shift of the Ac<sub>1</sub> and Ac<sub>3</sub> points is shown. Necessity of usage of high temperatures in austenitization furnace to provide wire rod temperature at the furnace exit in the range 930–980 °C is confirmed.

#### Introduction

High-strength reinforced stabilized ropes are the base of modern efficient building technologies for fabrication of prefabricated reinforced concrete with preliminary tension of reinforced bars, as well as constructions with consequent post-tensioning. Equipment and technological routes for manufacture of reinforced ropes used by the leading foreign and domestic hardware enterprises are analyzed. It allowed to establish that the modern development tendencies of science, machinery and technologies are characterized by searching the efficient complex for effect on metal microstructure with combination of processing methods with different physical nature. The aim of this effect is in reaching high-strength state of processing material with adding to it the complex of special properties, meeting the requirements of operating conditions of reinforcing products in iron ore constructions of responsible application.

Detailed examination and understanding of the physical mechanisms of forming of structural-phase states of applied materials is being more and more actual for realization of such tasks. These mechanisms substantiate temperature and temporal, force and deformation parameters of multi-stage treatment and provide maximal possible dispersity degree of steel microstructure parameters and its transition in high-strength state.

The method of trial quenching is used conventionally for examination of structural-phase transformations (critical points). This method is standardized in many countries, however, it has several deficiencies: labour intensity, low productivity and (sometimes) subjectivity. Thereby instrumental

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control methods of investigation, such as dilatometry, differential scanning calorimetry (DSC) and differential thermal analysis (DTA), allowing to fix temperature intervals of pgase transformations and to receive information about the values of critical points in metals and alloys are widely used at recent time. The obtained experimental values are used for building the thermo-kinetic diagram of continuous heating and the diagram of isothermal transformation. These diagrams are applying for examination of the essence of transformation processes occurring in different temperature zones and regularities of decomposition of overcooled austenite.

The regularities of forming of structure and properties of several grades of hot-rolled high-carbon steel billets (e.g. steel 80P via dilatometric method [1]) are examined rather widely at present time. However, such experiments for steel 80 using DTA/DSC method are not described in the literature; nevertheless, they are widely used for investigation of alloyed steels [2–9]. Additionally, DTA/DSC methods are most requested than dilatometric method, owing to their high sensitivity to coursing of phase transformations [10].

Thereby, the aim of this work is formulated as examination of structural-phase transformations of initial hotrolled billets made of high-carbon steel via the method of differential scanning calorimetry.

#### Materials and methods of investigation

To manufacture high-strength reinforced ropes with diameter 6.9-15.2 mm and with operating parameters meeting the requirements of GOST P 53772-2010, the carbon steel with the following chemical composition (mass. %) is recommended: 0.67-0.9 C; 0.17-0.37 Si; 0.4-0.8 Mn; not



Fig. 1. DSC curve and DSC derivative curve (on time) for continuous heating of hot-rolled steel 80 with rate 10 °/min



Fig. 2. DSC curve and DSC derivative curve (on time) for continuous heating of hot-rolled steel 80:

 $1 - \text{with rate } 10^{\circ}/\text{min}; 2 - \text{with rate } 20^{\circ}/\text{min}$ 

more than 0.15 Cr; not more than 0.15 Ni; not more than 0.2 Cu; not more than 0.025 P; not more than 0.025 S. This experimental work has been conducted with the samples of initial hot-rolled billet of steel 80 with chemical composition (mass. %): 0.82 C; 0.29 Si; 0.45 Mn; 0.02 Cr; 0.03 Ni; 0.03 Cu; 0.004 P; 0.002 S.

Thermal analysis via DSC method has been conducted on the sensor for synchronous thermal analysis STA Jupiter 449 F3 of Netzsch company. Disk-shaped samples with diameter 3 mm and height 2 mm have been cut for experiments; then they have been subjected to surface grinding with abrasive paper and degreasing using acetone. Capturing of thermograms has been made in protective controlled atmosphere with technical pure argon (GOST 10157-79). Heating of samples has been conducted in corundum crucibles in the temperature range 20–1000 °C with rate 10 and 20 °C/min, and cooling has been implemented from 1000 to 400 °C with rate 10 and 50 °C/min. The empty crucible has been used as a calibration sample.

Despite of wide usage of DSC methods, there is no common opinion at present time about determination of the finishing temperatures of phase transformations, recorded on DSC curves. It was suggested [11] that continuous heating is connected with termination of diffusion phase transformation in the interval between the extremal value of thermal effect and the temperature of extrapolated end of the effect. At the same time the authors of other researches [12–14] have used either straight the extremal temperature of thermal effect on the curve of continuous heating, or the extremal value of the first partial derivative of initial DSC signal in time as a targeted critical point. The paper [15] counts the inflection temperatures on DSC curves as start and finish of transformation. The values of critical points are different, depending on the method of determination of starting and finishing transformation temperatures. In presented paper the finishing temperature of phase transformation has been determined as the extremal temperature of thermal effect.

#### Experimental results and their discussion

Austenitization temperature is one of the main parameters for selection of the temperature and temporal modes of processing of hot-rolled billets. Modern heat treatment technologies for carbon steels are based on usage of increased metal heating temperatures in austenitization furnace to provide wire rod temperature at the furnace exit in the range 930–980 °C [16–18]. Selection of this temperature range is stipulated by regularities of the process of austenite forming in carbon steels. It should be noted that austenite forming during heating is limited to polymorphic ( $\alpha \rightarrow \gamma$ ) transformation and dissolution in the forming austenite of carbides. These

transformation are connected with enthalpy variation and are recorded as extremal values (peaks) or inflections on the DSC curve. This heating curve of the examined sample is presented on the fig. 1: the deep endothermic effect with maximum at the temperature 744.9 °C and inflection at 930.6 °C is observed. Endothermic effect testifies on ferrite transformation in austenite ( $\alpha \rightarrow \gamma$ ), while inflection indicates on dissolution of carbides in austenite. It is mentioned that heating above the temperature 930.6 °C accompanies with continuation of DSC curve lowering doen to the temperature 950 °C and it is connected with homogenization of austenite. Thereby all three stages of forming of homogeneous austenite are clearly seen on the DSC curve of steel 80. In addition to it, the analysis of DSC curve allows to determine the critical points of hot-rolled steel 80 during heating:  $Ac_1 = 721 \circ C, Ac_3 = 745 \circ C.$ 

Heating rate is considered as an important factor that vary location of  $Ac_1$  and  $Ac_3$  points. Influence of heating rate on location of the typical points of thermal effects on continuous heating curves has been examined via DSC experiments during continuous steel heating with different rates. Analysis of the obtained results (**fig. 2**) has displayed that rise of heating rate leads to shift of the endoeffect temperature interval. It can be noted that lowering of heating rate from 20 to 10 °/min has finalized in the temperature decrease of start (by 2.1 °C) and finish (by 8.8 °C) of endothermic effect. At the same time shift of maximal value (peak) made 6 °C.

The obtained results confirmed the conclusions of authors [19], who have revealed that  $Ac_1$  and  $Ac_3$  points shift by different values in the area of more high temperatures with rise of heating rate, thereby expanding of intercritical interval (ICI,  $Ac_3 - Ac_1$ ). The higher is heating rate the more is this value.

Investigation of austenite transformation in continuous cooling of steel 80 has been conducted at cooling rates 10 and 50 °/min (maximal possible cooling rate of the equipment) after heating of the sample in a calorimetric cell and its holding at this temperature during 10 minutes. Rise of cooling rate also leads to shift of the typical points of exothermic effect that are corresponded to austenite decomposition to ferrite-carbide mixture, towards low temperatures (**fig. 3**). DSC curve presents austenite decomposition during cooling with rate  $50^{\circ}$  (min. it can be observed in the temperature).

with rate 50 °/min; it can be observed in the temperature range 608–663 °C, while austenite decomposition during cooling with rate 10 °/min — in the range 637–672 °C.

Presence of one exothermic peak on the DSC curve and its first derivative (see fig. 3) testifies about extraction of one morphological construction during  $\alpha$ -phase cooling. Thereby, austenite transformation in the steel 80 with composition close to eutectoid one occurs in the mode of carbide-ferrite mixture, with extraction of  $\alpha$ -phase of one morphological construction during continuous cooling (with rate 10 and 50 °/min).

### Conclusion

The obtained experimental data of the DSC method have confirmed necessity of usage of austenitization in the temperature range 930–980 °C in production of high-strength reinforced ropes for responsible applications. This technology provide increase of austenite homogenization degree as well as suppression of free ferrite forming in high-carbon hot-rolled billets. Experimental data of DSC method are informative, their usage in combination with metallographic methods allow to test the heat treatment procedures, providing forming of dispersed ferrite-carbide mixture in steel microstructure.

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# Fig. 3. DSC curve and DSC derivative curve (on time) for continuous heating of hot-rolled steel 80:

 $1 - \text{with rate } 10^{\circ}/\text{min}; 2 - \text{with rate } 50^{\circ}/\text{min}$ 

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