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STUDY OF THE EFFECT OF NANOMODIFIERS FROM SILICON PRODUCTION WASTES ON MORPHOLOGICAL FORM OF GRAY CAST IRON GRAPHITES

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ABSTRACT

The influence of modifiers obtained from silicon production waste on the morphological form of graphite in gray cast iron was evaluated. Experimental melts were performed to study the effect of nanomodifiers. Series of ten samples were obtained, in each of which one sample was a control sample without the use of an experimental modifier. The other nine samples of each series were obtained using the studied additives. Cast iron was smelted in an induction furnace of the IST-0.6/0.4 brand, with a capacity of 500 kg. The conducted researches included: determination of the chemical composition of cast iron; determination of the hardness of samples using the Brinell method; tensile testing of samples; study of the macro- and microstructure of gray cast iron. The graphite phase in the modified cast iron was obtained using our proposed modifiers and is represented by vermicular graphite. The mechanisms of graphite formation using the suggested modifiers are considered.

Introduction

It is recognized [1] that all graphite forms that are deposited from metallic solution should develop from the main hexagonal ring structure, transforming in an open single-layered sheet. Multiple works mention [2–6] that graphite increases in metallic solutions with different morphology, including graphite plates, spheres, vermicular graphite, star-shape graphite and lately developed single-layered grapheme [6, 7]. Graphite formations of different morphology have decisive importance because they provide substantial effect on the properties of metal-graphite composite. Three basic graphite morphologies are crystallized from Fe-C-Si melt during solidification depending on composition and cooling rate: lamellar graphite (LG), compacted (compressed) or vermicular graphite (CG) and spheroidal graphite (SG).

Typical morphology of graphite microstructures determines physical and mechanical properties of iron. At present time the approved standards ASTM A 247-67, DIN EN ISO 945 and GOST 3443-87 help to classify graphite morphology of cast iron structures according to their form, location and size, using micro-pictures of the structure presented in these standards [8–10] as a sample.

The work [11] presents the review of technical literature devoted to formation of iron microstructure with compacted graphite. It is shown that iron with compacted graphite has intermediate graphite morphology between iron with lamellar graphite and iron with spheroidal graphite. The work [12] examines the effect of alloying elements and cooling conditions on iron with compacted graphite. It is shown that Mg has large effect on graphite morphology and causes significant increase of nodosity. Such elements as Cu, Si and Sn cause only slight variations of graphite form morphology. The authors of the work [13] tried to present the renewed information about achievements obtained in understanding of the mechanisms that control and manage nucleation and crystallization of spheroidal graphite and connected with it imperfect morphologies from Fe-C-Si melts. The authors of the work [14] did not agree with false affirmations made in [13], and discussion started [15]. At recent time the tendency of use of nanomodifiers for improvement of gray cast iron properties can be noted [16–22]. So, in the work [16] the complex evaluation of modifying effect, provided by ultra-dispersed powders of oxides of refractory metals and cryolite, on structure and deformation behaviour of cast gray iron of SCh25 brand was conducted. It was established that modifying of gray iron by ultra-dispersed powders of oxides of titanium, zirconium (containing Nb, Hf, Mg, Fe, Cr, Sr, Mo impurities not more than 5 %) and cryolite leads to essential variations of lamellar graphite distribution in a matrix and its sizes. At present time more than 10 mln. t of wastes needed to be processed are collected at slime fields of "Kremniy" JSC. The works [17, 18] display that use of modifiers made of silicon production wastes improves mechanical properties of gray iron. The authors of [16–22] works don't consider the effect of graphite forms that are deposited from metallic solution on mechanical properties, when nanomodifier of any composition is used.

Though growth of lamellar graphite during modifying is examined rather well, growth of compacted (compressed, vermicular) large-size and spheroidal graphite is still in the center of attention of investigations not only for conventional melting processes, but also for modifying cases. Efficiency of modifying effect on graphite morphological forms depends on many factors, such as composition and amount of a modifier, way of location, pouring temperature, choice of gating system, as well as geometry and size of a casting. As a rule, only several of these interacting factors were considered together in the published researches on this matter. It can be the main cause of different conclusions.

The aim of this work is reveal of forming of graphite morphological form during introduction of nanomodifiers from silicon production wastes.

Materials and methods of the research

Experimental melts were performed to study the effect of nanomodifiers. Series of ten samples were obtained, in each of which one sample was a control sample without the use of an experimental modifier. The other nine samples of each series were obtained using the studied additives. Two schemes of introduction of nanomodifiers were examined: melt pouring on modifier located on the ladle bottom and so-called "sandwich process", when metal was poured on modifier with addition of steel cutting wastes. It was established [18] that the tested schemes of modifier introduction have the same effect on the results, thereby more simple method of introduction was chosen further.

Cast iron was smelted in an induction furnace of the IST-0.6/0.4 brand, with a capacity of 500 kg. Charge materials include gray iron scrap (60 %), steel scrap (40 %), ferromanganese (with 80 % Mn), ferrosilicon (with 65 % Si), ferrophosphorus (with 26 % P). After charge smelting, the sample was taken to determine melt chemical composition. The following temperature procedure was kept during smelting: overheating temperature made 1460–1480 °C, temperature of introduction of modifiers was 1430–1450 °C, casting temperature — 1410–1420 °C. Pyrometer was used to control temperature.

Slag was removed from the melt surface before metal casting in the ladle with a capacity of 240 kg. Iron casting in sand moulds was conducted using one ladle. Modifiers with different composition [18, 22] were produced from silicon production wastes and then were used after special flotation processing.

Modifier 1 contained mainly amorphous carbon and carbon nanotubes (94 %), also reminders of silicon dioxide and silicon carbide (4 %) and non-reacted reminders of graphite anodes and other impurities (totally 2 %). Modifier 2 contained SiO_2 (quartzite phase) — 50 %, SiC (moissanite) — 35 %, SiO_2 (cristobalite) — 10 %, C (graphite) — 5 %.

Compaction of a modifier was conducted from obtained mechanical mixtures either via pelletization using press, or via manual globulization using paraffin. The conducted researches included: determination of the chemical composition of cast iron; determination of the hardness of samples using the Brinell method; tensile testing of samples; study of the macro- and microstructure of gray cast iron.

Results of the research

Determination of mechanical properties

It is known that minimal value of tensile strength and hardness are the main parameters of mechanical properties for gray cast iron. **Tables 1 and 2** present the results of mechanical tests for iron-testifier and samples after modification. Iron consumption was varied to evaluate the effect of modifier amount on cast iron properties (kg/t).

Table 1. **Testing results for the modifier containing 95 % of silicon dioxide**

Sample	Consumption of modifier, kg/t	Hardness, HB	$\sigma_в$, MPa	Corresponding to iron of brand
Initial	–	195, 201	143, 152	SCh10, SCh15
No. 1	0.5	196, 200	145, 151	SCh10, SCh15
No. 2	1	205, 208	165, 174	SCh15
No. 3	2	225, 231	260, 265	SCh25
No. 4	3	255, 260	305, 310	SCh30
No. 5	4	263, 270	350, 355	SCh35
No. 6	5	265, 269	360, 365	SCh35
No. 7	6	264, 271	360, 365	SCh35

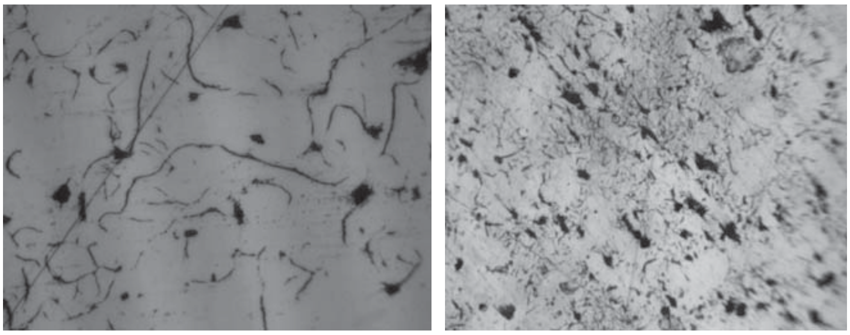
Table 2. **Testing results for the modifier containing 94 % of carbon**

Sample	Consumption of modifier, kg/t	Hardness, HB	$\sigma_в$, MPa	Corresponding to iron of brand
Initial	–	195, 201	143, 152	SCh10, SCh15
No. 1	0.5	195, 202	146, 153	SCh10, SCh15
No. 2	1	209, 214	225, 230	SCh20
No. 3	2	226, 233	270, 275	SCh25
No. 4	3	256, 258	315, 320	SCh30
No. 5	4	264, 270	365, 375	SCh35
No. 6	5	262, 267	368, 374	SCh35
No. 7	6	265, 269	369, 375	SCh35

Metallographic research

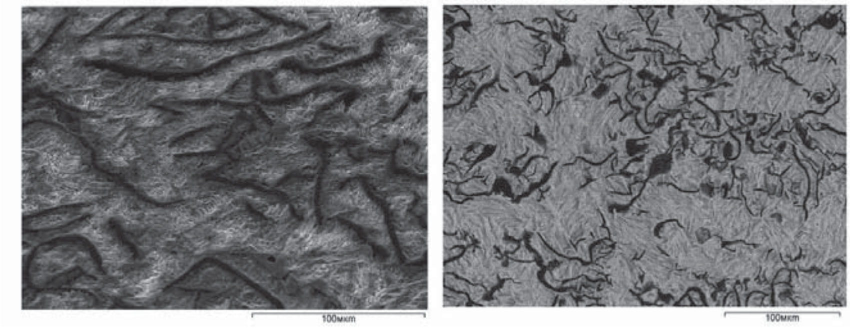
Study of macro- and microstructure of gray cast iron was conducted in accordance to GOST 3443-87 using optical and electronic microscopy, and allowed to reveal the features of the effect of modifiers. **Fig. 1 and 2** present the results of optical and electronic microscopy. Let us consider the features of microstructure of samples-testifiers obtained without modification (fig. 1*a* and 2*a*). The structure is presented by lamellar graphite, form of optical and electronic microscopy graphite inclusions is lamellar and straight (PGf1), length of their inclusions (PGd2) is within the range 90-180 µm. Distribution of graphite inclusions is heterogeneous and net-kind (PGr6). The amount of graphite inclusions (%) varies from PG2 to PG4. Kind and structure of metallic base contains pearlite and ferrite P85. The distance between plates in pearlite makes 1.4-1.6 mm, what testifies about low mechanical properties of this structure. Diameters of cells in phosphide eutectics made from 500 to 750 µm (Fed650). The nets of phosphide eutectics are torn (FEr2).

Graphite phase in modified cast iron manufactured with use of the modifiers suggested by the authors is presented by vermicular graphite (fig. 1*b* and 2*b*). The form of



(a) *(b)*

Fig. 1. Optical microscopy of the surface of cast iron samples: non-modified (*a*) and modified (*b*)



(a) *(b)*

Fig. 2. Electron microscopy of cast iron samples: non-modified (*a*) and modified (*b*)

graphite inclusions is VGf1 (node-type) and VGf3 (thickened). Distribution of graphite inclusions is socket-type. The amount of vermicular graphite inclusions (%) is VG70. Kind and structure of metallic base contains Pt1. Content of pearlite and ferrite in cast iron structure is P92. Pearlite dispersity in the structure of cast iron varies from Pd0.3 to Pd0.5. It is shown that dispersity increased in comparison with non-modified sample. Structure of inclusions of phosphide eutectics is determined as ternary small-needle FE3, while distribution of inclusions of phosphide eutectics — as torn FEr2.

Discussion of the results

Analysis of the results presented in the tables 1 and 2 shows that both modifiers are characterized by good modifying properties. Higher mechanical properties are revealed in the modified samples in comparison with a sample testifier. It was established based on these researches that rational consumption of the modifier makes 2-5 kg/t. Conventional melting process is finalized in production of cast iron with the properties corresponding to the properties of SCh10, SCh15 (sample testifier), while melting accompanied by modification using suggested modifiers leads to manufacture of cast iron with the properties corresponding to the properties of SCh25, SCh30, SCh35 (see tables 1 and 2). The suggested modifiers were compared with the standard modifier FS75 (it was mentioned earlier in the work [22]), and increase of the positive effect on the structure and properties is shown. It is known from the theory and practice of casting production that efficiency of modification in melting of gray cast iron can be checked during processing of cast iron with low carbon equivalent. It is typical for gray cast iron SCh30, SCh35. The chemical composition of cast iron was analyzed (see **table 3**), and mechanical properties of this cast iron correspond to those of SCh35 cast iron during modification by the modifier containing up to 95 % of silicon dioxide (melt 1), and by the modifier containing up to 94 % of carbon (melt 2). It was established that introduction of these modifiers influenced on varying of chemical composition: on carbon content (for carbon modifier) and on silicon (for silicon dioxide modifier). At the same time mechanical properties correspond to those of gray cast iron SCh35 (see **table 4**). The work [24] noted that increase of silicon content in malleable cast iron enlarged its tensile strength and decreased its ductility and destruction blow energy.

Our investigations displayed that mechanical properties does not disimprove at small silicon content elevation after modifying by silicon dioxide. The results of conducted experiments show that all examined modifiers improve mechanical properties of gray cast iron (from one side), but increase of hardness and tensile strength values is characterized by slight differences starting from 5 kg/t (from other side). These differences seem to be connected with different influence of the developed modifiers on the structure. Microstructure of metallic base as well as size and type of graphite plates

Table 3. **Chemical composition of cast iron (experimental melts 1 and 2, mass. %)**

Element	Carbon	Silicon	Manganese	Phosphorus	Sulfur	Chromium	Nickel	Molybdenum
Melt 1	3.00	1.60	1.04	0.086	0.052	0.59	0.06	0.14
Melt 2	3.15	1.56	1.05	0.098	0.055	0.61	0.06	0.14
Iron of brand SCh35 (GOST 1412-85)	2.9–3.0	1.2–1.5	0.7–1.1	≤0.2	≤0.12	–	–	–

Table 4. **Mechanical properties of the experimental melt of gray cast iron SCh35 with obtained modifiers**

Parameter	Value of SCh35 cast iron according to the GOST 1412-85	Actual value		Standards for testing methods
		Melt 1	Melt 2	
Tensile strength σ_B, MPa	≥350	385		GOST 27208-87
Hardness HB	179–290	255		GOST 9012-59

has the effect on properties of gray cast iron. Amount of graphite inclusions and their size, morphology and distribution of graphite plates have the decisive importance during mechanical tests. We can see on the sample testifier (**fig. 1*a*, 2*a***) that graphite has morphological plate-type form. The conducted tests displayed that dendrite structure with exogenous and partly endogenous dendrites along whole surface of metallographic specimen is clearly expressed in non-modified cast iron. When cast iron is processed by conventional modifier FeSi, a net of exogenous dendrites crossing the surface of metallographic specimen is presented with the background of homogeneously distributed graphite inclusions [22].

The work [25] displays that Al, Ce and Zr can increase amount of dendrites, providing direct influence on austenite nucleation. The authors of this work revealed that Sr-FeSi is efficient for cleaning of eutectic colonies and nucleation of primary austenite [26]. The authors of [27] informed that homogenous structure consisting of uniform distribution of A-type graphite and increased amount of eutectic colonies can be obtained using SiC nanomodifier. Dissociative carbon forming during $SiC + Fe \rightarrow FeSi + C$ reaction provides then graphite nucleation centers due to high activity and zero divergence [27].

In our case, when cast iron is produced with treatment by the modifier 1, including nanocarbon components, dendrites are not observed in the samples of this cast iron, while amount and size of graphite inclusions increased substantially. The features of lamellar graphite distribution looks like the colonies of dendrite directed structure, according to the GOST 3443-87. Essential decrease of lamellar graphite length was established. This length after modification is within the range from ~10 to ~80 μm and for a testifier sample this interval makes 45–160 μm, so this interval is 2 times smaller in comparison with non-modified sample.

Morphological form of graphite varies in the cast iron samples after treatment by the modifier 2 (containing up to 95 % of silicon dioxide in the form of spheres with size of particles up to 100 μm, meaning that 90 % of particles have size less than 30 μm). Compressed or vermicular graphite is forming (see fig. 2). Its typical morphological features on the surface of metallographic specimen are noted as "round, thickened and mostly unbranched short graphite plates". It is known [11-15,23], that vermicular graphite form provides not only increased mechanical properties of cast iron, but also varying of physical (heat conductivity) and casting (linear and volumetric shrinkage) properties. The mechanism of vermicular graphite forming is observed in the work [28], where especial role of non-metallic inclusions in the process of graphite forming is underlined. Especial role of non-metallic inclusions containing oxygen (oxides, oxysulphides etc.) is also noted in many other works. The authors of the works [1-4, 11-15, 28] think that just these compounds are the nuclei of graphite inclusions. So, it was found out [29-31] that oxygen and aluminium are presented in the centers of non-metallic inclusions having irregular (multi-plane) or round (spheroidal) form of (Mn, X)S type, where X = Fe, Al, O, Ca, Si, Sr, Ti etc. Such inclusions are often also coated by thin layer of silicates and they are the main centers of lamellar graphite forming in experimentally molten cast iron. Three-stage model of graphite nucleation in gray cast iron is suggested [30, 31], where small inclusions of oxides (less than 2 μm) are forming at the first stage, then more large (less than 5 μm) complex (Mn, X)S compounds are deposited, and at the last 3rd stage graphite is deposited at the sides of sulphides of (Mn, X)S compounds having low crystallographic divergence with graphite. The works [31,32] testify that silicon deoxidizes cast iron at lower temperatures, and at higher temperatures this function is realized by carbon. Oxygen activity is also in direct proportion to the form of graphite inclusions (lamellar, vermicular, spheroidal) [32]. According to the work [33], oxygen in graphitized cast iron has substantial effect on crystallization and cast iron properties. Direct relationships between oxygen content and form of graphite inclusions in cast iron that are modified by different alloying compositions (Ni-Mg and Fe-Si-Mg) [33] were established. The work [34] testifies that oxide bifilms are usually presented in a cast iron melt. These silicate oxide films provide substrate as a base for forming of oxysulphides and graphite nuclei). Presence of these bifilms explains the whole variety of graphite morphology. Lamellar graphite grows along the films, while spheroidal graphite grows during destruction of these films, e.g. at magnesium addition. The work [35] shows that appearance of vermicular graphite form was connected not only with interaction between silicon and carbon oxide, but also owing to interaction between silicon monoxide with graphite nuclei (in this case). Lowering of metallic melt temperature leads to SiO surface activity decrease: its mobility reduces and it is solving in graphite in the place of nucleation, replacing its morphology on vermicular one. It should be noted that the above-described works display the important fact — direct or indirect effect of oxygen (in the form of oxides) on forming the structure of Fe-C melts. However, no common mechanism of oxygen effect on structure forming was developed, and additional investigations in this field are required. It was revealed in our case that the particles of silicon dioxide were dipped in a matrix and have surface contact with graphite in all

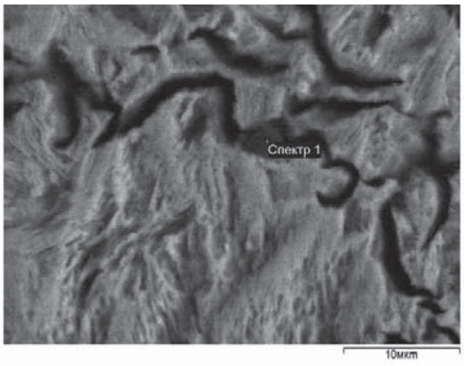


Fig. 3. Electronic image displaying SiO_2 in graphite plate

samples (**fig. 3**). Differences are mainly presented in morphology and distribution of particles.

It could be suggested as a working hypothesis, that solidification in modified hypoeutectic melt starts from independent nucleation of austenite and graphite dendrites. When dendrites grow and start to contact with graphite in the conditions of lowered temperature, the particles of lamellar graphite and austenite grow together and finally form eutectic colonies. Large amount of compounds with SiO_2 particles in cast iron means more centers of nucleation for austenite and graphite in the beginning of solidification and thereby more possibilities for interaction between primary austenite and graphite. As a result, growth of primary dendrites is suppressed due to more early growth of eutectic colonies. The fact that more short graphite plates and more large eutectic colonies were found out in cast iron processed by modifiers 1 and 2 confirms this suggestion.

Looking from the practical point of view, the suggested composition of nanomodifiers, obtained from the wastes of silicon production, was examined and tested in industrial conditions at "Irkutsk metallurgical company" JSC during manufacture of gray cast iron castings; essential decrease of casting rejects (by 25 % in average) was noted. Economical efficiency based on cutting the expenses on purchasing and consumption of charge materials (in particular, the conventional modifier FS75), as well as taking into account decrease of casting rejects amount, made 960,000 rubles per year for this enterprise. The results of investigations allow to recommend this type of modifier for gray cast iron production in order to decrease casting rejects and improvement of mechanical properties. The modifying mixture on the base of nanomodifiers can be applied at the operating production facilities without change of technological processes and using the existing plant equipment, without any additional devices and without necessity of personnel training.

Conclusions

The conducted researches, aimed on evaluation of modifying effect of the new modifiers (obtained from silicon production wastes) during melting of gray cast iron, displayed their high efficiency in comparison with conventional modifiers. Substantial decrease of the length of lamellar graphite was established for the modifier 1. The length of lamellar graphite after modification varies within the range from ~ 10 to ~ 80 μm, and for a testifier sample this interval makes 45–160 μm, so this interval is 2 times smaller in comparison with non-modified sample.

Dispersed composition of the modifiers has essential effect on variation degree of morphology of graphite inclusions. The developed modifiers lead not only to increase of the part of lamellar graphite form, but also to variation of the form from lamellar to nest-type (socket-type), vermicular, compact (modifier 2). It also rises strength of gray cast iron by 35–50 %.

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