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# Modification of Grey Cast Iron and Steel by Efficient Compositions Based on Nanodispersed Powders of Tungsten and Titanium Carbides

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## Abstract

Complex comparative analysis of some modifiers based on refractory ceramic particles was carried out. Compositions based on nanodispersed tungsten and titanium carbides were obtained by a combined method (self-propagating high-temperature synthesis in combination with mechanochemical treatment). In experiments on intra-mould modification of grey cast iron and 110G13L steel, they demonstrated the maximum efficiency compared to other studied modifiers: within ceramic phase concentrations of 0.013–0.2 %, for gray cast iron, an increase in tensile strength was to 20 %, in hardness – to 13 %, in wear resistance – to 69 %, in corrosion resistance – 2.6 times; for steel, an increase in temporary tear resistance reached 18.0 %, in relative elongation – to 40 %, grain size reduction – by 6.8 times. As demonstrated during modification of grey cast iron by additives containing nanodispersed ceramic phases, not only the concentrations and composition of modifiers but also various process parameters (the design, the volume of the mold, the method of introduction of modifiers, etc.) should be taken into account.

**Keywords:** intra-mould modification, titanium and tungsten carbides, self-propagating high-temperature synthesis (SHS), Hadfield steel (110G13L), grey cast iron

## INTRODUCTION

Iron-based alloys (cast irons and steels) are efficient construction materials that ensure mechanical and process properties alongside with a relatively low cost. The improvement of operating characteristics (an increase in breaking strength, hardness, wear resistance, corrosion resistance, and finally the operating life) of cast irons and steels is of relevance. The number of castings depends on a series of process parameters that affect melt crystallization processes (a feeling temperature, a moulding compound, chemical composition, the volume of casting, overheating of metals during melting, etc.)

[1]. In turn, the listed processing factors determine the composition and the number of phases, grain size, and various defects of a crystal lattice [2].

The number of castings according to the technology of melting and filling of the metal into moulds may be increased if we learn how to control a crystallization process. Defect-free iron crystals with a tensile strength of more than 1000 kgf/mm<sup>2</sup> (strength of carbon steel is 40 kgf/mm<sup>2</sup>) have been grown in a lab [3]. Attempts to improve mechanical properties by creating of the single crystal have failed, therefore one needs to go the opposite way, in other words, affect the crystallization process to obtain many small crystals (grains).

Currently, there is a growing interest in the use of nanomaterials in metallurgy to improve the quality of products. The introduction of small additions of substances (modifiers) that chemically do not interact with matrix allows affecting crystallization processes of cast iron and steel (changing metal grain size, varying the shape, size, and distribution of graphite inclusions) [4–11].

It has been found experimentally that the more nucleation centres are in the unit volume of the melt, the more crystals are generated, the smaller they are and the higher mechanical properties of the metal are.

Strength characteristics versus the grain size are well described by the Hall–Petch law [12, 13], according to which material hardness enhances in 3–5 times during decreasing the average grain size, and its plasticity during decreasing – in more than 10 times.

According to [14], particles that are present in metal volume affect dislocation dynamics preventing their movement. This material may be regarded as a dispersion-strengthened composite. Dislocation sources form dislocation loops surrounding the particles under the action of the applied voltage. The number of dislocations loops ( $n$ ) depends on the distance between the particles:

$$n = l_p \sigma / G_m |\vec{b}| \quad (1)$$

where  $l_p$  is the distance between the particles;  $\sigma$  is applied voltage;  $G_m$  – shear modulus of a matrix material;  $|\vec{b}|$  is the Burgers vector of a dislocation.

The shear stress acting on a particle is defined as

$$\tau = l_p \sigma^2 / G_m |\vec{b}| \quad (2)$$

while the yield strength of the material reinforced by the particles is determined as

$$\sigma_T = (G_p G_m |\vec{b}| / l_p C)^{0.5} \quad (3)$$

where  $G_p$  is the shear modulus of particle material;  $C$  is a constant characterizing the type of material.

The shear modulus of particle material ( $G_p$ ) should exceed the shear modulus of matrix material ( $G_m$ ), otherwise hardening would not happen. If the particle is not wetted by the material, there would be a bubble with a shear modulus equal to zero between the material and the particle. Therefore, according to formula

(3), yield strength is zero and, therefore, the material is weakened. In order for particles of another phase could change material properties, they have to wet this material. Otherwise, they act as structural defects that deteriorate material properties.

Mechanochemistry may substantially facilitate the way of reaching a positive outcome. Treatment in planetary high-energy activators allows not only grinding the material but also activating powder particles, while the additional cladding of particles ensures modifiers with good wettability by melts.

Despite the abundance of market supply, the issue of obtaining modifiers based on refractory ceramic particles wetted by a melt and evenly distributed in a metal matrix, as well as receipt problem of stable results during modification are still relevant.

The goal of the present work is a complex comparative analysis of some modifiers based on refractory ceramic particles and metal protectors for out-of-furnace treatment of cast irons and steels and assessment of their efficiency.

## EXPERIMENTAL

Assessment of the efficiency of modifiers was carried out under the following conditions.

Modification objects are SCh15 – SCh30 grey cast irons and 110G13L steel.

Melting of gray cast iron was carried out in induction furnaces with the following characteristics: crucible capacity of 400–750 kg, acid lining (quartz and 3 % boric acid). Pouring of cast iron into moulds was carried out at a temperature of 1350–1380 °C.

Cast iron mould is a system of a riser in the middle and six cylinders with a length of 300 mm and a diameter of 30–40 mm around it. Metal is poured into the central part (a riser in the middle of the semi-finished product), from where it enters by bottom casting (bottom-up) to cylindrical blank parts that are further used to perform various tests. To avoid floating-up of a modifier, it was placed between ceramic filters installed in mould riser, prior to pouring [15].

Dry sand-clay mixture (SCM) and cold-hardening soluble-glass mixture (CHM) of quartz sand were used as materials to prepare moulds.

Cast iron modification was carried out in a ladle or a mould (metal mass in the mould of 19–40 kg).

To ten samples (nine probes with different modifiers wrapped in aluminium foil, one without a modifier; empty aluminium foil was placed on the lower filter) were simultaneously poured during cast iron modification in a mould of one ladle.

When modifying cast iron in a ladle, the amount of metal required to fill one or two moulds was used.

To study the effect of modifier concentration on the corrosion resistance of cast iron, only mould riser (a mould in the form of a beaker) was used.

The mean value of tensile strength was determined according to six values, which ensured the correctness of the findings.

To characterize the hardness of materials, the Brinell method was used. Measurements were carried out in different sample points (no less than five prints) and they were compared to the values obtained during examining reference sample.

Corrosion resistance was studied according to GOST 9.908–85, for two prepared cast iron samples (modified and reference). To measure the relative corrosion resistance, the samples should have almost identical diameters and heights, in other words, the surface that contacts with an aggressive medium should be alike. The tests were performed in 25 % HCl (the samples were aged for 24 h at room temperature).

Relative wear resistance (in relation to reference sample) was determined according to GOST 17367–71 during the test for abrasive wear during the friction of the fixed abrasive particles.

Melting of 110G13L steel was carried out in a DC6-H1 furnace according to remelting technology of doped waste. Metal mass is 5.5 t. Steel deoxidation in the output was carried out by filing to the bottom of the bottling bucket of 8 kg of a special deoxidizer (70 % Al, 20 % Fe, and 5 % Ca, the rest is C, Si, and Cu) with the addition of 5 kg of granular aluminum. The temperature of the steel in the output (in the bucket) was 1460 °C.

Steel modification was carried out in a mould (metal mass is 125 kg); in all test moulds, two tidal samples of hatchet type were envisaged and filled to determine mechanical properties and microstructure.

For steel samples, a change in temporary

tear resistance ( $\sigma_b$ , %), a change in relative elongation ( $\delta$ , %) were determined; grain size assessment was carried out according to GOST 5639.

To process grey cast iron and steel, modifiers based on refractory ceramic particles obtained *via* different methods were used.

Complex modifiers were obtained in the iron matrix ( $\alpha$ -Fe,  $\text{TiC}_x\text{N}_y$ , SiC) *via* plasmochemical method (PC) from natural raw materials, *i. e.* by its interaction with methane in hydrogen-nitrogen plasma flow. Powders were obtained in Baykov Metallurgy and Materials Institute RAS. [16].

Reaction products represented powders with a specific surface area of about 1.6–20 m<sup>2</sup>/g. According to electron microscopy data, they comprised of particles with a size of 30–70 nm.

According to X-ray phase analysis results, the powders are compositional. Ferrite, austenite, and cementite phases prevail in their composition. The presence of iron oxide in the form of magnetite was noted in a small amount. In diffraction patterns, there are lines that correspond to titanium compounds ( $\text{Ti}_x\text{C}_y\text{N}_z$ ), and also to silicon carbide (SiC).

By the method of mechanochemical activation (MA), modifiers were obtained by processing various ceramic powders with metals in a planetary centrifugal activator.

Modifiers based on a mixture of tungsten and titanium carbides [WC ( $\text{W}_2\text{C}$ ) and TiC] were used for comparison. Nanodispersed powders of tungsten (in terms of WC) and titanium carbides (WC/TiC = (40–80) : (60–20) %) were obtained by self-propagating high-temperature synthesis (SHS) combined with MA according to the technique described in [17].

Hereafter, the resulting mixture of tungsten and titanium carbides was exposed to MA in the planetary centrifugal activator for 40–60 s with one or several metal protectors (iron, cobalt, copper, nickel, chromium, and zirconium).

Selection of WC ( $\text{W}_2\text{C}$ ) phases in the amount from 40 до 80 % (in terms of WC) and from 60 to 20 % of TiC, respectively, in the composition of a ceramic mixture is driven by synthesis conditions and is related to the presence of the required phases in reaction products [17]. Firstly, a composition of WC/TiC = 80 : 20 contains the minimum amount of titanium, at which SHS is probable. Secondly, in addition to the reaction products, only

TABLE 1

Composition, number of modifiers and properties of SCh20 cast iron samples (metal mass in a mould No. 1 of 20 kg)

Samples	Modifiers		Cast iron properties						
	Composition	Preparation technique ния	Composition of ceramic phase	$S_{sp}$ , $m^2/g$	Ceramic phase amount, %	Brinell hardness, HB	Hardness increase, %	Tensile strength at break, MPa	Increase of tensile strength, %
1	Reference	—	—	—	0	183.0	0	234.2	0
2	Y <sub>2</sub> O <sub>3</sub> +Cr	MA	Y <sub>2</sub> O <sub>3</sub>	2.7	0.0130	201.0	9.8	260.4	12.2
3	(WC,TiC)+Zr	SHS+MA	WC, TiC	5.5	0.0375	207.0	13.1	280.5	19.8
4	$\alpha$ -Fe, TiC <sub>x</sub> N <sub>y</sub> , PC SiC (Fe = 95 %)	—	TiC <sub>x</sub> N <sub>y</sub> , SiC	20.2	0.0015	201.0	9.8	262.0	11.9

unreacted tungsten in minor amounts may be present in a composition of WC/TiC = 40 : 60. Thirdly, there may be initial components that might lead to structure defects of crystallized metals and worsen properties when WC concentration is less than 40 % (TiC is more than 60 %, respectively).

This work mainly used compositions (in %) of WC/TiC = (70–75) : (30–25) (the size of coherent scattering region (CSR) in the order of 30 nm).

The specific surface area of powder of modifiers obtained by mechanochemical and combined (SHS + MA) methods varied within of 3–12 m<sup>2</sup>/g.

Selection of metal protectors is driven by preliminary experiments on modification and SHS conditions. For example, the synthesis of tungsten and titanium oxides is probable in the presence of cobalt and nickel in the reaction mixture.

The concentration of the cladding metal in

modifier composition at a level of 50–75 % ensures adequate mixing of carbide particles and the metal in the activator and an opportunity to use different methods of powder compacting.

Modifiers were used as powders or tablets. Powder compacting was performed using organic binders (methods of modifier compacting are described in [15]).

## RESULTS AND DISCUSSION

### Modification of gray cast iron and 110G13L steel

The effect of the concentration of modifiers on operating characteristics of cast irons and steels was explored in additional experiments [18]. Moreover, it was established that compacted

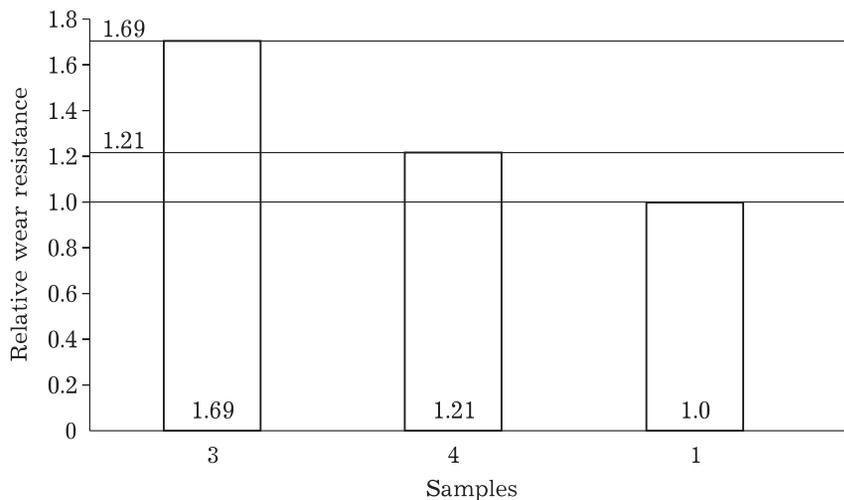


Fig. 1. Wear resistance of modified samples (3 and 4) in relation to reference sample 1.

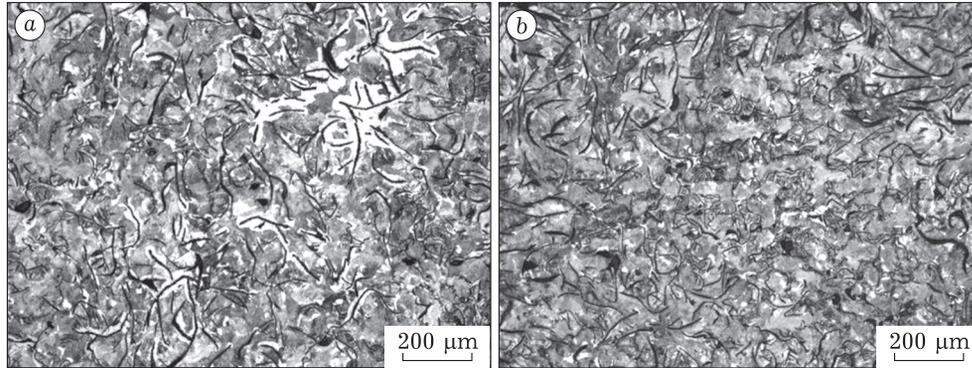


Fig. 2. Structure of grey cast iron samples, etching with 5 %  $\text{HNO}_3$  in alcohol: a – reference, b – sample 3.

modifiers (as tablets) [15] were more suitable in use. Importantly, modifier tablet strength should not too be high ( $\leq 5$  MPa). This is reached at the expense of organic binders based on methyl- and carboxymethylcellulose.

Comparison of the efficiency of modifiers produced by various methods, such as PC, MA, and SHS + MA) was carried out during intra-mould modification of grey cast iron [16, 18] and 110G13L high-manganese steel [19].

Table 1 indicates the data on the composition, preparation technique, and modifier concentration, and also properties of grey cast iron samples (Brinell hardness, tensile strength, a change in hardness and strength in relation to

reference sample). It can be seen that there are the maximum increase of hardness (by 13.1 %) and tensile strength (by 19.8 %) for cast iron modified with tungsten and titanium carbides with zirconium (combined (SHS + MA) preparation method) with a concentration of the ceramic phase of the modifier of 0.038 % (sample 3). The hardness for samples 2 and 4 with modifiers obtained by MA and PC methods increased by 9.8 %, while tensile strength – within 12 %.

Figure 1 gives the data on relative wear resistance of cast iron samples. The non-modified sample, wear resistance of which was accepted equal to one, was used as a reference. The relative wear resistance of samples 3 and 4 is by 69

TABLE 2

Composition and number of modifiers and properties of samples of 110G13L steel

Samples	Modifiers		Properties of steel samples			Distribution of carbides
	Composition	Content of ceramic phase, %	Tensile strength $\sigma_b$ , N/mm <sup>2</sup>	Tensile strength increase, %	Relative elongation, $\delta$ , %	
1	Copper	0	550		10	Small carbides inside austenite grains, rarely along borderlines
2	Boron carbide/copper (1 : 2)	0.056	630	14.5	8	Separate rare precipitates of carbides inside and along borderlines of austenite grains
3	Mixture of tungsten and titanium carbides/copper (2 : 5)	0.033	650	18.0	14	Small carbides inside austenite grains
4	Titanium carbide/copper (1 : 1)	0.083	600	9.0	10	Small carbides inside and along austenite grains



and 21 % higher, respectively, compared to that of the reference sample.

The corrosion resistance of sample 4 in hydrochloric acid with a concentration of refractory particles of 0.0015 % increased by about 12.3 %, and sample 3 at a concentration of refractory particles of 0.0375 % – by 28 %.

Plate-shaped graphite is present in the structure of the studied samples of modified cast iron (Fig. 2). The matrix is comprised of perlite (a volume fraction of 70 %) and ferrite (30 %). The latter is located around graphite inclusions. There is a change in the ratio of perlite to ferrite only for sample 3; a volume ratio of ferrite is only 8 % (see Fig 2, b).

To modify 110G13L steel, compositions based on boron carbide (Table 2, sample 2), a mixture of tungsten and titanium carbides (sample 3), and titanium carbide (sample 4) were used. Modifiers for the improvement of wettability with metal melt were preliminarily treated in a planetary centrifugal mill for 1 min by copper powder. Copper powder (sample 1) was also added to the reference sample.

Etching of samples to study microstructure was carried out with a 4 % alcohol solution of HNO<sub>3</sub>. Numeration of steel samples (1–4) corresponds to that of modifier specimen (1–4).

Table 2 gives the composition, the number of modifiers, and the results of mechanical tests of steels treated with these modifiers.

The use of dispersed modifiers containing boron carbide and a mixture of tungsten and titanium carbides during intra-mould modification of 110G13L steel contributes to the uniform distribution of carbides isolated inside and along the borderlines of grains to a significant decrease in grain size and to an increase in tensile strength compared to other modifiers.

Steels 2–4 demonstrated an increase in tensile strength by 14.5, 18.0, and 9.0 %, respectively. Furthermore, the use of modifier 3 (a mixture of tungsten and titanium carbides, ceramic phase concentration of 0.033 %) allowed increasing relative elongation by 40 %.

The microstructure of samples is comprised of austenite and carbides isolated inside and along the borderlines of grains, as established (Fig. 3). The grain size in samples 2 and 3 corresponds to two points (actual medium grain diameter is 0.387–0.694 and 0.338–0.581 mm, respectively), in sample 4 – more than three points (2.318–3.420 mm); actual medium grain diameter of reference sample 1 exceeds three points (2.325–3.168 mm).

Thus, compositions based on nanodispersed tungsten and titanium carbides obtained by the combined method (SHS combined with MA) demonstrated the maximum efficiency compared to other modifiers studied in experiments on intra-mould modification of grey cast iron and 110G13L steel.

#### Modification of grey cast iron

**Investigation of the effect of various process parameters on service characteristics.** Further experiments were carried out using the most efficient modifiers based on a mixture of tungsten and titanium carbides.

Two compositions of modifiers were used: M1 – 25 % (WC, TiC)/50 % Cr; M2 – 25 % (WC, TiC)/25 % Cr/50 % Cu and two moulds, such as No. 2 (standard – for 40 kg of metal, No. 3 – for 19 kg (as a beaker).

#### Study of the effect of modifier concentra-

TABLE 3

Cast iron modification conditions (metal mass in mould No. 2 of 40 kg)

Samples	Modifiers			Ceramic phase concentration, %	Modifier introduction technique
	Names	Ceramic phase (WC,TiC), %	Metal-protector Cr/Cu, %		
2	M2	25	25/50	0.033	Форма
3	M2	25	25/50	0.013	Форма
4	M2	25	25/50	0.034	Ковш
5	M1	50	50/0	0.062	Ковш

Note. Sample 1 is reference (non-modified).

TABLE 4

Properties of cast iron samples

Samples	Tensile strength, MPa	Brinell hardness, HB	Change of strength, %	Change of hardness, %	Relative corrosion resistance, %
1	178	177			
2	211	166	18.5	-6.6	13.7
3	209	197	17.4	11.3	7.2
4	190	182	6.7	2.8	44.1
5	180	194	1.1	9.6	37.6

**tion and introduction method.** Material composition to prepare moulds is sand-clay mixture (SCM). There were used M1 and M2 modifiers with a concentration of ceramic phases of modifiers within 0.013–0.062 %.

Table 3 gives modification conditions (modifier composition and amount and introduction method): sample 1 is reference one without a modifier, samples 2–4 – with modifier M2, sample 5 – with modifier M1. Modifiers for samples 2 and 3 were placed into mould riser (between two filters), into samples 4 and 5, the former were introduced into a ladle under metal jet (its amount in there is identical to that in a mould, about 40 kg).

Table 4 gives properties of cast iron samples (tensile strength, Brinell hardness, and a change in strength and hardness). As can be seen, an increase in tensile strength was from 1.1 to 18.5 % (samples 2–5), in Brinell hardness – from 2.8 to 11.3 (samples 3–5), in hardness for sample 2 reduced by 6.6 % in relation to reference sample 1.

The relative corrosion resistance increased from 7.2 to 44.1 %, moreover, the maximum values (37.6 and 44.1 %) were acquired during modi-

fication in a ladle (samples 4 and 5, respectively).

Figures 4–6 give micrographs of thin sections of cast iron samples. There is grinding of graphite inclusions for all modified cast iron samples (see Fig. 5 and 6) compared to the reference sample (see Fig. 4). Samples modified in a mould (Fig. 5, samples 2 and 3) and in a ladle (Fig. 6, samples 4 and 5) differ by distribution and shape of graphite inclusions.

#### Study of the effect of natural ageing.

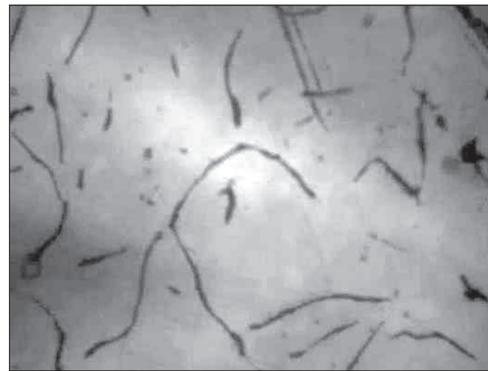


Fig. 4. Micrograph of unmodified sample. Magnification of 400.

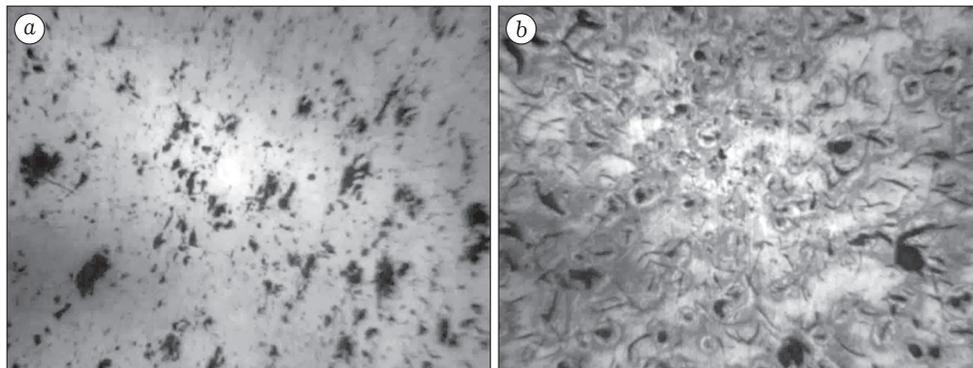


Fig. 5. Micrographs of samples modified in a mould: a – sample 2 (WC, TiC – 0.033 %); b – sample 3 (WC, TiC – 0.013 %). Magnification of 400.

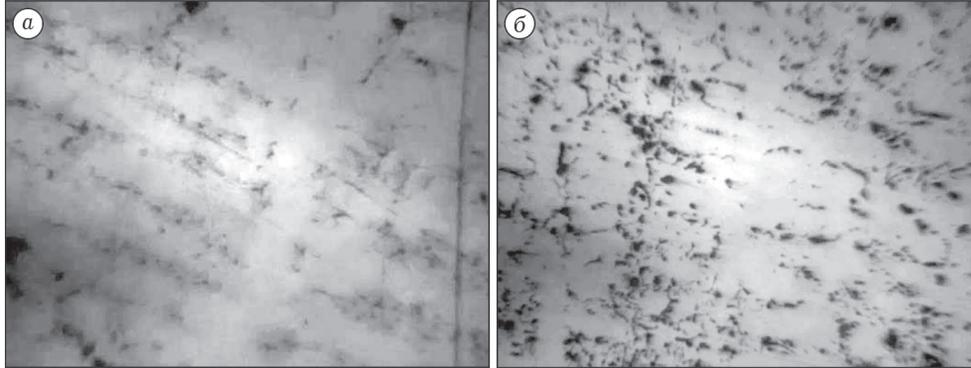


Fig. 6. Micrographs of samples modified in a ladle: *a* – sample 4 (WC, TiC – 0.034 %); *b* – sample 5 (WC, TiC – 0.062 %). Magnification of 400.

After 3.5 months of storage (natural aging), the strength of samples 2 and 3 modified in a mould decreased by about 1–2 %, in other words, almost did not change; reference sample strength decreased by 4.7 % (sample 1) (see Table 5).

The strength of samples 4 and 5 modified in a ladle (the concentration of ceramic particles of 0.034 and 0.062 %, respectively) increased by about 16–18 %.

**Modifier concentration effect on corrosion resistance.** Modifier concentration effect on corrosion resistance was explored for two cast iron grades that were SCh20 and SCh30. A model of mould No. 3 was used. Material composition to prepare moulds is SCM and CHM (SCM was utilised for the reference sample).

There was used M2 modifier; the concentrations of the ceramic phase of the

TABLE 5

Effect of natural aging

Entry	Initial samples		Samples after 3.5 month of storage		Change in strength resulting from aging, %
	Tensile strength, MPa	Change of strength in relation to K, %	Tensile strength, MPa	Change of strength in relation to K <sub>1</sub> , %	
1	178 (K)		170 (K <sub>1</sub> )		-4.7
2	211	18.5	209	22.9	-0.9
3	209	17.4	205	20.6	-1.9
4	190	6.7	220	29.4	15.8
5	180	1.1	213	25.3	18.3

TABLE 6

Modifier concentration effect on corrosion resistance (metal mass in form No. 3 of 19 kg)

Entry	Cast iron grade	Modifier	Concentration of ceramic phase, %	Mould mixture	Mass loss (25 % HCl, 24 h, 23°C), %	Increase of relative corrosion resistance, times
1	SCh20	–	–	IIFC	10.3	0
2	SCh20	M2	0.009	IIFC	10.3	0
3	SCh20	M2	0.025	IIFC	7.1	1.45
4	SCh20	M2	0.050	IIFC	7.2	1.42
5	SCh30	M2	0.060	XTC	7.5	1.37
6	SCh30	M2	0.080	XTC	6.4	1.61
7	SCh20	M2	0.100	IIFC	6.2	1.66
8	SCh20	M2	0.200	IIFC	4.0	2.58

modifier within 0.009–0.2 % were studied.

The relative corrosion resistance in 25 % HCl increased by 1.4–2.6 times (samples 3–8, Table 6) within ceramic phase concentrations of 0.025–0.2 %.

## CONCLUSION

### 1. Modification in mould No. 2.

1.1. The use of M2 modifier (ceramic phase concentration of 0.033 %) during intra-mould modification of grey cast iron contributes to an increase in tension strength by 18.5 %, in the relative corrosion strength in hydrochloric acid – by 13.7 %. However, this decreases hardness by 6.6 % in relation to the reference sample. Both hardness (by 11.3 %) and tension strength (by 17.4 %) are increased with ceramic phase concentration of the modifier of 0.013 %; the relative corrosion strength increases by 7.2 %.

1.2. During treatment of melt in a ladle with M2 modifier (ceramic phase concentration of 0.034 %), tension strength increased by 6.7 %, hardness – by 2.8 %; the relative corrosion resistance rose by 44.1 %.

1.3. During treatment of melt in a ladle with M1 modifier (active phase concentration of 0.062 %), tension strength increased slightly (by 1.1 %), hardness – by 9.6 %, the relative corrosion resistance – by 37.6 %).

1.4. After 3.5 months of storage, the tensile strength of reference sample and samples modified in a mould almost did not change, while that of samples modified in a ladle (concentrations of ceramic particles of 0.034 and 0.062 %) increased by about 16–18 %.

### 2. Modification in mould No. 3.

2.1. The relative corrosion resistance of cast iron samples in hydrochloric acid rose by 1.4–2.6 times within ceramic phase concentrations of 0.025–0.2 %.

The findings attest to the fact that during the study of modification processes of grey cast iron with modifiers containing nanodispersed ceramic phases, one should consider not only modifier concentrations and compositions but also various

process parameters (design, mould volume, and modifier introduction method, etc.).

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