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*A study of the effect of holding the cast iron melt at temperatures of 1,300, 1,450 and 1,600 °C for 20, 55 and 90 minutes on the structure and properties of cast iron in a liquid state and after crystallization was carried out. The studies were carried out on samples with a diameter of 30 mm; cast iron containing 3.61–3.75 % carbon, 1.9–2.4 % silicon, 0.03 % manganese, 0.081–0.084 % phosphorus, 0.031–0.039 % sulfur was poured into green-sand molds. The samples were cast from the original cast iron (unmodified), modified with ferrosilicon 75 GOST 1415-93 (FS75), rare-earth metals (REM) and together with the REM+FS75 complex. The structure of cast iron was investigated by optical metallography, electron microscopy and X-ray structural analysis. An increase in the holding temperature and time of the cast iron melt leads to an increase in its hardness. An increase in temperature at a short holding time leads to an increase in strength in the entire investigated temperature range (1,300–1,600 °C). Holding for 90 minutes at a temperature of 1,450 °C corresponds to an extremum, after which, with a further increase in temperature, a sharp drop in strength is observed. The change in the toughness of cast iron is characterized in a similar way*

*Keywords: data mining, heat-time treatment, chemical parameter, ferrosilicon, rare-earth metals, engineering, pyroelectric, transformation, optimization, structure*

# INFLUENCE OF MELT TREATMENT PARAMETERS ON THE CHARACTERISTICS OF MODIFIED CAST IRON IN THE METALLURGICAL INDUSTRY USING INTELLECTUAL ANALYSIS METHODS

**Denis Boldyrev**

Doctor of Technical Sciences, Professor  
Department of Nanotechnologies, Material Science and Mechanics  
Togliatti State University  
Belorusskaya str., 14, Togliatti, Russian Federation, 445020  
E-mail: denis.boldyrev@vaz.ru

**Roman Dema**

PhD, Associate Professor\*  
E-mail: demarr78@mail.ru

**Oleg Latypov**

Postgraduate Student\*  
E-mail: latolegraf@list.ru

**Anton Zhilenkov**

Department of Cyber-Physical Systems  
Saint-Petersburg State Marine Technical University  
Lotsmanskaya str., 3, Saint-Petersburg, Russian Federation, 190121  
E-mail: zhilenkovanton@gmail.com

**Vitalii Emelianov**

Doctor of Technical Sciences  
Department of Business Informatics  
Financial University under the Government of the Russian Federation  
Leningradsky ave., 49, Moscow, Russian Federation, 125993  
E-mail: v.yemelyanov@gmail.com

**Alexey Nedelkin**

PhD  
Department of Informatics  
Plekhanov Russian University of Economics  
Stremyanny lane, 36, Moscow, Russian Federation, 117997  
E-mail: nedelkinanov@gmail.com

\*Department of Metal Forming Machines and Technologies  
Nosov Magnitogorsk State Technical University  
Lenin str., 38, Magnitogorsk, Russian Federation, 455000

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## 1. Introduction

Cast iron is widely used in mechanical engineering despite the constant appearance of new functional and

structural materials. Cast iron molded parts and products are used in agricultural engineering, automobile and machine tool industries, etc. [1]. Therefore, obtaining cast iron castings with the required structure and high

mechanical properties is an important problem in metallurgical production.

One of the main directions in controlling the structure and improving the mechanical properties of cast iron castings is melt processing. The most common methods of influencing the liquid state of cast iron are heat-time treatment (HTT), melt modification and secondary refining [2, 3].

A promising direction of influence on crystallization processes, and through them on the structure and properties of cast iron castings, is heat-time treatment of the alloy in the liquid state before pouring. Thermal treatment means heating the cast iron melt and isothermal holding to obtain a homogeneous melt [4]. The large variability of the influence of holding parameters (temperature and time) with the use of various modifiers on the structure and properties of lamellar iron castings represents a huge field of study.

By using different HTT parameters and modifiers, it is possible to obtain cast irons with different structures: lamellar cast iron, point graphite cast iron, white cast iron (all carbon in cementite), spherical cast iron, and compacted graphite iron (CGI). CGI is a promising construction material. It combines balanced physical and chemical properties with technological and operational characteristics. The peculiarities of the CGI properties are determined by the structure of graphite inclusions. This is in contrast to lamellar gray cast iron in which the graphite plates grow from a single center and are bonded to each other within the eutectic colony. Each particle of vermicular graphite grows from a separate center.

Obtaining CGI with the required mechanical properties is an important task of metallurgical production.

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## 2. Literature review and problem statement

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In [5], the researchers introduced the term “heat-time treatment program”. This term means the following set of measures: analysis of temperature dependences of structurally sensitive properties of molten iron (or steel) and identification of characteristic temperatures for a given alloy and analysis of the effect of the duration of melt holding at different temperatures. In [2, 6], the research and implementation of technology at the enterprises of the Kuznetsk coal basin are described. The work [7] investigates the increase in the strength properties of cast iron in molds using HTT in conjunction with the technology of out-of-furnace treatment of the melt with neutral gas by the method of resonant-pulsating refining by tuyeres with gas-dynamic pulsators. However, it should be noted that the use of this method entails an increase in the already prohibitively high noise load on workers in industrial conditions. In [8], the influence of various parameters of casting and processing in a ladle on the physical and chemical parameters of cast iron is investigated, including the temperature of liquid iron holding (from 1,420 °C to 1,540 °C) for 120 minutes on IGQ (cast iron quality index) and nucleation rate. The results showed a decrease in IGQ and nucleation rate with increasing holding temperature. In the above papers [2, 6–8], the influence of the modifiers used during HTT on the properties of iron castings is not investigated. In [9], the influence of SiC and FeSi on the characteristics of gray cast iron is investigated. The use of SiC resulted in an increased amount of A-type graphite with a more uniform distribution and increased

fluidity and hardness than the addition of FeSi. In [10], the results of a study of the use of 60 % FeSi75+40 % REM and 20 % FeSi75+80 % Sr composite modifiers are presented, which showed that the castings modified with REM have high values of strength, hardness and quality factor. Castings treated with the modifier with Sr have the smallest variation in mechanical properties in their cross-section. As you can see, in [9, 10], on the contrary, the results of studies of the influence of modifiers on the properties of gray iron castings without varying the HTT parameters are given. Thus, there is a need to study the influence of the holding parameters of liquid cast iron on the properties of castings.

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## 3. The aim and objectives of the study

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The aim of the study is to obtain the dependences of the ultimate strength, impact toughness, Brinell hardness, lattice period of ferrite ( $a$ , Å) and intensity of the cementite line ( $J$ , imp/sec) on the time and temperature of cast iron holding using FS75 and REM modifiers for subsequent application in real production conditions.

To achieve the aim, it is necessary to solve the following objectives:

- to develop a composite second-order orthogonal plan, including the necessary factors (parameters) affecting the structure and mechanical properties of cast iron during HTT, carry out the necessary experiments according to the given plan;
- to process the results of experiments and obtain the dependencies indicated and conduct electron microscopy and X-ray spectroscopy of the samples.

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## 4. Materials and methods of research

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The studies were carried out on specimens with a diameter of 30 mm made of cast iron of the following composition: carbon – 3.61–3.75 %, silicon – 1.9–2.4 %, manganese – 0.03 %, phosphorus – 0.081–0.084 %, sulfur – 0.031–0.039 %. The melt was poured into wet sandy molds. Before pouring, molten iron was held in a smelting furnace at temperatures of 1,300, 1,450 and 1,600 °C for 20, 55 and 90 minutes. The samples were cast from the original cast iron (unmodified), modified with FS75, REM and together with the REM+FS75 complex. A Tamman vacuum furnace was used. The maximum working space temperature was 2,100 °C.

The study of the mechanical properties and structure of cast iron depending on melt holding time ( $X_1$ ) and temperature ( $X_2$ ) was carried out using a symmetric composite orthogonal plan. Ultimate strength (limit), impact strength ( $CS$ ), Brinell hardness ( $HB$ ), ferrite lattice period ( $a$ , Å) and cementite line intensity ( $J$ , imp/sec) were chosen as responses.

Electron microscopy and X-ray spectroscopy were performed on a Carl Zeiss Sigma scanning electron microscope (Germany) (resolution of the electron column at an optimal WD of 1.3 nm; range of movement:  $X$  – 125 mm;  $Y$  – 125 mm;  $Z$  – 50 mm; tilt 0–90°; 360° rotation). The microscope is equipped with an EDAX analytical system (USA) with an Apollo detector and a Hikari back-scattered electron detector. This equipment was used to determine the intensity of the cementite line.

The tensile strength was determined according to GOST 1497-84 on a UMM-10 tensile testing machine model.

Impact strength was determined using a Galdabini Impact 300 pendulum driver on a sample with a U-type concentrator at room temperature. The maximum impact energy of the pendulum is 300 J, the depth of the concentrator is 2 mm in accordance with GOST 9454-78. Brinell hardness was determined using a TB 5006 device in accordance with GOST 9012-59. The shape of graphite in the cast iron structure was determined according to GOST 3443. The ferrite lattice period was determined using a PEM-100-01 transmission electron microscope and a standard expression for determining the cubic lattice period, which is calculated by the formula:

$$a_i = \frac{c}{r_i} \sqrt{H_i^2 + K_i^2 + L_i^2}, \tag{1}$$

$C$  – electronograph constant;  $r_i$  – radius of the  $i$ -th ring;  $H_i^2 + K_i^2 + L_i^2$  – sum of the squares of the interference indices corresponding to the  $i$ -th ring.

## 5. Results of the research

### 5.1. Experimental design and results

The design matrix with  $N=9$  experiments is presented in Tables 1, 2.

Table 1

Symmetric composite second-order orthogonal design

No.	$X_0$	$X_1$	$X_2$	$X_1X_2$	$\sigma_B$ , MPa		KC, J/m <sup>2</sup>	
					Original	FS75	Original	FS75
1	+1	+1	+1	+1	258	233	5.1	4.5
2	+1	-1	+1	-1	188	191	5.4	4.8
3	+1	+1	-1	-1	173	174	4.8	4.8
4	+1	-1	-1	+1	166	133	4.8	4.1
5	+1	+1	0	0	309	361	7.0	6.7
6	+1	-1	0	0	165	122	4.8	3.8
7	+1	0	+1	0	219	272	5.7	4.8
8	+1	0	-1	0	188	112	5.1	4.8
9	+1	0	0	0	231	231	6.0	6.0

Table 2

Symmetric design of structural components

No.	HB			$a$ , Å		$J$ , imp/sec (Fe <sub>3</sub> C)		
	Original	FS75	REM	Original	FS75	Original	FS75	REM
1	250	250	460	2.8573	2.8592	532	473	1426
2	190	200	220	2.8592	2.8610	400	363	722
3	175	165	215	2.8598	2.8621	425	442	495
4	160	160	185	2.8600	2.8623	319	246	310
5	215	230	390	2.8596	2.8611	455	420	528
6	170	160	150	2.8599	2.8618	364	308	360
7	225	245	255	2.8585	2.8602	475	440	1254
8	160	165	215	2.8602	2.8624	378	297	432
9	200	195	270	2.8598	2.8619	405	390	451

### 5.2. Dependences of changes in the mechanical properties of cast iron on HTT parameters

Based on the results of all nine experiments of the design matrix, using the well-known formulas [11] (these formulas were also used in [12–17]) and using the constants, all regression coefficients, their variances and root-mean-square errors were calculated, and then the confidence intervals for each group of coefficients at a significance level of  $\alpha=0.05$ .

Comparison of the absolute values of the calculated regression coefficients with their confidence intervals made it possible to select statistically significant values of the coefficients and discard the regression coefficients with unconfirmed significance for the accepted confidence probability. The regression equations obtained in coded variables are shown in Table 3. In accordance with the conditions of this experiment, the coded ( $X_i$ ) and natural ( $X_j$ ) values of the factors are related by the ratios:

$$X_i = (X_j - X_{j0}) / \mathbf{DX}_j; X_j = X_{j0} + X_i \mathbf{DX}_j, \tag{2}$$

where  $\mathbf{DX}_j$  - variation interval.

Fig. 1–3 visually represent some of the obtained dependencies presented in Table 3.

Table 3

Regression equations in coded variables

Cast iron properties	Modification	$a_0$	$a_1$	$a_2$	$a_3$	$a_4$	$a_5$
$\sigma_B$ , MPa	Original	237.1	36.83	23	15.17	–	–39.5
	FS75	240.03	53.66	46.2	–	–	–55.2
KC, kJ/m <sup>2</sup>	Original	6.27	0.33	0.25	–	–0.42	–0.87
	FS 75	5.91	0.55	–	–0.25	–0.49	–0.99
HB	Original	199.99	20.0	28.334	11.25	–4.575	–4.58
	FS 75	203.63	20.83	34.167	11.25	–10.45	–
	REM+FS 75	264.75	85.0	53.3	52.5	19.4	–15.6
$a$ , Å	Original	2.8883	–0.0004	–0.0008	0.00044	–	–
	FS 75	2.8902	–0.0005	–0.0011	0.0004	–	–
	REM+FS 75	2.8897	–0.0007	–0.0013	0.00026	–	–
$J$ , imp/sec (Fe <sub>3</sub> C)	Original	418.57	54.83	47.5	–	–9.755	7.24
	FS 75	391.3	53.0	65.17	–	–22.465	–18.0
	REM+FS 75	507.73	176.17	360.84	129.75	–82.13	316.9

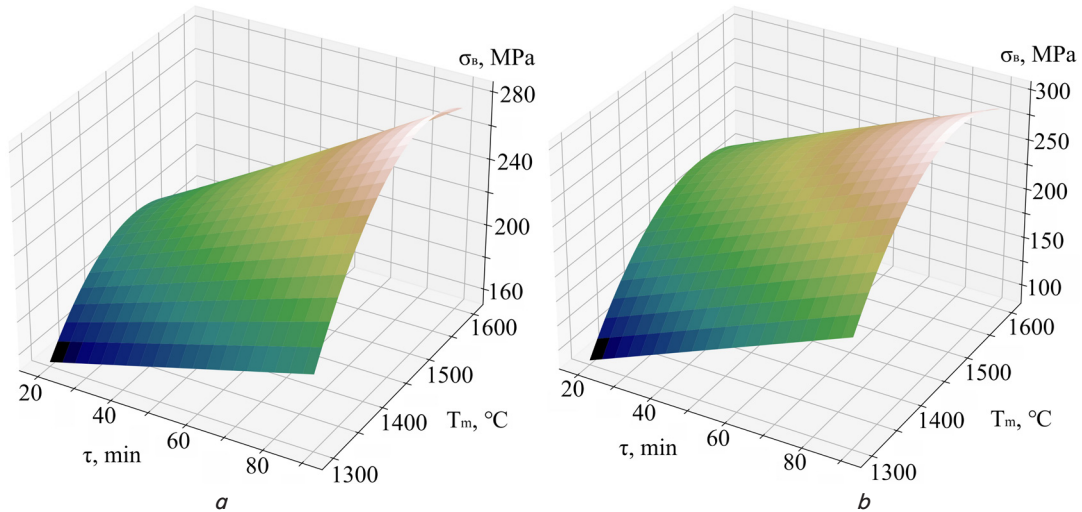


Fig. 1. Dependence of ultimate strength ( $\sigma_B$ ) on holding time ( $\tau_m$ ) and temperature ( $T_m$ ) of cast iron melt:  
 a – unmodified:  $\sigma_{Bmax}=283$  MPa at 90 min, 1,520 °C,  $\sigma_{Bmin}=153$  MPa at 20 min, 1,300 °C;  
 b – modified FS75:  $\sigma_{Bmax}=303$  MPa at 90 min, 1515 °C,  $\sigma_{Bmin}=85$  MPa at  $\tau_B=20$  min, 1,300 °C

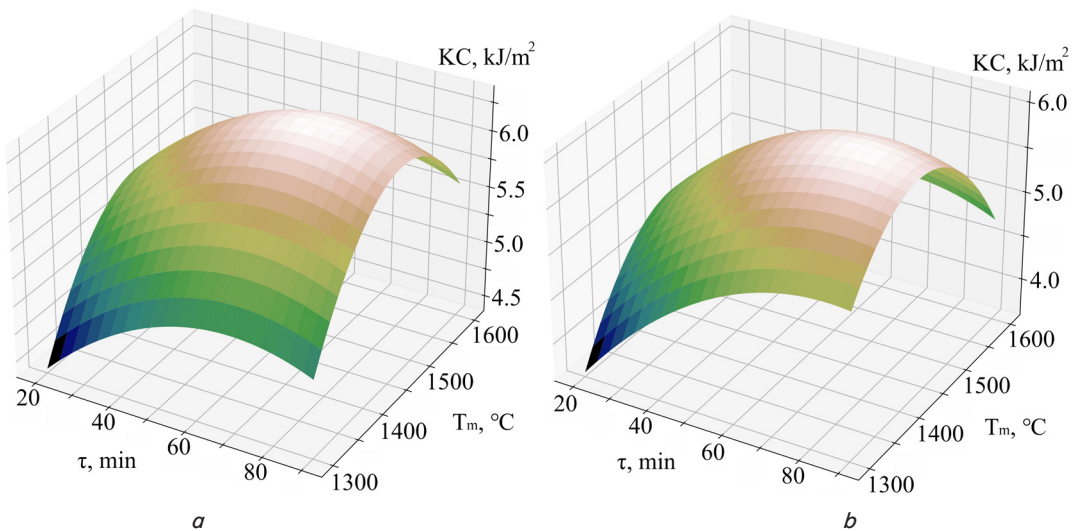


Fig. 2. Dependence of toughness ( $KC$ ) on holding time ( $\tau_m$ ) and temperature ( $T_m$ ) of cast iron melt:  
 a – unmodified:  $KC_{max}=6.35$  KJ/m<sup>2</sup> at 69 min, 1,470 °C,  $KC_{min}=4.40$  KJ/m<sup>2</sup> at 20 min, 1,300 °C;  
 b – modified FS75:  $KC_{max}=6.07$  KJ/m<sup>2</sup> at 75 min, 1,440 °C,  $KC_{min}=3.63$  KJ/m<sup>2</sup> at 20 min, 1,300 °C

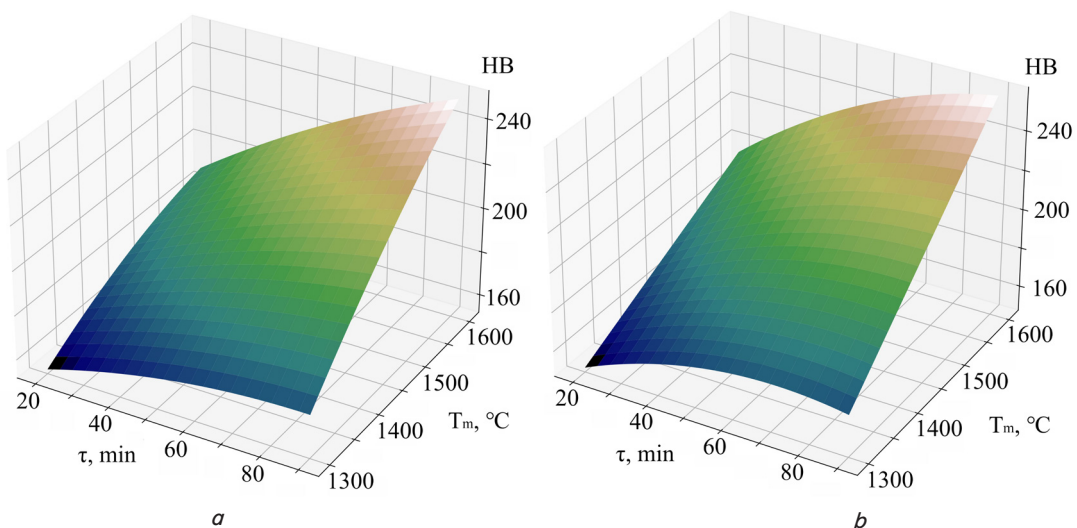


Fig. 3. Dependence of Brinell hardness ( $HB$ ) on holding time ( $\tau_m$ ) and temperature ( $T_m$ ) of cast iron melt:  
 a – unmodified:  $HB_{max}=250$  at 90 min, 1,600 °C,  $HB_{min}=154$  at 20 min, 1,300 °C;  
 b – modified FS75:  $HB_{max}=259$  at 90 min, 1,600 °C,  $HB_{min}=149$  at 20 min, 1,300 °C



Visual models of graphics allow you to visualize all technological transformations. The structural bases for the formation of technological and chemical processes are formed in the temperature-time order of indicators.

## 6. Discussion of the results of the study of mechanical properties and structural transformations in the samples obtained after HTT

Analysis of the results obtained (dependences, results of electron microscopy and X-ray spectroscopy) showed the following structural transformations and changes in mechanical properties during HTT.

An increase in the temperature of overheating of liquid iron from 1,300 to 1,600 °C leads to an increase in the size of austenite grains and a decrease in the length of the graphite plates. With an increase in the holding time of the cast iron melt, the sizes of austenite grains increase less significantly than with an increase in temperature, while prolongation of holding time at 1,600 °C makes it possible to grind graphite inclusions from lamellar to pointlike. Modification of the FS75 cast iron melt at temperatures of 1,300 and 1,450 °C followed by holding for 20, 55 and 90 minutes did not have a significant effect on the cast iron structure. At 1,600 °C, the nature of inclusions of phosphide eutectic changed from FE3 to FE5 GOST 3443-87. Modification with the complex (REM+FS75) at 1,300 °C led to an increase in the compactness of graphite at a short holding time. At a temperature of 1,450 °C and short holding time, the ferritization of the metal base of cast iron increases, with an increase in holding time, the amount of ledeburite in the structure increases with the formation of compact and vermicular graphite. At a temperature of 1,600 °C, the precipitation of point graphite is observed. At a short holding time, the ferritization of the matrix increases near the inclusions of point graphite. The precipitation of nonmetallic inclusions of the type of REM sulfides is observed with a long holding time. The results are consistent with the research [18].

The change in the mechanical properties of cast iron is in accordance with the change in its structure (Fig. 1–4). An increase in the holding temperature and time of the cast iron melt leads to an increase in its hardness (Fig. 3). An increase in temperature at a short holding time leads to an increase in strength in the entire investigated temperature range (1,300–1,600 °C). Holding for 90 minutes at a temperature of 1,450 °C corresponds to an extremum, after which, with a further increase in temperature, a sharp drop in strength is observed (Fig. 1). The change in the toughness of cast iron is characterized in a similar way (Fig. 2).

Modification of cast iron with REM at a temperature of 1,300 °C did not significantly affect its structure, except for the undissolved inclusions of the master alloy. Vermicular graphite is formed, the amount of ledeburite increases, and a bainite component appears in the central zones of dendrites with an increase in the holding time at a temperature of 1,450 °C. Vermicular graphite is formed (at 20 and 55 minutes of holding) when overheated to 1,600 °C, chill is enhanced up to continuous at 90 minutes of holding. A bainitic component is observed in the center of the dendrites, pearlite is observed at the periphery, cracks and inclusions of undissolved master alloy are also found. An increase in the superheat temperature of the melt and the holding time leads

to the formation of needed martensite forms both during the modification with REM and with the REM+FS75 complex. An increase in the holding temperature and time of the melt leads to the formation of quenching phases and structural components in the structure of the crystallized metal. In unmodified cast iron and cast iron modified with FS75, the number and size of cementite inclusions in the composition of phosphide eutectic increase during structure formation. In cast irons modified with REM additives, the amount of ledeburite is formed and increases, and at temperatures of 1,450, 1,600 °C and holding time of 55 and 90 minutes, bainite is formed in the center of primary austenite grains. The results are consistent with the research [19–23].

The obtained results of structural transformations can be explained by the following physicochemical processes occurring in the cast iron melt during HTT. On the liquidus line, in the area of cast irons, carbon atoms are in a weakly valent state:  $Cc^{0.17-}$  for a concentration of 3.28 %,  $Cc^{0.32-}$  for a concentration of 4.34 % carbon,  $Cc^{0.42-}$  for a concentration of 5.03 %, in the state  $Cc^{0.51-}$  for a concentration of 5.71 % and  $Cc^{0.64-}$  for a concentration of 6.67 % carbon. Iron atoms on the liquidus line are in the ionized state  $Fe^{1+}$ ,  $Fe^{2+}$ ,  $Fe^{4+}$ ,  $Fe^{6+}$ ,  $Fe^{8+}$  and  $Fe^{10+}$  for carbon concentrations of 2.78; 3.28; 4.34; 5.03; 5.71 and 6.67 %. Carbon atoms are in the melt in a covalent state  $Cc^{1.5-}$  for a carbon concentration in cast iron of 3.61 % at a temperature of 1,300 °C, since this temperature is 106 °C below the liquidus line (1,406 °C) and carbon atoms from iron form covalent bonds. Carbon atoms fill the  $2p^2$  level to  $2p^6$ , attaching 4 electrons according to the  $C^0 \rightarrow Cc^{1-} \rightarrow Cc^{1.5-} \rightarrow Cc^{2-} \rightarrow Cc^{3-} \rightarrow Cc^{4-}$  scheme, passing from the metal state  $-C^0$  (radius 0.55 Å) to the covalent state  $Cc^{4-}$ , with a radius of 0.77 Å. The covalent carbon atom  $Cc^{4-}$  goes over into the ionic atom  $Ci^{4-}$ , one of the bonds of which remains covalent [17]. Graphite is formed on the basis of the ionic carbon atom. With an increase in the melt temperature, the transition of carbon atoms occurs from the covalent state  $Cc^{1.5-}$  (0.64 Å) at a temperature of 1,300 °C to a weakly ionized state  $C^{0.03+}$  (0.543 Å) at a temperature of 1,450 °C and into an ionized state  $C^{0.3+}$  (0.473 Å) at a temperature of 1,600 °C, which is higher than the liquidus line by 44 and 194 °C, respectively. Iron atoms are also in the melt in the ionized state  $Fe^{1+}$ ,  $Fe^{2+}$ , which facilitates their compound with carbon atoms in Fe-C microgroups, while the interatomic bonds of the same atoms [C-C] and [Fe-Fe] are weakened. When the holding time of cast iron is 20 minutes, the stimulating ferrite formation and the ionized state of carbon atoms are preserved. With an increase in the holding time of the melt to 55 and 90 minutes at a temperature of 1,450 °C, the mechanism of crystallization of hypoeutectic cast iron shifts towards the mechanism of crystallization of eutectic and hypereutectic cast iron with the transition of carbon atoms from the ionized to the covalent state:  $C^{0.03+} \rightarrow C^0 \rightarrow Cc^{0.17-} \rightarrow Cc^{0.32-} \rightarrow Cc^{0.42-}$ , the ionization of iron atoms in this case increases to the level of  $Fe^{3+}$ ,  $Fe^{4+}$ ,  $Fe^{5+}$ ,  $Fe^{6+}$ . As a result, ledeburite is released in the structure and a bainite component is formed in the center of dendritic grains. At a holding temperature of 1,600 °C and time of 55 and 90 minutes, the transition of carbon atoms from the ionized to the covalent state also occurs:  $C^{0.3+} \rightarrow C^0 \rightarrow Cc^{0.32-} \rightarrow Cc^{0.42-} \rightarrow Cc^{0.51-} \rightarrow Cc^{0.64-} \rightarrow Cc^{1-}$ , the degree of ionization of iron atoms increases even more to the level of  $Fe^{8+}$ ,  $Fe^{9+}$ ,  $Fe^{10+}$ , as a result, the tendency to chill and after 90 minutes of holding time, white cast iron crystallizes. The strengthening of Fe-C bonds is also

facilitated by modification of cast iron with REM, which leads to an increase in the tendency of cast iron to chill and the formation of bainitic structures during crystallization.

The results of the study turned out to be quite extensive. The main difference is the study of the influence of a wide range of factor values (time and temperature and their range) and the influence of modifiers (FS75 and REM modifiers) on the mechanical properties of cast irons. Based on the obtained dependencies, it is possible to determine the optimal HTT parameters for obtaining cast iron castings with the required structures and mechanical properties. In contrast to the well-known works, the research results cover a wide range of factors and varieties obtained by cast iron, but it is worthwhile to understand that it is possible (and most likely) due to such a range of missed many extreme and transition points in terms of the experiments of this study. Therefore, more extensive research is needed to find them. It is also necessary to continue research in the direction of using more varieties of modifiers and varying their volumes.

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## 7. Conclusions

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1. The symmetric composite second-order orthogonal plan made it possible to obtain the results necessary at this stage of the study in the minimum possible number of experiments.

2. Visual models of graphics made it possible to show more clearly all technological transformations of structures and components. The structural bases for the formation of technological and chemical processes formed the criteria for converting the temperature-time order of indicators of specified ranges. As a result of modeling and elaboration of practical aspects of the research, practical results were obtained.

– the optimal conditions for the formation of vermicular graphite is the modification of cast iron with the REM+FS75 complex at a melt holding temperature of 1,450 °C and time of 20 minutes;

– an increase in the holding temperature and time of the cast iron melt modified with REM additives leads to an increase in ledeburite in the structure;

– overheating of the metal modified with REM additives above 1,450 °C promotes the appearance of martensite and acicular cementite in the central zones of dendrites;

– non-metallic inclusions of complex sulfides of rare-earth metals appear in the cast iron melt after prolonged holding time at a temperature of 1,600 °C and treatment with REM-containing modifiers. Overheating of the metal up to 1,600 °C promotes crack formation.

Analysis of the data obtained showed that with an increase in the temperature of overheating of the melt and/or an increase in the holding time during overheating, the relative content of cementite in the cast iron structure increases. REM modification to the greatest extent increases the content of free cementite in the cast iron structure, especially at a temperature of 1,600 °C. Modification of cast iron melt with the REM+FS75 complex allows reducing surface chill and the content of free cementite in the cast iron structure with the stable formation of vermicular graphite (or compacted graphite iron (CGI)).

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