

Research of Properties and Structure of Boron-modified Roll-foundry Iron

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Abstract

The aim of the research is to determine the properties and the structure of boron-alloyed iron used for rolls production. It was found that boron can form different carbides in iron, which significantly improve both hardenability and microhardness. Iron borides $Fe_3(B,C)$ are also formed in cast iron as only 40% of carbon atoms in cementite matrix can be replaced by boron. Besides, boron influences the temperatures of structural constituents decomposition (increases the rate of cementite decomposition), it also decreases the temperatures of developing phase transformations. The research group determined the kinetics of martensite decomposition, which is formed when chilled cast iron is poured into a metal mold and then undergoes thermal treatment to the temperature of 400 °C. In the temperature range of 210 – 400 °C the main process is decomposition of the retained austenite into bainite, while in the temperature range of 400 – 500 °C, the main process is decomposition of martensite and forming a ferrite-cementite mixture. In order to get the necessary properties of the roll face, it is necessary to provide its thermal treatment (tempering), when it is heated to the temperature of 400 °C to avoid martensite decomposition, because otherwise in the process of roll operation it can result in crack formation.

Keywords: boron, roll, martensite, tempering, rolling, hardness, cast iron.

1. Introduction

Currently there is a number of well known research works about the effects of boron on steel, but the question of the impact of boron on iron is poorly studied. In steels it is used as an alloying element that increases the wear resistance and high-temperature strength (Bolkhovitinov, 1958). Hardenability of steel is significantly improved by boron while it is in a solid solution (Bolkhovitinov, 1958).

Boron is one of the chemical elements, which has not previously been used in roll-foundry iron. That is why it is necessary to investigate behavior of boron in wear-resistance iron of the roll face, its interaction with other chemical elements and its influence on the structure and properties of roll-foundry iron.

Boron is №5 element of Mendeleev periodic system (group III, period 2), its atomic weight is 10.811, its atomic radius is 0.091 nm. Due to the position in the periodic table and its structure, boron has a high affinity to carbon. However, boron has a larger diameter of the atom than carbon (0.067 nm) and oxygen (0.048 nm), that is why its solubility in iron is lower, but its impact on the iron lattice is stronger than the impact of the elements mentioned above (Vinarova, 1961; Krukovich, Prusakov, & Sizov, 2010).

Boron refines dendritic structure of alloys and increases the amount of carbides formed, in addition, at constant rate of heat removal, the solidification time of samples is markedly increased (Poddubny & Romanov, 1999).

Having studied the two-component equilibrium diagram of Fe-B, three-component equilibrium diagrams of Fe-B-C, Fe-Cr-B, Cr-Mn-B, Cr-Mo-B, Cr-V-B, Fe-V-B, four-component equilibrium diagram of Fe-Cr-Ni-B in (Krukovich, Prusakov, & Sizov, 2010; Krukovich, Prusakov, & Sizov, 2010; Krukovich, Prusakov, & Sizov, 2010; Elliot, 1970; Hansen, 1958; Marder, 1973; Pearson, 1958; Shunk, 1965), the authors discovered that boron is capable of forming chemical compounds with such elements as Fe, C, Mn, Cr, Ni, Mo, which are present in the grade of alloyed roll-foundry iron under study.

Boron reacts with metals forming solid solutions and chemical compounds, borides (Krukovich, Prusakov, & Sizov, 2010); moreover, Voroshnin, Liakhovich, Panich, and Protasevich (1970); Voroshnin and Liakhovich (1978); Krukovich (1995); Dubinin and Kogan (1979); Thomas and Leak (1955); ASM (1988) found that iron

borides have increased microhardness in the range 1320...2800 HV. Vinarova (1961); Voroshnin, Liakhovich, Panich, and Protasevich (1970); Kaufman (1969); Carroll, Darken, Filer, and Zwell (1954) found that boron is capable of replacing carbon atoms in the compounds of Fe-C, forming the so-called "borocarbides of iron" or "borocementite" compounds, having chemical formulas $Fe_{23}(C_{1-x},B_x)_6$ and $Fe_3(C_{1-x},B_x)$.

2. Research Technique

Alloyed cast iron «ЛПХМД-70» used for roll face production has been researched. It has the following chemical composition, wt%: C - (3.0-3.4), Si - (0.7-1.2), Mn - (0.5-1.2), S - <0.10, P <0.15, Cr - (1.4-2.0), Ni - (4.0-4.7), Mo - (0.20-0.50). Ferroboron «ФБ20» (GOST 14848) containing 20 % of boron was used to modify the cast iron.

Determination of chemical composition of the samples of roll-foundry iron with different boron content was carried out by spectral and X-ray methods of analysis. Chemical composition was determined using the spectrometer OBLF GS 1000 according to the methods described in GOST 27611-88 "Cast Iron. Photoelectric method of spectral analysis", and using X-ray method - on the x-ray fluorescent spectrometer «Tiger S8».

Some samples were produced in a 60kg induction furnace with basic lining to investigate the influence of boron on the structural constituents and mechanical properties of the roll-foundry iron grade under study. Boron content in the cast iron of experimental heats varied, wt%: 0.01...0.09. The test samples were cast into a dry sandy-clay form (SCF), a wet SCF and a chill mold (C). Chemical composition of the obtained test samples is shown in Table 1.

The research group conducted the research work using X-ray diffractometer (model «Advance D8» of «BRUKER AXS GmbH») to study the chemical compounds of boron with iron and boron with carbon formed in the roll-foundry iron.

Metallographic investigations of the test samples microstructure were carried out using the microscope Olympus GX, with the installed image processing software - a specialized spreadsheet for processing SIAMS Photolab images. Photographs were taken before etching at 100 times magnification, after etching - at 100 and 1000 times magnification. The solution of 5 ml HNO₃ + 50 ml HCl + 50 mL H₂O was used for etching of the samples (GOST 5639-82).

Table 1. Chemical composition of roll-foundry iron samples modified by boron

Heat №	Chemical composition, wt, %								
	C	Si	Mn	S	P	Cr	Ni	Mo	B
1	3.12	0.70	0.82	0.017	0.050	1.70	4.67	0.45	-
2	3.14	0.69	0.85	0.016	0.050	1.72	4.66	0.43	0.01
3	3.13	0.73	0.84	0.015	0.049	1.69	4.65	0.42	0.02
4	3.14	0.72	0.83	0.014	0.051	1.71	4.67	0.44	0.03
5	3.15	0.69	0.85	0.016	0.052	1.70	4.66	0.45	0.04
6	3.13	0.71	0.84	0.014	0.051	1.72	4.68	0.43	0.05
7	3.12	0.72	0.83	0.015	0.049	1.68	4.67	0.44	0.06
8	3.11	0.73	0.83	0.018	0.052	1.71	4.63	0.42	0.07
9	3.15	0.70	0.84	0.017	0.046	1.69	4.66	0.42	0.08
10	3.12	0.71	0.82	0.015	0.051	1.70	4.67	0.43	0.09

Hardness of the samples was determined using a hardness tester GALILEO COMP 25. The research group made at least three measurements of hardness of each sample.

Microhardness of the samples was measured using the hardness tester MH-3. Microhardness of both metal matrix and cementite constituent was measured.

Castability was determined using a test helical sample with a trapezoidal channel of 6×8 mm. Linear shrinkage was defined as the difference between the linear dimensions of casting pattern and the casting after complete cooling.

Determination of the samples wear resistance was carried out at the laboratory installation (Figure 1) using the method of testing of material wear resistance by friction against loose abrasive particles. Steel 45 was used as a reference standard and corundum grit №16П was used as an abrasive.

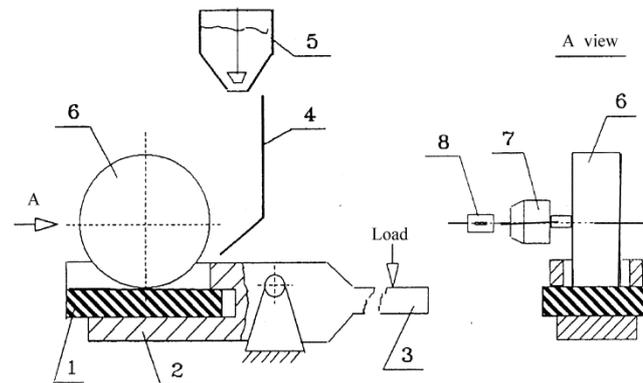


Figure 1. Installation for wear tests: 1 – sample; 2 – sample adapter; 3 – lever; 4 – guiding tray; 5 – metering device; 6 – roller; 7 – drive; 8 - revolutions counter of roller

Changes in the phase composition in the process of heating and cooling of samples were studied using a synchronous thermoanalytical apparatus STA (Jupiter 449 F3) produced by “NETZSCH” under continuous heating of the sample at the rate of 30 °/min. in the inert gas (argon) atmosphere to the temperature of 1000 °C. The accuracy of temperature measuring of the sample was 0.1 °C. The thermocouple was calibrated by the melting temperatures of the state standard reference samples where pure metals were used. Structural characteristics of the test samples of cast iron with similar chemical composition without any thermal treatment were determined for samples with the following boron content, wt, %: 0.0, 0.03 and 0.091 and for the thermally treated sample containing 0.076 wt, % of boron.

X-ray diffractometer (Figure 3) was used to study the microstructure of the test samples (Figure 2) and their phase composition.

The chemical composition of the cast iron is given in Table 2. The test samples were cast into sandy-clay form.

Table 2. Chemical composition of the samples, wt, %

Sample №	C	Si	Mn	S	P	Cr	Ni	Mo	B
1	3.12	0.72	0.82	0.014	0.050	1.68	4.67	0.42	0.091
2	3.13	0.70	0.83	0.014	0.051	1.71	4.68	0.42	0.062
3	3.12	0.73	0.83	0.014	0.051	1.70	4.65	0.42	0.057

The tensile strength, the relative elongation were determined using a standard sample 20 mm in diameter, the impact strength was determined using a sample 55x10x10 mm, V concentrator, 0.25 mm in radius (Table 3).

Table 3. Mechanical properties of roll-foundry iron before alloying and after it

Impact strength, J/cm ²	Hardness, HRC	Yield stress, σ_T , MPa	Ultimate resistance σ_B , MPa	Relative elongation, δ , %	B, wt, %
3.65	51	74	396.5	1.60	-
3.71	55	73	411.4	1.55	0.033

3. Results and Discussion

Having studied the influence of boron on the microstructure of the roll-foundry iron cast into a sandy-clay form and a chill mold, it was found that the microstructure consists of austenite matrix with a small amount of martensite needles (up to 3 %) and carbide constituent established in the matrix. The microstructure and the phase composition of the test samples are given in Figures 2-4.

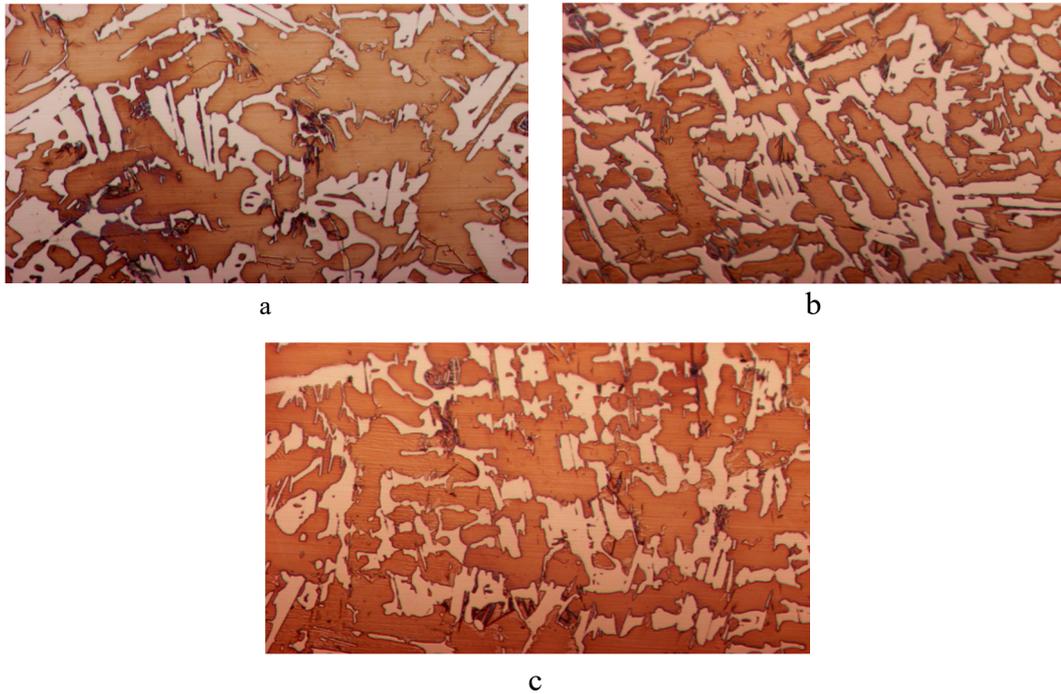
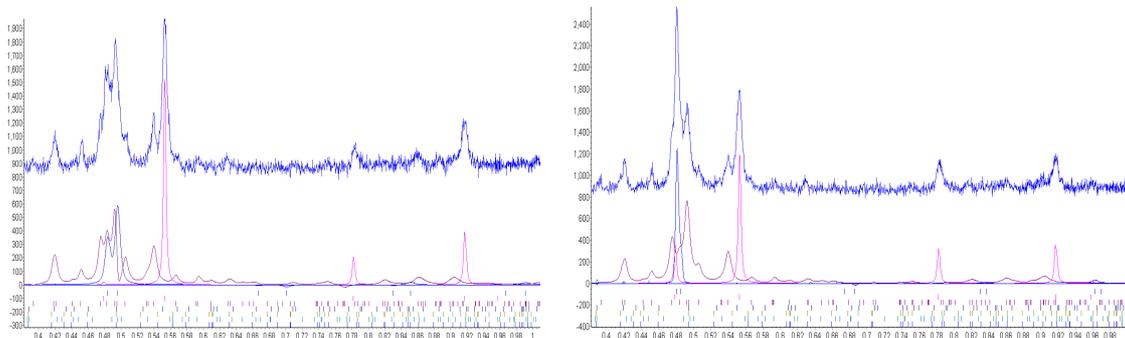


Figure 2. Microstructure of cast iron (x500):

a – boron content is 0.091 %, b – boron content is 0.062 %, c - boron content is 0.57 %



Alpha Iron tetra	8.95 %
Iron gamma Austenite	52.01 %
Cementite Fe ₃ C	39.00 %
B ₁₀ C	0.00023 %
B ₄ C	0.00031 %
B ₁₃ C ₂	0.00004 %
Fe ₃ B	0.0034 %

a

Alpha Iron tetra	6.54 %
Iron gamma Austenite	52.83 %
Cementite Fe ₃ C	30.51 %
B ₁₀ C	0.00016 %
B ₄ C	0.00032 %
B ₁₃ C ₂	0.00009 %
Fe ₃ B	0.0028 %

b

Figure 3. Diagrams of phase composition of roll-foundry iron

a – boron content is 0.091 %, b – boron content is 0.062 %



Figure 4. Microstructure of cast iron (x 1000): boron content is 0.033 %

Table 4. Martensite grade of the test samples of cast iron

Boron content in test samples of roll-foundry iron, wt, %	-	0.01	0.02	0.03	0.04	0.05	0.06	0.07	0.08	0.09
Martensite, grade GOST 8233-56.	7-10	5	2-3	1-2	2-7	5-6	4-7	4-7	4-8	3-7

The microstructure of the investigated cast iron cast into a chill mold is the following: Bainite + Martensite + and Cementite. No graphite was found in the samples produced by casting into a chill mold. The amount of martensite in the metallic matrix is 15-25 %, the rest is bainite.

It was found that changes in boron content in roll-foundry iron cause changes in martensite grade in the metal matrix (Table 4).

Peaks corresponding to compounds of boron with iron and boron with carbon were found in the diagrams of phase composition of the samples. Among the compounds of boron with iron only one type of borides Fe₃B was found, although Vdovin and Zaitseva (2011) describe the existence of four types of iron borides: Fe₃B, Fe₂B, FeB and FeB₂, which can be formed in Fe-C alloys. This is due to a temperature barrier for the possible formation of the other borides during production of the roll-foundry iron under study. Temperature ranges for formation of borides Fe₂B, FeB, FeB₂ and Fe₃B are in the ranges of 1389...1407 °C, 1540...1652 °C, 2070 °C ± 50 and 1150...1250 °C respectively (Krukovich, Prusakov, & Sizov, 2010). Only borides type Fe₃B are formed in iron structure, because the melting temperature of the analyzed roll-foundry iron is lower than the temperatures of formation of iron borides types Fe₂B, FeB, FeB₂.

It should be noted that the metal matrix of cast iron with boron content 0.062 % is distributed more evenly than with boron content 0.091 %. The following relation between the amount of boron in cast iron and martensite needle was obtained (Figure 5).

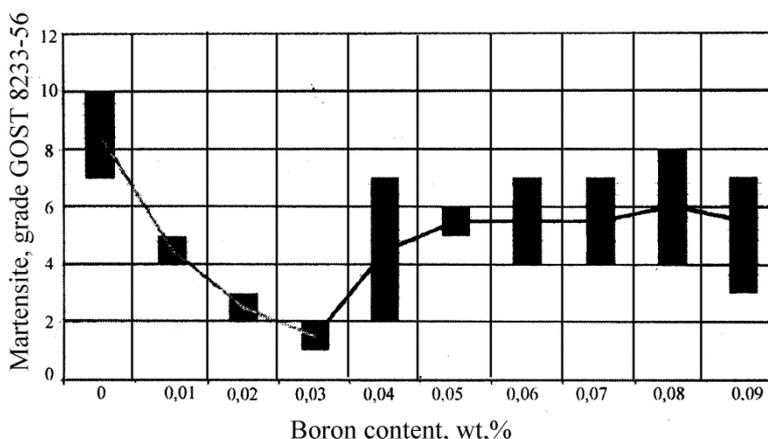


Figure 5. Relationship between martensite needle and boron content

At the same time, according to the analysis of the phase composition (Figure 3), cementite contains solid solution of boron and different boron carbides.

The following boron carbides were found in the test samples: B_4C , $B_{10}C$ and $B_{13}C_2$ (Figure 6).

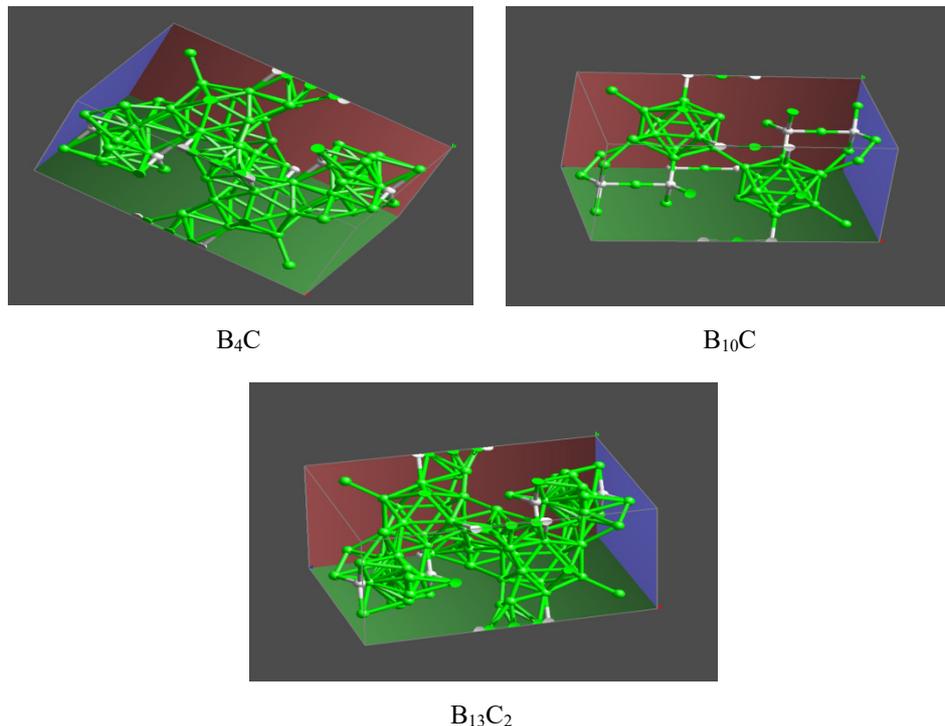


Figure 6. Unit cells of boron carbide atoms

Of all the compounds of boron with iron, borides type Fe_3B , which can exist only in the presence of carbon, are of particular interest. In this case, they are the product of substitution of carbon in cementite lattice and have the formula $Fe_3(B,C)$. Boron is able to substitute 40 % of carbon atoms in the phase $Fe_3(B,C)$ without changing its crystalline structure. The formed borocarbides promote the increase of cementite phase microhardness and, consequently, the increase of roll-foundry iron hardness (Krukovich, Prusakov, & Sizov, 2010).

Rolls «LPXNMd» studied in the present paper have a strong core and hard wear resistant roll face. These rolls having martensitic metal matrix and high solid carbides can spall in the process of rolling. It is connected with the fact that martensite formation occurs with an increase in volume, which increases the tension of the roll face. Such rolls are subjected to backing to reduce the stress. The basic requirement is that the chilling part should not undergo any substantial changes either during the heat treatment of the roll or in operation (Vdovin, Pivovarova, & Zaitseva, 2014).

The complexity of phase transformations during tempering is caused by the possibility of partial or total carbon diffusion and different ways of structural changes in the carbide phase and the metal base. The existence of some of them is easily detected by thermal (DTA or DSC) methods having high sensitivity to the development of phase transformations. These methods are based on the measurement of absorbed or released heat in the process of programmed heating.

The samples without heat treatment were studied by calorimetry (DSC) method at the first stage of research of kinetics of martensite decomposition and development of other structural transformations in the chilled cast iron of the roll face with the following microstructure (Figure 7): the metal base of the samples is austenite with martensite needles that started to form: №1 sample – 95 % of austenite and 5 % of martensite, №2 sample – 95 % of austenite and 5 % of martensite and №3 sample - 92-100% of austenite, 0-8 % of martensite. After that the mass (TG) and heat flow rate curves (Figure 8 and 9) were drawn for the test samples.

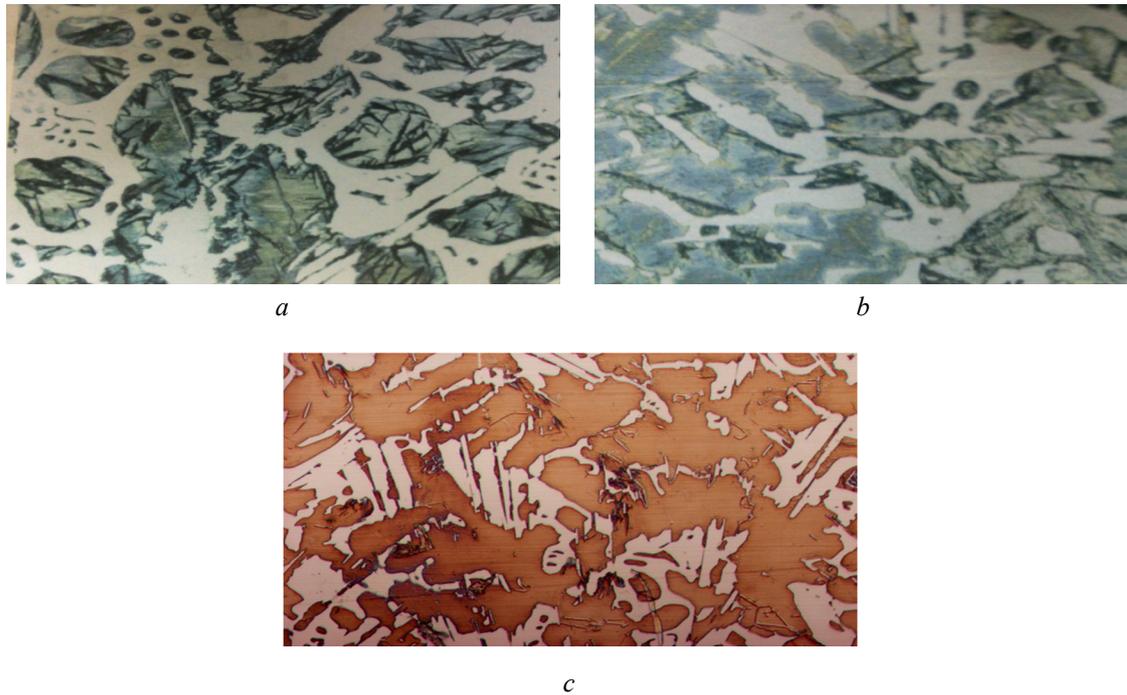


Figure 6. Microstructure of the test samples of cast iron that did not undergo heat treatment (x500):
a – without boron, *b*– boron content is 0.03 %, *c* – boron content is 0.09 %

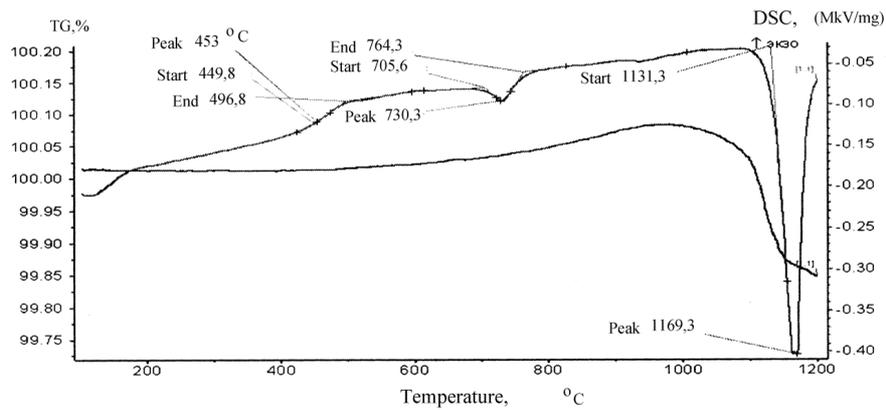


Figure 7. The mass (TG) and heat flow rate curves of the untreated roll-foundry iron without boron

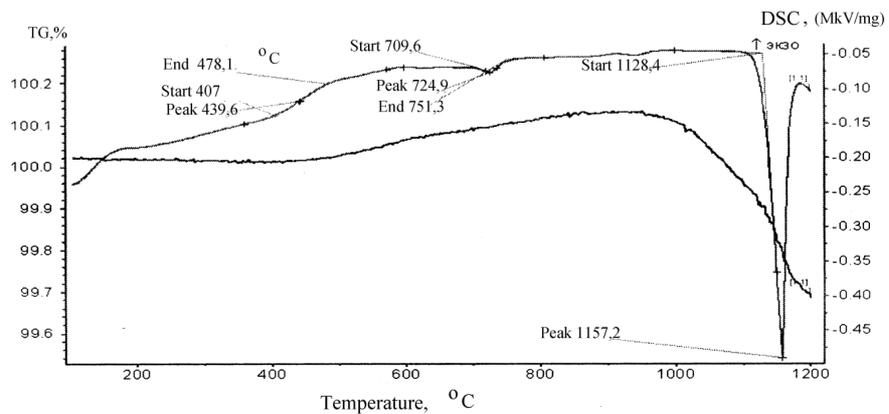


Figure 8. The mass (TG) and heat flow rate curves of the untreated roll-foundry iron containing 0.03 % of boron

Percentage ratio of structural constituents in the test samples of untreated cast iron is shown in Table 5.

Table 5. Structural characteristic of the test samples of untreated cast iron

Sample №	Boron content, wt, %	Metal matrix, %	Cementite, %
1	-	63	37
2	0.03	64	36
3	0.09	65-70	30-35

There are three thermal effects on all mass (TG) and heat flow rate curves of the test samples. The exothermic effect in the temperature range of 400...500 °C corresponds to the decomposition of martensite into ferrite-cementite mixture. The first endothermic effect in the temperature range of 700...770 °C corresponds to the transformation of ferrite-cementite mixture into austenite (austenite transformation), the second effect corresponds to decomposition (splitting) of cementite (1100...1200 °C). In this case, the decomposition rate of cementite for the sample without boron is 0.4 g/h, for cast iron containing 0.03 % of boron the decomposition rate is 0.5 g/h, and for cast iron containing 0.09 % of boron it is 0.65 g/h (Vdovin & Zaitseva, 2011). It was found that boron influences the decomposition rate of cementite, the increase in boron content in chilled alloyed cast iron of the roll face results in the increase of the decomposition rate of cementite.

Temperatures of the start, maximum and end of phase transformations for roll-foundry iron «ЛПХМД-70» during heating are shown in Table 6.

Table 6. Temperature range of phase transformations for roll-foundry iron

Boron content, wt, %	Decomposition temperature, °C								
	of martensite			of ferrite-cementite mixture			of cementite		
	start	max.	end	start	max.	end	start	max.	end
-	450	453	497	706	730	764	1131	1169	≥1200
0.03	407	440	478	710	725	751	1128	1157	1200
0.09	424	439	475	709	723	753	1129	1152	1200

It was found that the temperature maximums of transformations occurring in cast iron during heating reduce their values with the increase of boron content in cast iron.

At the second stage of the study of martensite decomposition kinetics and development of other structural transformations in chilled roll-foundry iron using heat treated samples, the research group determined the nature of development of phase transformations in the sample made from the analyzed roll-foundry iron modified by boron before and after heat treatment. Chemical composition of the analyzed cast iron is shown in Table 7. Temperature-depth profile of chilled cast iron before heat treatment is shown in Figure 10.

Chemical composition of the test samples, wt, %

Table 7. Chemical composition of the samples, wt, %

C	Si	Mn	S	P	Cr	Ni	Mo	B
2.8	0.45	0.75	0.010	0.040	0.8	4.1	0.5	0.076

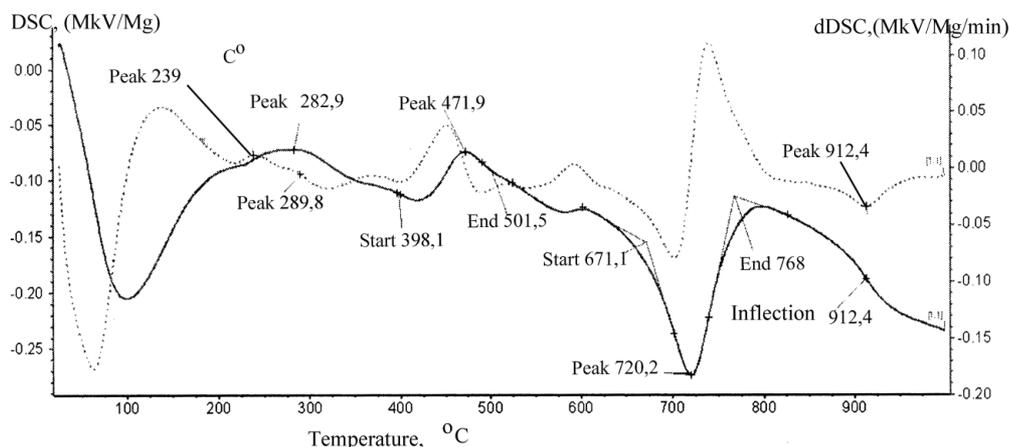


Figure 9. DSC - curve and DSC curve derivative of continuous heating of chilled cast iron «ЛПХМД-70» before heat treatment, the heating rate is 30 °C/min

It was found that during heating of the sample, beginning with the temperature of 138 °C (inflection point), the DSC-curve rises smoothly to the temperature of 210 °C that indicates the beginning of the process, which is accompanied by heat generation in the temperature range corresponding to the first stage of tempering. Further, in the temperature range of 210...320 °C the exothermic effect was registered with the maximum at the temperature of 283 °C, corresponding to the transformation of retained austenite. It is consistent with the results of (Vakula, Osipov, & Sudakov, n.d.), where it was experimentally established that in the temperature range of 270...290 °C retained austenite decomposes into bainite in chilled cast iron. Upward peaks on the DSC curve derivative located approximately at the temperatures 230 and 280 °C indicate the overlapping of exothermic effects. In the temperature range that coincides with the second stage of tempering, the basic process is decomposition of residual austenite to bainite. During further heating of cast iron from 400 to 500 °C (the third stage of tempering) on the DSC-curve the clear exothermic effect with the maximum at the temperature 472 °C is recorded, accompanying the main process of martensite decomposition into ferrite and carbides. Similar results were obtained by dilatometric method in (Evsikov, Demina, Hulin, & Loshkarev, 2011) for high-duty and gray cast irons. During further heating of cast iron above 650 °C in the temperature range 700...830 °C, endothermic effect with the peak at 720 °C was recorded corresponding to transformation of ferrite-cementite mixture to austenite (austenitic transformation). During heating from 830 °C to 1000 °C a sharp decline of the DSC-curve (with the inflection point at 912 °C) is observed that indicates that the process is accompanied by heat absorption and it is confirmed by the peak at the temperature of 912 °C on the DSC-curve. Processes of cementite decomposition proceed in this temperature range.

Characteristic temperatures on the recorded effects were identified in addition to the peaks on the DSC-curve: extrapolated start (T_s) and the end of the effect (T_k). We accept that T_s corresponds to the temperature of the transformation start, and T_k corresponds to the temperature of the transformation end. In the sample of roll-foundry iron under study the decomposition of martensite is observed in the temperature range of 398...502 °C. In connection with this, the heat treatment of roll-foundry iron «ЛПХМД-70» should be carried out below 398 °C (Vdovin, Pivovarova, & Zaitseva, 2014).

Due to this, the research group carried out backing of the analyzed cast iron in a laboratory at 390 °C, the microstructure of this cast iron is shown in Figure 10. After thermal treatment the microstructure consists of bainite-martensite matrix with high carbides content. Graphite exists in the surface zone of the roll from ПГФ1, ПГД 180 (at the depth of 45 mm its content is 6 to 7 %), ВГФ3, ВГр2 (at the depth of 15 to 35 mm its total content is 1.5 %) to ШГФ4, ШГД45-90 (at the depth of 55 mm its content is 7.3 %). The structure of the metal base is bainite + retained austenite to the depth of 45 mm, martensite grade is 3, the area of cementite according to the scale is 10A (%) GOST 8233-56 from 29.0 at the depth of 5 mm to 8.5 at a depth of 55 mm.

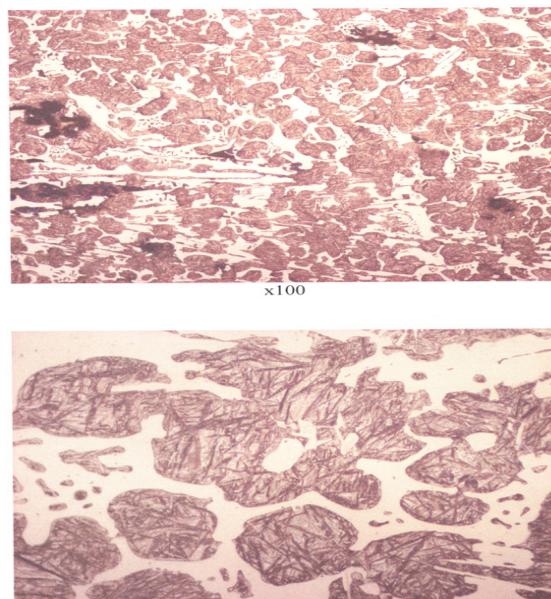


Figure 10. Microstructure of roll-foundry iron «ЛПХМД-70» containing 0.076 % of boron after thermal treatment

Temperature-depth profile of the roll-foundry iron after backing is shown in Figure 11.

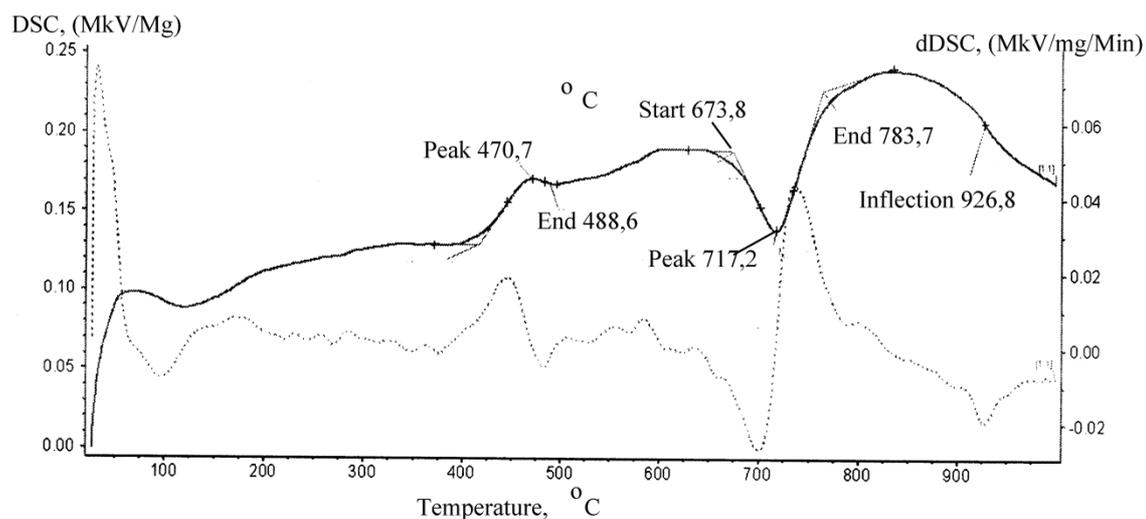


Figure 11. DSC - curve and the DSC curve derivative of continuous heating of chilled cast iron «ЛПХМД-70» after backing, the heating rate is 30 °C/min

There are no thermal effects to the temperature of 400 °C on DSC-curve of the sample of the analyzed cast iron «ЛПХМД-70» after heat treatment, which are characteristic for cast iron. However, two thermal effects are observed above 400 °C at approximately the same temperatures as for cast iron: exothermic one (peak at 470 °C), corresponding to decomposition of bainite-martensite matrix, and endothermic one (peak 717 °C), corresponding to transformation of ferrite-cementite mixture to austenite.

The decomposition of the martensitic matrix after the low-temperature heat treatment is also observed in the temperature range of 417...489 °C. It follows that in the process of operation of chilled rolls with martensitic structure in the deformation region (local heating of the surface of work rolls is achieved in hot rolling mills at

450...650 °C) one can see the processes associated with martensite decomposition, which may lead to the formation of stress cracks in the surface layer of the roll (Vdovin, Pivovarova, & Zaitseva, 2014).

4. Conclusions

The results of the research work show that:

- boron forms the following carbides in roll-foundry iron: B_4C , $B_{10}C$ and $B_{13}C_2$, which increase the hardenability and the microhardness of the carbide phase thus contributing to the increase in the surface layer of roll-foundry iron;
- borides of $Fe_3(B,C)$ type, the so called "boride-carbide" of iron are of particular interest as only 40 % of carbon atoms in the cementite lattice of Fe_3C can be replaced by boron and the microhardness of this compound is higher than that of conventional cementite;
- boron influences the temperature of decomposition of structural constituents of roll-foundry iron (increasing the rate of cementite decomposition), it decreases the values of temperature maximums of developing phase transformations.

The research group determined the kinetics of decomposition of martensite, which is formed when chilled cast iron is poured into the metal mould and then tempered at 400 °C;

- The authors defined the nature of martensite transformations in the roll face of the chilled alloyed cast iron:
 - without heat treatment it is proceeded in several stages and depends on the heating temperature: in the temperature range of 210...400 °C the main process is decomposition of retained austenite into bainite, in the temperature range of 400...500 °C the main process is decomposition of martensite and formation of ferrite-cementite mixture;
 - after heat treatment (backing) it is proceeded in one stage in the temperature range of 400...500 °C;
 - heat treatment of the analyzed alloyed iron «ЛПХМД-70» should be performed at the temperatures of up to 400 °C because above this temperature the process of martensite decomposition starts, which can eventually cause defects in the roll, namely the formation of cracks in the roll face.

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