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MODERN APPROACHES FOR STUDY OF EUTECTOID STEEL OXIDATION AND DECARBURIZATION

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Abstract. This paper describes the results of a laboratory study into the high-temperature surface oxidation and decarburization of eutectoid steel performed using thermal gravimetric analysis which makes it possible to understand the steel surface oxidation kinetics in non-isothermal conditions as the steel specimen is continuously heated to a specified temperature. An exponential relationship is obtained between the heating temperature and the iron loss in steel. A relationship is established between the heating temperature applied and the surface oxidation rate observed in a eutectoid steel specimen. It is shown that when the temperature of the specimen is raised from 900 to 1000°C, it leads to a triple increase in the surface oxidation rate, whereas the temperature increase to 1200°C results in an eightfold increase in the surface oxidation rate. It is noted that, within the temperature range of 720-950°C, the phase transformations observed are accompanied with intensified scale formation and surface carbon depletion. Using the emission spectrometry technique, the concentration of carbon is determined in the surface layer in relation to the heating temperature and time. The results obtained indicate that eutectoid steel is subjected to an intense surface decarburization at the temperatures of 600-1200°C.

Keywords: eutectoid steel, thermal gravimetric analysis, differential scanning calorimetry, oxidation, decarburization

1. Introduction

Nowadays high-carbon steel wire rod is in demand on the world market for the production of high-strength rebar stabilized ropes, which are the basis of modern effective construction technologies for the manufacture of precast concrete with pre-tensioning of reinforcement, as well as structures with subsequent tension of reinforcement on concrete. Among the various types of prestressed reinforcement, including smooth and profiled reinforcing wire and rod reinforcement, reinforcing ropes occupy a special place, which is caused by the combination of their properties unattainable for other types of reinforcement.

The raw material for their production is a high-carbon wire rod, obtained in modern high-grade rolling mills. Production of high-strength ropes is a complex technological process with a high metal consumption coefficient. Therefore, the metal intended for production of the specified products shall conform to the rigid requirements imposed to its quality.

When producing wire rods, quality parameters are strongly influenced by processes in continuous furnaces before rolling. Such processes include decarburization and the iron loss in steel, which affect the steel quality during a forming operation, as well as finished steel

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products [1,2,24-26]. Heating of steel billets before hot rolling in furnaces causes intensive development of scale formation, depletion of surface layers with carbon and redistribution of alloying elements in the surface layers. At the same time, the resistance of metal to alternating loads, which is typical for ropes, is determined by the depth of the carbonized layer, that is, the actual difference between the microstructure on the surface and the structure of the base metal.

Decarburization by heating occurs as a result of the interaction of oxidizing gases with carbon, which is in a solid solution or bound to iron carbides. Decarburization rate is determined mainly by the process of bilateral diffusion, which occurs under the influence of the difference in the gradient of media. On the one hand, decarbonizing gases come to the surface layers of the metal, and on the other – the resulting gaseous products containing carbon, move in the opposite direction. In this case, carbon from the inner layers of the metal diffuses into the surface layer. Therefore, decarburization and scale formation, occurring on the metal surface, in most cases are considered together. The evolution of steel rope structures and technologies of their production is due to the constant desire to increase the strength and durability, by strengthening the cold deformation or heat treatment.

When the metal is deformed by cold drawing, by pulling the rebound rolled through a system of monolithic dies, the maximum stresses are concentrated on the surface of the rolled metal. Therefore, in the manufacture of high-quality range of high-carbon rebound rolled it is necessary to ensure a minimum and uniform depth of decarburization on the surface of the wire rod. To solve such problems, fundamental concepts of decarburization, diffusion saturation and oxidation processes are needed, which occur in parallel with decarburization in metals and alloys due to their contact with various gases.

Steel surface oxidation and decarburization issues are rather deeply analyzed and theoretically evaluated, in particular, formation features of a scale phase composition and decarburization of metals and alloys have been thoroughly studied [3-13,28,29]. A general survey of high temperature oxidation in metals and alloys, demonstrating how different environmental conditions and chemical composition of the alloys determine the mode of oxidation process is presented in [14].

Principles governing the oxidation of metals are formulated in [15]. It was shown that theories which have been proposed to explain the growth of thin oxide films at low and intermediate temperatures are based on different rate-limiting processes such as electron transfer at the metal-oxide or oxide-gas interface, ion or electron migration through the oxide under the influence of electrical potential gradients or chemical potential gradients and either with or without space-charge effects and ion transfer at the oxide-metal or oxide-gas interface. These theories lead to inverse or direct logarithmic, parabolic, cubic, quartic or linear equations for oxide growth.

However, despite this, these studies remain relevant at the present time, due to the fact that the technology of obtaining wire rod, remain unresolved a number of important issues related to improving the quality and expansion of the range of finished products, which to some extent depend on the relationship of phase and structural transformations with the processes of scale resistance and decarburization in carbon wire rod.

Among known methods of oxidation kinetics of metals and alloys in different gas medium, in addition to a conventional gravimetric method, a widely used technique is a thermogravimetric method, as the simplest and most reliable one. A method showing good results is a thermal gravimetric analysis (TG), applied together with differential thermal analysis (DTA) and differential scanning calorimetry (DSC), and particularly recommended as reflecting to the fullest extent all the processes occurred during specimen heating and cooling, and ensuring good comparability of results [16,30]. However, having analyzed

literary studies, we found no thermoanalytical studies on eutectoid steel. This paper aims at the thermal analysis of eutectoid steel surface oxidation and decarburization kinetics.

2. Methods

The study was conducted on the eutectoid steel specimen, whose chemical composition is given in Table 1. Microstructure of eutectoid steel is presented in Fig. 1.

Table 1. Eutectoid steel chemical composition, % wt.

C	Si	Mn	S	P	Cr	Ni	V	Al
0.82	0.33	0.59	0.0020	0.0031	0.022	0.032	0.0015	0.0010

The examined high-carbon steel has the following mechanical properties: time resistance to rupture $\sigma_B = 1205$ MPa, conditional yield strength $\sigma_{0,2} = 890$ MPa relative elongation δ_{10} = 9 %, relative contraction φ = 31 %. The structure of the studied samples is a plate perlite. The size of the pearlite colonies is 4...8 μm. Cementite is represented mainly by lamellar form. The interplate distance inside the pearlite colonies in steel reaches a maximum of 0.12 μm.

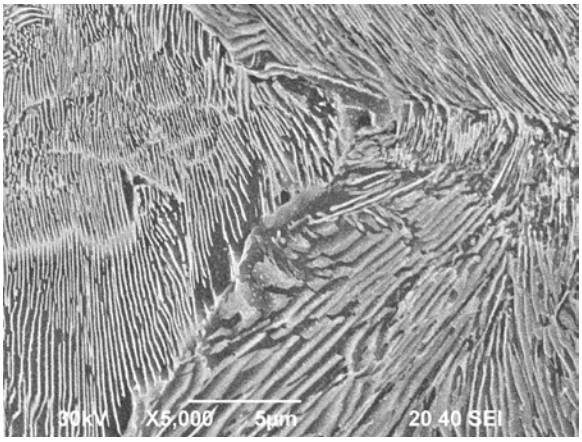

Fig. 1. Microstructure of high carbon steel 0.8 %C

The laboratory study was performed using the STA Jupiter 449 F3 simultaneous thermal analyzer. It ensures both a differential scanning calorimetry and a thermal gravimetric analysis of a specimen at the same measurement, providing a possibility to compare results of TG and DSC directly and eliminate effects of material non-uniformity, specimen preparation and measuring conditions [17,27].

DSC has been used to fix a temperature difference, which is in proportion to a difference in a heat flow between a reference (an empty crucible for STA) and a sample in another crucible from the same material. TG has been used to measure changes in the specimen weight, depending on temperature at specific controlled conditions.

To carry out experiments, we have cut disc specimens, 3 mm in diameter and 1 mm high, ground the surface with an abrasive paper SiC 1200 grit, and degreased with acetone. Weight of the specimens amounted to 55-56 mg. Measurements were performed in corundum crucibles. Before analyzing, the device was calibrated with reference to melting temperatures of pure metals. A temperature measurement error did not exceed ± 0.1°C. During the studies a specimen weight was continuously controlled with an electronic microbalance. Its design ensures a fixed position of the hanging specimen relative to a furnace chamber, when measuring weight. Precision of weighing was ± 0.01 mg.

Thermal curves of the specimens were recorded at a speed of 10°C/min in a flow of argon (protective gas – 10 cm^3/min, working gas – 20 cm^3/min) within a temperature range of 30-1000°C and 30÷1200°C in an argon/air mixture. Such mixture meets requirements for equipment operation: the balance receives argon as protective gas (10 cm^3/min), which is then supplied to the furnace and mixed with air as working gas (20 cm^3/min). A total flow rate of argon and the mixture was 30 cm^3/min. Before measuring the specimen in an argon flow, a specimen holder of DSC together with the crucibles was pre-heated to 1000°C, in an air flow – to 1200°C. When the specimen was installed and the crucible was put on the specimen holder, the furnace was tightly closed and heated as stated in the above. TG and DSC curves were automatically fixed. Data obtained were processed by Netzsch Proteus Analysis software.

To study surface decarburization, specimens (30 mm long and 11 mm in diameter) were put into a high-temperature chamber electric furnace of the PL 20/12.5 type, preheated to a set temperature, held within a specified period (10 and 30 min) at a constant temperature (600, 800, 1000, and 1200°C) and cooled down with the furnace. Carbon content in a surface layer was calculated as per standards ASTM E415-08, ASTM E1086-08, ASTM E1009-95 by the SpectroMAXx optical emission spectrometer.

3. Results and Discussion

It is known [18] that to develop the surface decarburization process, gas atmosphere of the furnace during heating should not produce an intense oxidation effect. Carbides of IVa-Via group metals are particularly vulnerable to the effects of oxygen impurities, and its dissolution in their lattice is accompanied by depositing carbon and a relevant metal [19,20]. Thus, a thermal analysis of eutectoid steel surface oxidation and decarburization was performed in inert (argon) and oxidizing (air) atmosphere.

Figure 2 presents a thermogram of the steel specimen subjected to high-temperature heating in the weakly oxidizing medium, whose oxidizing gases are oxygen impurities (up to 0.002 %) and water (up to 0.001 %), occurring in argon (see GOST 10157-79, Table 1). The DSC curve shows a deep endothermal effect with a peak value at 744.9°C, evidencing a phase transformation of pearlite to austenite ($\alpha \rightarrow \gamma$), and a bend at 930.6°C, corresponding to a breakdown and dissolution of carbides in austenite [21,31].

The TG curve, starting from 589.6°C, shows a minor increase in the weight of the specimen, continuing to 900°C. Within this temperature range, a weight gain amounted to 0.014 % due to steel oxidation. Steel oxidation is taking place at the same time with decarburization, evidenced by weight loss (0.07 %) within a temperature range of 900-1000°C. The decarburization process is clearly observed at 897.1°C and in an active progress to 1000°C. Within this temperature range carbides are broken and dissolved in austenite. As a result, Me-C bonds are broken, carbides are dissolved, while forming carbon (C) and elements, included in their composition (Fe, Mn, Cr), with their further intense oxidation during heating. Carbon oxidation and removal of its oxides into a gas phase contribute to some decrease in specimen weight (i.e. decarburization), which can be seen in the TG curve.

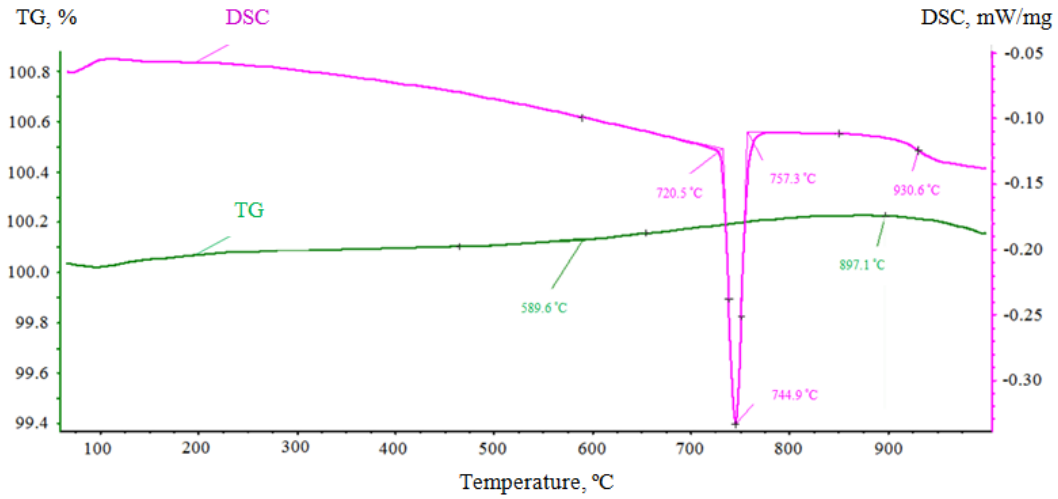

Fig. 2. Thermogram of eutectoid steel specimen continuous heating in argon

Carbon depletion of steel starts from the surface due to oxidation of carbon occurred in a heated specimen as iron carbides, with gases in furnace atmosphere. As a rule, a steel outer layer is almost fully decarburized, which is proved by results of the below experiment (see Table 2).

Table 2. Carbon content on the surface of the eutectoid steel specimen (with a carbon weight percent of 0.82 % in an original specimen) at high-temperature heating in the chamber electric furnace

Temperature [°C]	Carbon weight percent for the holding time	
	10 min.	30 min.
600	0.81	0.73
800	0.60	0.51
1000	0.52	0.448
1200	0.395	0.212

Regarding the above values of carbon content in the steel surface layer with relation to temperature and time, it follows that the surface decarburization process starts at 600°C and intensively takes place to 800°C. When the temperature is over 800°C, the process slows down to some extent, which is explained by a slower decarburization rate than its oxidation rate at 800°C and over, a diffusion rate of carbon towards oxygen is lower than that of iron [22]. An almost two-fold increase in a heating period entails a sharp decrease in carbon content in the steel surface layer, especially at 1200°C. At temperatures around 600°C a eutectoid steel surface decarburization process takes place rather slowly.

A composition of furnace atmosphere together with temperature and the heating period strongly influences both decarburization and oxidation rates. Such processes in an oxidizing medium take place much more intense than in a weakly oxidizing medium, which is obviously shown in the thermogram of eutectoid steel specimen heating in air.

It should be noted that when studying steel oxidation processes during heating before rolling, a term of loss is often used. The iron loss in steel is a loss in weight (due to oxidation of iron, alloying elements, and carbon) of steel after heating [23].

The DSC curve (Fig. 3) in a region of over 850-900°C shows significantly intensified oxidation, which is characterized by exothermal peaks with maximum values at 989.0; 1125.3; 1161 and 1185.6°C. The first peak corresponds to the beginning of intensified

oxidation (the iron loss in steel) after steel transformation into an austenite state (in a temperature range of 720.5-757.3°C) and formation of wustite, and others correspond to continued intensification of the iron loss in steel within a temperature range of up to 1200°C.

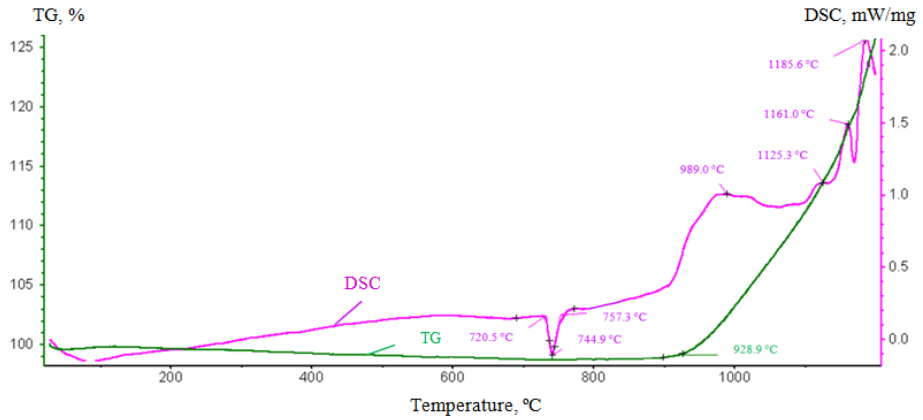

Fig. 3. Thermogram of steel grade 80 specimen continuous heating in air

The TG curve, starting from 900°C, shows a sharp increase in the specimen weight due to oxidation of iron and other components. Within this temperature range, at the same time with breakdown and dissolution of carbides, alloy oxidation processes are under way, resulting in overlapping of effects, while an exothermal effect prevails over endothermal one, and the TG curve shows a weight increase only. It usually occurs, when heating steel before hot rolling at temperatures above 1100°C, and when burning fuel with excess air. There is an exponential relationship between the rate of the iron loss in steel and temperature, which is used to determine a critical temperature [21] for iron-carbon alloys. It should be noted that such relationship, as a change in weight of a heated specimen (%) in relation to temperature, presenting the iron loss, is fixed in the TG curve. A critical temperature for eutectoid steel, calculated by the TG curve, amounts to about 929°C; above this temperature oxidation processes take place at a high rate.

By differentiating the TG curve, we obtain the DTG curve (Fig. 4), allowing us to evaluate a high-temperature oxidation rate of eutectoid steel.

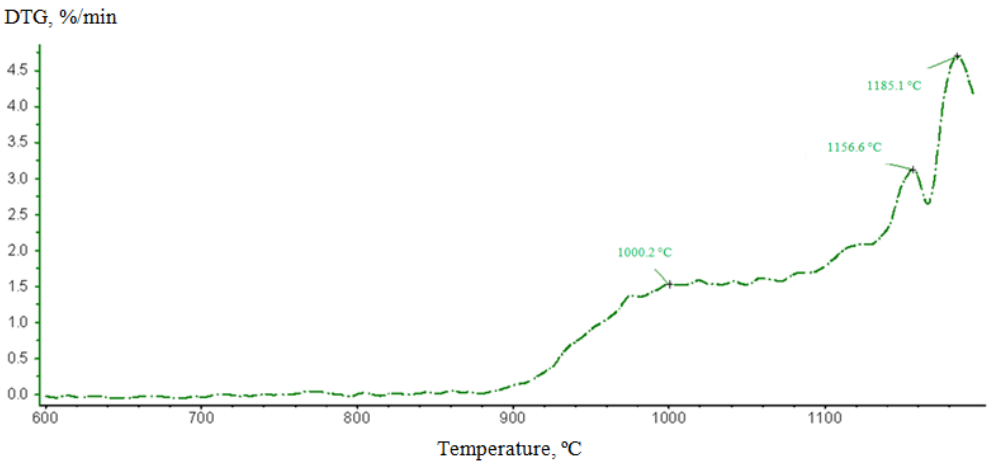

Fig. 4. Relationship between the eutectoid steel oxidation rate and temperature

Judging by the DTG curve, the oxidation rate is constant up to 900°C. When temperature is over 900°C, it steadily increases and achieves a maximum value at 1000°C, and then it starts decreasing almost to 1100°C. Above 1100°C the oxidation rate shows a sharp increase. Thus, when the temperature of the specimen is raised from 930 to 1000°C, it leads to a triple increase in the surface oxidation rate, whereas the temperature increase to 1200°C results in an eightfold increase. A maximum temperature of heating steel before rolling is usually by 100-150°C lower than a solidus curve for eutectoid steel. This temperature is about 1100-1250°C. Therefore, what is important is to follow a minimum steel oxidation rate in a temperature range of 1100°C and over. If one is taken as the oxidation rate at 1100°C, at 1157°C it will increase almost by 1.5 times, at 1185°C – by over twice. Thus, within a temperature range of 1100-1250°C, the minimum oxidation rate of eutectoid steel is at 1157°C. Such temperature is optimum, when heating eutectoid steel before rolling, corresponding with literary data given in [21], namely 1120-1160°C for 70-85 steel grades.

4. Conclusions

1. For the first time, a thermal gravimetric analysis and differential scanning calorimetry have been used to study oxidation and decarburization of a eutectoid steel surface within a temperature range of 20°C to 1200°C in air and argon. Having analyzed the thermograms obtained and identified extreme values of differential curves (TG and DSC), we could describe processes, occurring during heating of eutectoid steel. Within a temperature range of 720-950°C, when heating a eutectoid steel specimen, phase transformations ($\alpha \rightarrow \gamma$) are accompanied with intensified scale formation due to formation of wustite and surface carbon depletion, resulting from breakdown and dissolution of carbides in austenite.

2. It is shown that when heating a eutectoid steel specimen in air medium, the iron loss in steel is sharply intensified at 989-1000°C, entailing an increase in its weight up to 5 %, at 1200°C – to 25 %. A minimum oxidation rate of the specimen within a temperature range of 1100-1200°C is identified at 1157°C.

3. The thermal analysis results obtained have contributed to changes in temperature and time of rolling of eutectoid steel wire rods on hot rolling section mills.

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